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Research Article

Development of botanical-based wood preservative formulation for plywood and evaluation of its Anti-termite properties in a field trial

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ABSTRACT

An alternative type of wood preservative formulation, which can replace the toxic chemical based wood preservatives, is the need of the hour. In this line, many researches are being explored. However, an effective formulation is not yet being developed. In case of plywood, development of alternative wood preservatives that can be mixed with glue line is a bigger challenge because some properties of preservatives are likely to hinder the plywood bonding qualities. In the present study a preservative formulation using a synergetic mixture of different botanicals, along with copper sulphate, lignin powder and silica powder was developed. Test samples were prepared by mixing the two concentrations (1.5% and 2%) of preservative formulation with Urea-formaldehyde resin to perform the glue line poisoning and control samples without preservatives were prepared. Water resistance test to evaluate bond quality were carried out for both control and test samples. Field trials against termites were also carried out although the UF resin was used only for interior grade works and field trials were not necessary. The fungal resistance studies failed to show any significant result indicating the preservative formed is not effective against wood destroying fungus prevention.

Keywords: Wood destroying fungus, Termites, Plywood, Urea formaldehyde, Internal bonding

INTRODUCTION

Treated wood extend the service life of structural materials and reduces the costs and pressure on forests by decreasing the number of repairs and replacement of wood products (Khademibami and Bobadilha 2022). Thus, the utilization of treatments is necessary in order to improve the useful life of wood and wood products, consequently, to protect against biodegradation. At present, wood products are mostly treated with synthetic organic and inorganic substances, which are harmful to the human health and environment. The wood treatment with toxic compounds, such as CCA (Chromated Copper Arsenate) increases the environmental impacts due to the presence of arsenic. Moreover, the commercialization of products with arsenic is limited, proving the necessity of the development of new products for wood preservation. Consumer awareness related to environmental issues caused by toxic chemicals is resulting in avoiding the use of toxic chemicals and develop new technologies based on low environmental impact agents and sustainable principles. As a result, serious efforts are being made globally to develop alternative protection methods based on natural products with little or no toxicity. Botanical based wood preservation methods could be suitable alternatives in this regard. The plants reviewed shows good insecticidal properties against borers, fungal and termites.

Although botanicals have generated extraordinary interest in recent years as potential sources of natural insecticide control agents, they are used as insect repellent from time unmemorable. Way back in the middle of the 17^{th} century itself pyrethrum, nicotine and rotenone were recognized as effective insect-control agents (Crosby 1966).

Today over 2000 species of plants are known that possess some insecticidal activity (Jacabson, 1989). Plants produce a range of aromatic and non-aromatic compounds, some of which are recognized anti-insecticidal/antimicrobial agents. Secondary

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metabolites from plants such as Phenols, terpenoids, alkaloids, lectins and polypeptides are being extensively used in various insecticidal applications (Geissman 1963). Different plant parts, such as bark, wood, leaves, seeds and fruits which are known to possess a different kind of secondary metabolite derivatives are been evaluated for their wood protection properties (Yang 2009). Among sources of plant oils, flax seeds (Lyon et al. 2007), cinnamon (Lin et al. 2007), citrus peels (Macias 2005) and Tung seeds have been experimented as potential wood protectors, showing diverse activities like antibacterial, antifungal, anti-termite and anti-nematode agents.

