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DECAY RESISTANCE AND COLOR STABILITY OF WOOD TREATED WITH *JUGLANS REGIA* EXTRACT

Osman Goktas, Ergun Baysal, Ertan Ozen
Department of Wood Science andTechnology, Faculty of Technology,
Mugla University, Mugla, Turkey

Ramazan Mammadov
Department of Biology, Faculty of Arts and Sciences, Mugla University,
Mugla, Turkey

M. Emin Duru
Department of Chemistry, Faculty of Arts and Sciences, Mugla University, Mugla,
Turkey

ABSTRACT

This study was designed to determine antifungal activities and color stability under ultraviolet (UV) exposure of wood treated with aqueous solutions of *Juglans regia* extract. The extract was prepared from *Juglans regia*'s shells skins in 96 % ethyl alcohol. Then, its aqueous solutions were prepared as % 0.125, 0.25 %, 0.50 %, and 1.50 %. Extract impregnated into wood blocks of beech (*Fagus Orientalis* L.*)* and Scots pine (*Pinus sylvestris* L.) according to ASTM D 1413–76. Treated blocks were exposed to *Postia plesenta* and *Trametes versicolour* attack for 3 months by the soil block method using the procedure set out in ASTM D 1413-76. Determination of color stability of wood specimens treated with aqueous solutions of *Juglans regia* extract, they were exposed to UV lightfastness test for 500, 1000, and 1500 hours exposure.

Results showed that mass losses for all treated wood specimens decreased in some extent confirming the effectiveness of extract solution in enhancing decay resistance. According to decay test results, mass loss of both wood specimens treated with % 0.5 aqueous solution of *Juglans regia* was the lowest for both decay fungi after 3 months decay exposure.

Lightness values of both of wood specimens silightly changed as a result of 500, 1000, and 1500 lightfastness exposure. It was observed that color stability of beech wood was higher than untreated Scots pine wood after 500, 1000 and 1500 h exposure. The best color stability for both of wood specimens were obtained treated with madder without mordant solution and a mixture of madder and iron, respectively.

KEY WORDS: *Juglans regia* extract, decay resistance, color stability, beech, Scots pine, lightfastness test

INTRODUCTION

Throughout the course of history wood has remained one of the most important renewable natural resources available to man. It is-a natural, cellular, renewable resource, has excellent strength-to-weight properties, a relatively low price and is easily produced composite material of botanical origin-possesses unique structural and chemical characteristics that render it desirable for broad variety of end uses (Hingston et al. 2001). However, as a naturel organic material wood is degraded by many organisms, principally fungi and insects (Schultz and Nicholas 2002). So, it is generally treated with a chemical preservative to prevent damage by these aggressive biodeteriogens (Craiig et al. 2001).

A large number of preservative compounds have beeen introduced on to the market but many of them not gained acceptance either because of chemical toxicity, low efficacy, high cost, or corrosiveness (Murphy 1990). Since certain wood preservatives have been banned or limited for some applications such as chromated copper arsenate (CCA) in some European countries, the United States, and Japan (Kartal et al. 2004). Penthachlorophenol (PCP) and many biocides were also prohibited at many European countries long time ago due to their detrimental effect on the natural balance and human health (Bozkurt et al. 1992). Therefore, in recent years, Wood Preservation Industry prefers non-chemical based and vegetable based chemicals for wood protection. Since some natural extractives contain tannin or have toxic effects against biotic agents, they could be preferred in use for protection of wood or wood based objects against destroying organisms (Bozkurt and Goker 1986, Schulta and Nicholas 2000, Temiz 2000).

Chang et al. (1998) reported antifungal activities of α-cadinol, α-cedrol, hinokiol, sugiol, ferruginol, helioxanthin, savinin, and taiwanin C that were isolated from Taiwania heartwood. Among all this, α-cadinol has demonstrated to possess the highest antifungal effectiveness. Konodo and Imamura (1986) had also investigated the antifungal compounds in heartwood extractives of *Chamaecyparis obtusa* using gas-liquid chromatography analysis; they deduced that the main antifungal compounds of Chamaecyparis *obtusa* were cadinane skeletal sesquiterpenoids.