This fungal and termite resistance ability of selected weed plants was already established on the solid wood. Botanical extracts from leaves of *Lantana camara*, *Ricinus communis*, *Catharanthus roseus* and *Calotropis procera* are already tested and proved for their insecticidal and anti-termite effect. Leaves of *L. camara* plant extracts have exhibited a broad range of biological activities such as antimicrobial, antimutagenic, allelopathic, antioxidant, anti-proliferative, bactericidal, fungicidal, nematicidal (Ghisalberti 2000; Ganjewala et al. 2009; Sharma et al. 2007; Sousa et al. 2012) and anti-insecticidal (Innocent et al. 2008; Abdel-Hardy et al. 2005). In the ray of anti-insecticidal activity leaves of *L. camara* plant extracts exhibit anti-feeding, anti ovipositing, larvicidal, repellent and toxic effects (Fatope et al. 2002; Innocent et al. 2008; Saxena et al. 1992; Verma and Verma 2006). Anti-termite activity of the leaves of L. camara plant extracts was established by the studies conducted by Ding and Hu (2010). Under Indian context, Verma and Verma (2006) studied, the effect of *L. camara* leaves chloroform extract against termite species *Microcerotermes beesoni Snyder* and found that 5% concentration showed LD_{50} values and this is because of triterpenoids present in the L. camara plant leaves. Aqueous extracts of *R. communis* and *C. procera* was tested against termite species *Odontotermes obesus Rambur* (Sahay et al. 2014; Upadhyay 2013) and extracts were highly effective and caused 100 % mortality in a termite.

The studies related to the toxic effect of aqueous extract of C. procera against termite species *Heterotermes indicola* and *Coptotermes heimi* clearly indicated that it is highly effective to control termite (Badshah et al. 2004). The highest termite mortality (50%) was found in termite species *Anacanthotermes ochraceus* (Bourmita et al. 2013) when aqueous extract of *C. procera* was used. This is attributed to the presence of flavonoids, a major class of phytochemicals constituting 5%-10 % of the known secondary metabolites in plants (Upasani et al. 2003; Sahay et al. 2014), similarly, extracts of *C. roseus* leaves also showed the promising effect as a botanical insecticide (Prajapati et al. 1998) by acting as a feeding deterrent, growth inhibitor and ovicidal on the activity of various insect pests.

Although each botanical extractive can act as termite resistance substance, we used the mixture of all the plant extract as synergistic effects of different plant extract will provide more biocidal activity than the individual plant extract. In the synergetic mixture, different metabolites result in higher bioactivity compared to the isolated components (Hummelburnner and Isman, 2001; 2004; Omolo et al. 2004; Nerio et al. 2010; Gillij et al. 2008). In case of panel wood protection, this particular quality of small concentration providing higher level of protection is extremely desired, as there is a chance that preservative added in the glue line will impede the bonding property of the final product. Based on which study was under taken with following scope and objective.

Scope and objective

There are increasing awareness and interest in the use of natural products as a wood preservative as these products ensures the good health of human as well as the environment. As a part of the project, we tried to develop wood preservative formulation for plywood with following objectives.

- Screening of weed plants for secondary metabolites
- Modification of extractives (by addition of inorganic metals or fractionation) for development of different formulations for wood panel products protection.
- Optimization of preservative formulation for the panel products.
- Test the extractive efficacy against termites and fungal attack.

MATERIALS AND METHODS

Screening of weed plants for secondary metabolites

Screening and selection of weed plants was done based on the earlier literature which already mentioned that leaves of *L. camara*, *Datura stramonium*, *R. communis*, *C. roseus* and *C. procera* were highly effective as solid wood preservative. For a plywood preservative formulation preparation, also same plants were selected.

Modification of extractives (by addition of inorganic metals or fractionation) for development of different formulations for wood panel products protection.

Wood preservative formulation preparation: Leaves of above-mentioned plants were collected separately from the institute campus. Leaves selected for extraction were free from any diseases, fungal and insect infestation. Leaves were cleaned thoroughly under running water and shade air-dried in the laboratory at room temperature. The cold-water extraction procedure was used to prepare the extractive. 100 g of each plant leaves were weighed on weighing scale. Post that all different species of leaves were mixed together and were ground with 800 ml of water using a mixer grinder this provided us the synergetic mixture. Initially only 500 ml of water was used but as the grinding was not possible so additionally, 100 ml of water was added based on requirement repeatedly which lead up to 800 ml of water. The solution obtained was filtered through a muslin cloth and What mann filter paper and filtrate was collected in a beaker and stored in the refrigerator. The residue obtained after filtering was weighed and was kept for drying. The dry weight of the extractive was noted. The solid content of the extractive was calculated based on the formula.

$$\textit{Extraction yield} = \frac{W1}{W2} \times 100$$

Where;
W1 is dry extract after extraction and
W2 is plant sample weight before extraction
3.5 g of copper sulphate and 10 g of silica powder were added in 100 ml of extractive and mixed thoroughly to avoid precipitate formation. 10 g of NaOH was dissolved in 20 ml of distilled water and this solution was added to the above mixture post 24 h of formation along with 20 g of lignin powder. This formulation was used as a wood preservative (pH-6.8) to treat the plywood in glue line treatment. The formulation was mixed with UF resin at different concentrations and was tested for its efficacy against termite in a field experiment.
Before finalizing the above-mentioned formulation, we tried other formulations such as which involved hot and cold extraction of materials from selected weed plants. Hot extraction was carried out with Petroleum ether (soxhlet extraction) and cold extraction with water. Plywood samples were prepared with mixing 1%, 2%, 5% concentrations of these hot and cold extractives in both UF and PF resins. When these samples were exposed to fungal, mould and termite studies, they failed miserably.
Infect in the extractive stock solution itself, fungal growth was observed making it clear that solution cannot be kept for longer duration and do not have any anti-fungal properties. Hence, copper sulphate is added for the fungicidal property. Lignin and silica were added to provide the insect resistance properties. Moreover, the solid content obtained from the extractive was very meagre, 2% to 3%. This small concentration of metabolites may not be sufficient to impart the wood preservative properties.
The work was continued with the hope that extractive modification with addition of small quantity of inorganic material it may have larger impact. Still, we started with higher concentration *i.e.*, 3.5 g of copper sulphate, 10 g of silica powder and 20 g of lignin powder. If this concentration was effective, the idea was to gradually reduce the concentration of this material in further formulations.

Optimization of preservative formulation for the panel products

Optimization of preservative formulation for panel product was based on the criteria that the preservative used should not affect the bonding quality of the panel product. Hence, initially we started with the small quantity of preservative (1.5% and 2% concentration) in the glue line.

Test board preparation

MR (Moisture Resistant) grade plywood 4 mm thick three-ply constructions were manufactured using rubber wood veneers of size 33 × 33 cm. Moisture content of veneers before glue coating was maintained at 8%-10%. Test samples of concentration 1.5% and 2% were prepared, for which The prepared wood preservative formulation was admxied with UF resin along with other additives mentioned in Table 1 as glue additive (Glue line treatment of plywood). Control samples were prepared without adding wood preservative formulation. The core veneer was coated with glue mixed with the preservative formulation and after the requisite pre-assembly time, glue-coated veneers were hot pressed in hot press at a temperature of 105ºC ± 5ºC, specific pressure 10 kg/ cm^2 and curing time was equal to the thickness of the ply.

Table 1. Adhesive formulation and flow time.

Materials	Weight
UF resin	200 g
Maida	20 g (10%)
Melamine	5 g (2.5%)
Ammonium Chloride	0.8 g (0.4%)
Water	1.6 g
Flow time of resin	22.34 seconds

Water resistance test (Cyclic test)

Bonding property tests of the plywood samples with extractives was carried out as per IS 848 (IS-2006). The samples were placed in the water at 60ºC ± 2ºC for 3 h in a hot water bath and dried for 8 h at 65ºC ± 2ºC and were repeated for three consecutive cycles. After three cyclic test, the samples were subjected to knife test to evaluate the bond integrity.

Test the extractive efficacy against termites and fungal attack

Termite resistance test: Prepared plywood stakes; 10 replications for each concentration and control were implanted in the soil with half-buried below and half exposed above the ground by following a Random Block Design in the termite test yard of the Indian Plywood Industries Research and Training Institute, Bengaluru located at 770.53’E longitude and 13o.03’N latitude.
The area is situated on the south Deccan plateau of peninsular India and has a landscape of dry deciduous forests and scrubs. Three distinct season, winter (December to February), summer (March to May) and monsoon (June to November) are found in this area. The area has an average rainfall of 859 mm and maximum temperatures of 36°C and a minimum temperature of 14°C with a humidity range of 35%-80%. Red sandy soil and highly undulating terrain are generally witnessed in this study area. Post implantation of stakes the surrounding soil was firmly pressed around the stakes to ensure good contact with the soil.
Visual examinations and physical check of the wooden stakes at all the testing sites was carried out at an interval of three months for a year after implantation. Any visible damage/attack by termites was recorded. Measurement of decay assed by probing the stakes and the material around each stake with a sharp pocketknife and assigning and rating of attack. If signs of termite attack/activity were found, the specimen was given a visual rating for termite attack based on a 10, 9, 8, 7, 6, 4, 0 rating system (Lebow et al. 2006) (Table 2).