Digrak et al. (1999) investigated the antimicrobial activities of valex, the extracts of mimosa bark, gallnut powders, *Salvia aucheri* Bentham var. *aucheri* and *Phlomis bourgei* Boiss. The results indicated that mimosa bark extracts had the greatest antibacterial activity. Tang et al. (2006) reported that the ethanolic extracts from the bark of *Accacia confuse* exhibited strong antioxidant activity. Schultz and Nicholas (2002) reported that heartwood extractives may be alternative wood preservatives as they have fungicidal and antioxidant properties. Kartal et al. (2006) investigated the activities of various essential oils and extracts from plants against wood decay fungi. They found that the formulations with cinnamic acid, ferulic acid, and cinnamaldehyde showed strong antifungal activities. Sequalene and wood tar oil containing formulations were also effective against wood degrading fungi in the test.

Wood surfaces exposed outdoors are rapidly degraded because lignin strongly absorbs UV light, which leads to radical-induced depolymerisation of lignin and cellulose, the major structural constituents of wood (Evans et al. 2002). The ultraviolet (UV) light portion of the solar radiation and the presence of moisture are the main causes for the weathering degradation of wood (Feist and Rowell 1982, Denes and Young 1999). Changes in chemical, physical, and optical properties of wood lead to discolaration, loss of gloss, roughening of surface, and are also accompanied by alteration of mechanical properties of the three main components of wood-cellulose, hemicelluloses, and lignin appears to be oxidized and degraded by UV light more rapidly (Denes and Young 1999). The rate of degradation is increased by water (rain, dew, and snow), changes in relative humidity, increased temperature, and windblown sand and/or other particulates. Attack by decay fungi is not considered weathering, nor is mildew growth on the wood surface, which usually accompanies weathering. Weathering of wood is primarily a surface phenomenon that results in the slow erosion of wood fibers from the surface. The chromated copper arsenate (CCA)

impregnation can greatly extend the life time and durability of partially UV- light-transparent stain applied to the treated wood surface because the chromium stabilized the wood surface against UV light degradation. In addition to improving the durability of finishes on wood substrates, chromium salts impart other benefical properties to wood surfaces, such as fungal resistance, decreased swelling by water, increased water repellency (Tshabalala and Ganstad 2003). Since environmental awareness has forced the use of environmentally safe and arsenic free chemicals for wood and wood based composite protection (Evans 1995, Suzuki 1995), alternatives to arsenic containing preservatives are required.

Therefore, the most effective method of preventing the photodegradation of wood involves treatment with dilute aqueous solutions of inorganic salts (Kiguchi and Evans 1998) but, most of the inorganic treatments are toxic and strongly stain the wood, resulting in an unattractive appearence (Denes and Young 1999). So, alternative technologies are needed that are as effective as chromating technologies in enhancing the durability of wood products (Tshabalala and Gangstad 2003). Notwithstanding some experimantal developments like acetylation of wood (Mitsui and Tolvaj 2005), plasma-polymer coating (Agnes and Young 1999), using grafted photostabilisers (Kiguchi and Evans, 1998), benzoylation of wood (Evans et al. 2002), coating of wood surfaces by the sol-gel process with a combination of methyltrimethoxysilane and hexadecyltrimethoxysilane (Tshabalala and Gangstad 2003). etc.

The objective of the present study was to investigate the decay resistance and color stability under UV light irradiation of wood treated with aqueous solutions of *Juglans regia* extract.

MATERIAL AND METHODS

Plant material

The *Juglans regia* used in this study were collected from the region of Mugla-Turkey. *Juglans regia*'s shells skins collected were authenticated according to conventional method (Davis et al. 1965). Dried and powdered shells skins of *Juglans regia* (100 g) was refluxed with 4000 ml water. After 3 hours, the suspension was passed over a Buchner filter, and a sample of 500 ml of the filtrate was taken. The remainder of the filtrate was put into freeze dryer in order to dry under reduced pressure.