Table 2. Rating system for severity of termite attack

Rating	Description of condition
10	Sound; one to two small nibbles permitted
9	Slight evidence of feeding to 3% of cross section
8	Attack from 3% to 10% of cross section.
7	Attack from 10% to 30% of cross section
6	Attack from 30% to 50% of cross section
4	Attack from 50% to 75% of cross section
0	Failure

Fungal resistance test

The fungal tests were carried out as per IS: 4873-1 (IS 2008). Weighed test and control blocks of prescribed dimension (6 replications for each) were introduced into Kolley's flask's containing the test fungi *Oligoporus placenta* (Brown rot) and *Trametes hirsuta* (White rot) respectively. Complete experimental setup was kept at room temperature of 25ºC ± 5ºC and 60%-70% of relative humidity. After span of 30 days, samples were removed from the flask and mycelium's adhering to the samples were cleaned with caution such that splinters of the samples were not removed. Then the test blocks are dried in the oven and the final weight was taken.

Statistical analysis

Data analysis was performed using SigmaStat ® 4.0 statistical software. Data obtained were subjected to Shapiro-Wilk normality test and Brown-Forsythe equal variance test. One-way analysis of variance was conducted to see the variance in the rate of infestation between the different concentrations of test samples and as well as control samples. All pairwise multiple comparison procedures were performed using the Holm-Sidak method.

RESULTS AND DISCUSSION

Optimization of preservative formulation for the panel products

Water resistance test (Cyclic test): Result of cyclic tests indicated that a test sample with 2% extractive concentration had delaminated, but the test sample with 1.5% extractive as well as the control test sample had passed. Which makes it clear that for the prepared formulation 1.5% concentration in glue line is optimal dosage. Glue line poisoning is the most preferred method to treat the plywood with preservatives as it is a simple, economical and less cumbersome process in which, toxic ingredient gets diffused into the veneers from the glue line during hot pressing making the plywood resistant to insect attack. Moreover, plywood is treated under glue line-poisoning method did not show any discolouration, pleasant to handle and relatively safe as the glue line is a safe location for toxic chemical. But the challenges in this process is to provide effective preservative properties with a small dosage of preservative chemicals as it was evident that preservative may interfere with gluing processes and associated bond-strength development during the hot-pressing stage of manufacture (Kalawate and Kiran 2015).

In our study, the concentration of 2% was highly effective dosage against termites but at this concentration of wood preservative formulation, the quality of plywood prepared was compromise and delamination of plywood layers was evident. This indicates the strong dose-dependent results between the quality of plywood prepared and the amount of wood preservative concentration. Slightest increase in dose (1.5% concentration to 2% concentration) resulted in decreases in bonding. We feel that the presence of silica dust might have disturbed the process of binding. Further research is needed in this line of study.

Test the extractive efficacy against termites and fungal attack

Termite resistance test: Samples subjected to graveyard test remained in the field for the maximum period of 24 months indicating that the botanicals preservative formulation prepared is not able to provide protection for the 36 months' period that is mandatory to approve any preservative formulation as effective as per BIS standards.

The control samples were different from test samples as the termite infestation started on them very early compared to test samples. We were able to observe the slight or negligible amount of termite activity sign when the stakes were removed after the implantation of six months. Whereas on the test samples the sign of termite attack was seen between 12 to 18 months. By the time of 18 months all termites ate control samples completely but it took 24 months to for the test samples to be completely eaten by termites.

However, the observation taken for the stakes implanted in graveyard test for the period of 24 months indicates that controlled sample has more termite infestation as it has an average rating of only 6.37 followed by concentration 1.5 ml preservative which has an average rating of 5.20 and concentration 2 ml preservative with an average rating of 7.17 (Figure 1).

The data obtained passed the Shapiro-Wilk normality test ($P=0,203$) and equal variance test (Brown-Forsythe; $P=0,675$). One-way ANOVA results showed that there is a statistically significant difference ($P=<0,001$; $F=21,829$ and $DF=29$). Holm-Sidak method of all pairwise multiple comparison procedures also showed a significant difference between different groups.

Control samples significantly varied from test samples of 1.5% concentration (difference of means=-0, 729; t=2, 511and P=0,018) and test samples of 2 % concentration (difference of means=-1,900; t=6,548 and P=0,001). Test samples of concentration 1.5% and concentration 2% also varied significantly (difference of means=-1,171; t=4, 037 and P=0, 001).

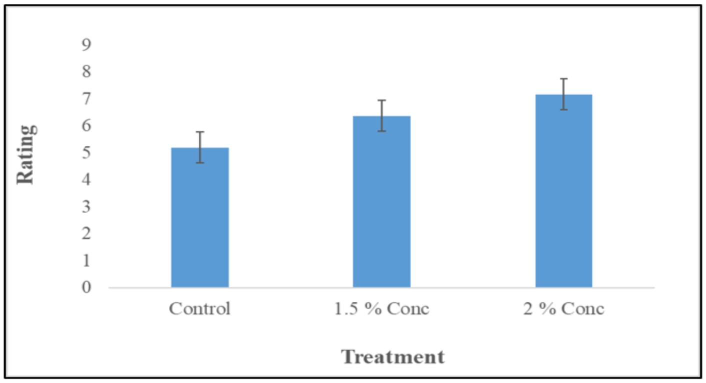

Figure 1. Graph showing performance rating of different treatments in graveyard test.

The result of graveyard test clearly indicates that preservative formulation developed fails to provide the protection for 36 months, which is minimum, pass standard recommended by Bureau of Indian standard to wood preservative chemical acceptance and efficacy. Although it failed to provide protection for 36 months, it has a positive impact and definitely contribute to the termite resistance ability of wood. Higher the amount of preservative greater the resistance to termite feeding.

The dose-dependent response of botanicals including essential oil ajowan (*Trachyspermum ammi*), allspice (*Pimenta dioica*), caraway (*Carum carvi*), dill (*Anethum graveolens*), geranium (*Pelargonium graveolens*) and litsea (*Litsea cubeba*) against termite was already been established by the studies conducted by (Seo 2009; Elango et al. 2012; Upadhyay 2013). Especially in the case of extracts of C. procera mortality rate was found a dose and time-dependent as it was found to be increased with an increase in the dose (Upadhyay 2013; Sahay et al. 2014). All these studies were carried out on solid wood and when we carried out the same study in plywood was could not confirm the above result only using the botanical extractives as result modifications were made with addition of copper sulphate, silica powder and lignin powder. In case of wood preservation using botanicals, to get the desired effect we have to use the large quantity of botanicals extractives, which is not fissile in case of panel wood as a large quantity of preservative is likely to cause the problem in bond strength (Kalawate and Kiran 2015) especially when preservatives are admixed with adhesive system.

Chemical interference of the preservative with the curing of the adhesive, reduction of the wettability of the wood and physical blockage of the surface where the adhesive attaches to the wood are likely to create the poorer bond ability of treated wood (Frihart 2003). Even the solvents used to treat wood with preservatives can sometimes cause bonding problems (Herzog et al. 2004). Considering the negative impact of preservatives on the bonding property of plywood, we have to use the small or negligible quantity of preservative in glue line considering the wood. Hence, it becomes mandatory to get a higher fungicidal or insecticidal effect with a small concentration of preservative. Considering the fact that synergetic mixture different metabolites may result in higher bioactivity it wise to develop botanical preservative with different plant extracts and mix of organic and inorganic salts.

Fungal resistance test

Data obtained was pooled together and Kruskal-Wallis one way analysis of variance on ranks was performed as the data obtained failed to pass the equal variance test result of which indicates that there is no significant difference between the different treatment groups and control samples (H=3,429; df=2 P(est.)=0,180 P(exact)=0,333).