Preperation of Juglans regia extract

Juglans regia's shells skins blended into 1-2 mm size and additioned 100 ml ethyl alcohol for each 10 g then placed into alcohol bath at ca 50 °C for five h. The extract was filtered in a glass funnel fitted with glass wool and the funnel and flask rinsed with a small quantity (about 30 ml) of fresh ethyl alchol. The extract solution was evaporated to constant weight in a vacuum oven (rotary evaporator) at ca 50°C. Then extract was diluted by distiled water and freezed in the deep freeze, then succed this distiled water by freeze dryer to seperate from essence of extract. Remained extract was protected in deep fereeze at -20°C for after usages.

Dying procedures

In the experiments, $FeSO_4 7H_2O$ (technical grade 96% purity, Merck) and alum $KAl(SO_4)_2.12H_2O$ (puriss. p.a. Fluka), were mixed as concentrated solution with the addition of mordant (3 % and 5 %) to give a final dyebath concentration of 30 g L^{-1} and 50 g L^{-1} mordant (Trotman 1984, Guzel ve Akgerman 2000, Wickens 1983).

Prepared dyestuff have been seperated into three containers, and then heated to 60 °C. The wood panels were immersed into the dyebath containing the dyestuff extract for 30 min. Extra solution which was left on the specimens were sweeped with a clean cloth. Afterwards, specimens were left to dry under the teperature of 20±3 ^{0}C on a vertical position.

Treatment method

The test specimens were impregnated with aqueos solutions such as 0.125 %, 0.25 %, 0.50 %, and 1.50 % of *Juglans regia* extract according to ASTM D 1413-76. Treatment solutions were prepared the day before the impregnation for homogenizing. A vacuum desiccator used for the impregnation process was connected to a vacuum pump through a vacuum trap. Wood specimens were subjected to 30 min of vacuum at 760 $mmHg^{-1}$ followed by 30 min of diffusion in the treatment solutions. Weight percent gain (WPG) (%, w/w) due to chemical load was calculated from the fallowing equation:

$$WPG = \frac{W_{of} - W_{o1}}{W_{o1}} \quad (\%)$$

Where; W_{o1} is the oven-dry weight (g) of a wood specimen before impregnation, and W_{o1} is the final oven-dry weight (g) of a wood specimen after impregnation.

Decay test

Wood specimens measuring 19 (tangential) x 19 (radial) x 19 (longitudinal) mm were prepared for decay test, from air dried sapwood of beech (*Fagus orientalis* L.) and Scots pine (*Pinus sylvestris* L.) which met the reguirements of ASTM D 1413- 76 (1976).

Prepared culture boottles filled with 120 cm^3 of soil mixture and 62 g distilled water were added to each bottle. This quatitive of added water was calculated according to "Water Hold Capacity" and "Water required" that clarificated in ASTM D 1413-76. Bottles were sterilized in an autoclave for 30 min. at 121°C. After the sterilized soil culture bottles were throughly cooled, approximately cutted 10-mm square fungus inoculum sections from a petri dish culture, and placed the square of inoculum in contact with an edge of the feeder strip on the soil. Inoculated with the fungal species, and incubated at 27 °C and 72.0 % relative humidity for 3 weeks. Then, wood specimens were subjected to a modified decay resistance test. Five replicate specimens of each wood type were dried to constant weight and steam-sterilized at 100 ± 2°C for 20 min. After cooling, wood specimens were placed in the cultur bottles under air laminar flow condition to avoid contamination. Screwed bootles cap loosen one-quarter turn. Then, exposed to *Postia plesenta* (Fries) M. Larsen et Lombard (Mad 698), (a brown-rot) and *Trametes versicolour* (L.ex Fr.) Quel. (FFPRI 1030) rown-rot (a white-rot) fungi, in a modified soil-block test according to ASTM D 1413-76 test method for solid wood. For incubation period for 3 months, blocks holded at temperature 27°C (81 °F) and a relative humidity (RH) 75 %. At the end of the incubation period, blocks were removed from the test bottles and the mycellium was carefully brushed off the samples. Tested specimens were then reweighted after 4 weeks seasoning, to reach relative humiditiy, in the open laboratory. Mass loss was calculated from the conditioned weight of the wood specimen immeditely before and after testing, as follows:

$$\text{Mass loss} = (100\,(T_2 - T_1)\,/\,T_1) \quad (\%)$$

Where;

T_1 = weight of wood specimen plus remaining preservative after condioning and before exposure to the test fungus,

T_2 = weight of the wood specimen after test and after final conditioning.

Lightfastness test

Samples of beech (*Fagus orientalis* Lipsky) and Scots pine (*Pinus sylvestris* L.) sapwood were used in this study. Specimens measuring 10mm (radial) x 100mm (tangential) x 150 (longitudinal) mm

were cut and stored in the laboratory at 20˚C ± 2˚C and % 65 ± %5 relative humidity in order to reach equilibrum moisture content.

Lightfastness test was made in a Q-panel QUV weathering tester equipped with UVA–351 lamps. The temperature of tester was 60 ± 2 ˚C. The wood specimens were exposed to UV light directly at a distance of 5 cm. The total exposure times ranged from 0 to 1500 h. (3 periods x 500 h). The color of the samples was measured after each irradiation period. Five replicates were made for both wood species.

Color changes

The color of the specimen was measured by a colorimeter (Minolta, type CR-10; light type D65; filter type silicon photocell). The measuring spot was adjusted to be equal or not more than one-third of the distance from the center of this area to the receptor field stops. The color difference, *(ΔE*)* was determined for each wood as follows (ASTM D 1536–58 T 1984):

$\Delta a^* = a^*_{f-} a^*_i$
$\Delta b^* = b^*_f - b^*_i$
$\Delta L^* = L_f - L^*_i$

$(\Delta E^*) = [(\Delta a^*)^2 + (\Delta b^*)^2 + (\Delta L^*)^2]^{1/2}$

Where Δa^*, Δb^*, *and* ΔL^* are the changes between the initial and final intervals values.

RESULTS AND DISCUSSION

Weight percent gain and mass loss

Higher retentions were achived in Scots pine because of the easy penetrability of these species compared to beech wood. Weight percent gain was found to be between 0.13 and 2.08 % for both of wood specimens. The highest WPG were obtained treated with 1.50 % aqueous solutions of Scots pine with 2.08 %. Results showed that the higher concentration levels of treatment solutions the higher extract content was obtained in wood.

Percentage mass loss of the both wood specimens caused by brown rot fungus, *Postia placenta* and white rot fungus, *Trametes versicolor* are given in Tab. 1. The mass loss of Scots pine specimens treated with aqueous solutions of *Juglans regia* extract were higher than beech specimens treated with aqueous solutions of *Juglans regia* extract after 3 months by *Postia placenta* exposure. Untreated both wood specimens were severely attacked by *Postia plesenta* with the large mass loss, but the values of mass losses for all treated specimens decreased in some extent confirming the effectiveness of extract solution in enhancing decay resistance. Mass loss of both wood specimens treated with % 0.5 aqueous solution of *Juglans regia* was the lowest for both decay fungi after 3 months decay exposure. Therefore, concentration level of *Juglans regia* extract against the types of decay fungi appeared to be around % 0.50 for both wood specimens. Decay test results showed that both wood specimens impregnated by aqueous solutions of *Juglans regia* extract at concentrate levels of 0.125 %, 0.25 %, 0.50 %, and 1.50 % were effective in suppressing the attack of *Postia plesenta.* But, it was not observed for both wood specimens treated with same extract solutions and exposure to *Trametes versicolor* attacks. Because, except for % 0.50 concentration levels of *Juglans regia*, mass loss of untreated both wood specimens treated with 0.125 %, 0.25 %, and 1.50 % aqueous solution of *Juglans regia* extract were lower than untreated both wood specimens. In our study, we obtained that 1.50 % concentration of extract resulted in higher mass loss compared to 0.50 % concentration level. This was probably due to decreased saponin derivates and alcoloits of the extract which losses its toxic effect against fungal decay (Sen et al. 2002).