Exposure of different treatments to the fungi *O. placenta* (brown rot) and *T. hirsuta* (white rot) showed very interesting result (Figure 2). Brown rot fungi showed clear cut dislike for the treated sample B. Clearly indicating that minimum inhibitory action of prepared wood preservative was seen only when 2 ml of preservative was added in the glue line. In contrast to this, White rot fungi showed higher level of feeding preference towards the treated samples B. followed by A and then C. Although this can be justified by the fact that white rot has the ability and to digest the lignin, as it is able to produce enzymes, such as laccase, which can digest the lignin and in case of white rot lignin is usually utilized at a somewhat faster rate in comparison to cellulose and hemicelluloses (Karim et al. 2017). This differential preference of test samples by made us think and when data was split into performance of wood preservative formulation against white rot fungi and brown rot fungi.

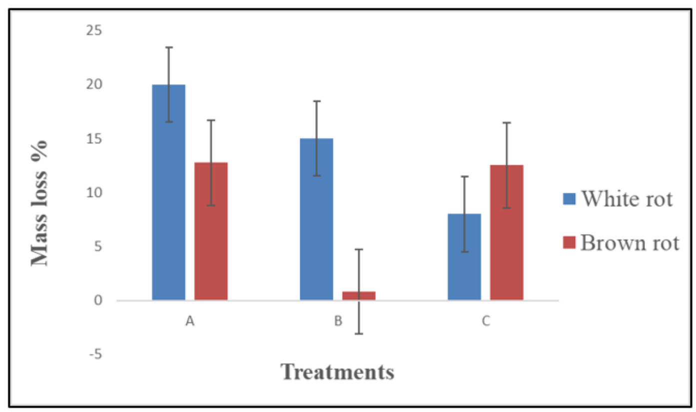

Figure 2. Percentage Mass loss of samples different treatments against test fungi *Oligoporous placenta* (Brown rot) and *Trametus hirsuta* (White rot).

Results of exposure trials against brown rot fungi *O. placenta* showed that there is a statistically significant difference (Kruskal-Wallis one-way analysis of variance on ranks H=9,961 with 2 degrees of freedom with P=0,007) between control, 1.5% concentration and 2% concentration samples. All pair wise multiple comparison was performed by Tukey test also showed that test samples with 2% concentration are statistically different from test samples with 1.5% concentration (q=3,977; P<0,05) and control (q=3,365; P<0,05) samples.

Results of exposure studies against White rot fungi *T. hirsute* also showed that there is a statistically significant difference (Kruskal-Wallis one-way analysis of variance on ranks H=7, 666 with 2 degrees of freedom with P=0,002) between control, 1.5% concentration and 2% concentration samples. All pair wise multiple comparison was performed by Tukey test also showed that test samples with 1.5% concentration are statistically different from control (q=3, 862; $P<0.05$) samples but not different from 2% concentration (q=1, 759; $P<0.05$) samples.

CONCLUSION

From the experiments, we can conclude that botanical extractives alone were not imparting any resistance to wood destroying organisms. Even the modified preservative formulation formed using botanicals was found to be ineffective to provide protection for the desirable time line. Additional to this the problem faced with bonding of panel when the prepared preservative formulation was incorporated in glue line. Although the idea of eco-friendly, botanical wood preservative looks lucrative, the challenges are many starting from getting uniformity in the extractive content across the season and region to the shelf life of the extractive itself. Hence it may be wise to use only silica, lignin and copper sulphate based mixture as a wood preservative formulation similar to Copper Chrome Boron (CCB) and in this line we are continuing our research. Even from the studies conducted at initial stages, we found that botanicals extractives alone were not imparted any kind of protection to the wood it is more likely that protection that was achieved for 24 months was mainly due to copper sulphate and other materials incorporated. Moreover, different forms of silica are known to impart fire retardant properties to panel product in that line also study needs to be carried out. If we could achieve wood preservative with fire retardant properties, it will be a great beginning to start with.

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