Generally, while *Postia placenta* caused higher mass loss for beech, *Trametes versicolor* caused higher mass loss for Scots pine.

Tab. 1: WPG and mass loss of beech and Scots pine treated with Juglans regia's shells skins after decay test

Wood species	Concentration (%)	WPG (%)	Mass loss* (%)	
			Postia placenta Mean ± SD	*Trametes versicolor* Mean ± SD
Beech	Control	-	27.37 ± 3.36	21.54 ± 4.78
	0.125	0.13	20.79 ± 4.11	31.92± 2.61
	0.250	0.32	13.75 ± 1.21	30.89 ± 3.69
	0.500	0.86	9.05 ± 2.36	20.59 ± 3.13
	1.500	1.83	14.59 ± 2.70	30.16 ± 3.95
Scots pine	Control	-	30.66 ± 4.00	8.64 ± 1.71
	0.125	0.16	20.83 ± 3.54	9.55± 1.43
	0.250	0.37	22.02 ± 0.66	11.25 ± 3.29
	0.500	1.27	13.27 ± 4.09	6.91 ± 1.83
	1.500	2.08	15.03 ± 3.64	14.46± 1.63

** Five replicates were tested for each decay fungus.*
SD: Standard deviation

Color stability

Tab. 2 shows the color differences as ΔE^*, ΔL^*, Δa^*, and Δb^* of beech and Scots pine wood specimens treated with aqueous solutions of *Juglans regia* extract after 500, 1000, and 1500 h lightfastness test. ΔL^* values of untreated Scots pine were higher than untreated beech wood before lightfastness exposure. The highest lightness values were obtained from untreated Scots pine wood before lightfastness exposure because of it is light color of timber. Both wood specimens exposed to UV radiation of 350 nm for 1500 h showed positive values of ΔL^*. For beech wood, while values of the ΔL^*, Δa^*, and Δb^* nearly constant during all exposure priods. For Scots pine, while the trend of the ΔL^* and Δb^* values are decreased, the value of the Δa^* is slightly increased. Lightness values of (ΔL^*) of both wood specimens treated with a mixture of *Juglans regia* and alum were higher compared to the other treament solutions before lightfastness exposure. Lightness values of both wood specimens silightly changed as a result of 500, 1000, and 1500 lightfastness exposure. We found that changes of Δa^*values of Scots pine were higher than changes Δa^* values of beech wood after 1500 hours exposure. Δb^* values of both wood scpecimens were remarkably higher compared to Δa^* values of both wood specimens before lightfastness exposure. It was observed that total color differences (ΔE^*) of untreated Scots pine wood were higher than beech wood after 500, 1000, and 1500 h exposure. The reason for the differences between two wood species were not fully understood but it may be speculated with the differences of chemical compositions of two different wood species (hardwood and softwood) results in the differences against photo-degradation effect of UV irradiation. However, the roughness of color stability wood is a complex phenomenon because wood is an unisotropic and heterogenous material. Several factors such as anatomical differences, growing characteristics, machining properties of wood, pre-treatments (e.g. steaming, drying, etc.) (Temiz et al. 2005).

Treatment of beech wood with madder without mordant solution provided the best color stability 1500 h UV exposure. For Scots pine wood, treatment of a mixture of *Juglans regia* and iron resulted in higher color stability compared to the other treatment solutions. While ΔE^* values of untreated and treated beech wood specimens were found to be between 0.12 and 2.72. ΔE^* values of untreated and treated Scots pine wood specimens were found to be between 1.03 and 9.29. Among these treatment, Scots pine wood treated with a mixture of *Juglans regia* and alum was particularly susceptible discolaration (ΔE^*= 8.47). While, the total color difference of untreated beech wood was slightly increased during the all exposure periods, it was sharply increased during all exposure periods for Scots pine. This color differences was attributed to the carbonyl groups of conjugated ketones, aldehydes, and quinies resulting from the modification of lignin and related compounds. The values of Δa^* and Δb^* for both treated and untreated wood specimens suggested that the more yellowish color than reddish color resulting after lightfastness test.

The general increase in the chromaticity coordinates Δa^* and Δb^* for both wood species confirms the modification of the wood chromophoric groups. Lignin is the wood component with chromophoric groups capable of absorbing UV light in the ranging from 300 to 550 nm (Davidson 1986). Wood ion complexes formed at the wood surfaces possibly provide resistance to wood surface by blocking the free phenolic groups, which are the reactive sites of photochemical reactions (Temiz et al. 2005).

Tab. 2: Color stability of beech and Scots pine after lightfastness test

Wood species	Treatment	Before lightastness test			After 500 hours				After 1000 hours				After 1500 hours			
		ΔL^*	Δa^*	Δb^*	ΔE^*	ΔL^*	Δa^*	Δb^*	ΔE^*	ΔL^*	Δa^*	Δb^*	ΔE^*	ΔL^*	Δa^*	Δb^*
Beech	Control	61.9	13.4	23.1	1.35	62.9	13.3	24.0	0.24	61.8	13.5	23.3	0.87	61.2	13.9	23.2
	Madder (without mordant)	46.4	10.4	18.9	0.12	49.5	10.4	18.8	0.48	49.1	10.5	18.6	0.83	48.8	10.7	18.5
	Madder and alum	70.4	11.3	25.4	1.37	69.2	12.0	25.4	2.72	68.1	12.4	24.3	2.55	68.2	12.0	24.5
	Madder and iron	65.5	11.1	21.4	0.26	65.8	11.3	21.6	1.79	64.1	11.4	20.6	1.96	63.9	11.6	20.8
Scots pine	Control	78.8	8.7	26.9	3.07	76.4	10.4	26.3	5.70	74.2	11.9	25.8	9.29	70.9	13.4	25.7
	Madder (without mordant)	66.8	8.8	24.1	1.71	64.7	9.8	23.6	3.46	63.3	10.7	23.2	5.58	61.3	11.6	23.0
	Madder and alum	75.5	8.2	27.3	6.90	70.1	12.3	26.0	7.91	69.5	13.4	27.2	8.47	68.6	12.9	26.1
	Madder and iron	36.2	4.1	11.8	1.03	35.3	4.6	12.1	2.25	37.9	5.4	12.4	2.30	38.4	4.3	12.2

CONCLUSION

Decay resistance and color stability of wood specimens treated with aqueous solutions of *Juglans regia* extract were studied.

Results showed that *Postia placenta* caused higher mass loss for Scots pine than beech wood after 3 monts of decay exposure. Mass losses of both wood specimens were lower in some extent compared to untreated both wood specimens. The most effective dosage of *Juglans regia* extract was 0.5 %. All extract dosages of *Juglans regia* were found efficacious in suppressing attack of *Postia placenta.* But, it was not observed for both wood specimens treated with same extract solutions and exposure to *Trametes versicolor* attacks. While *Postia placenta* caused higher mass loss for beech, *Trametes versicolor* caused higher mass loss for Scots pine. The total color difference of treated beech wood was lower than treated Scots pine after 1500 h lightfastness exposure.

We observed that ΔL^* values of untreated Scots pine were higher than untreated beech wood before lightfastness exposure. Lightness values of of both wood specimens treated with a mixture of

Juglans regia and alum gave higher values compared to the other treament solutions before lightfastness exposure. While beech wood treated with madder without mordant solution provided the best color stability, Scots pine wood, treated with a mixture of *Juglans regia* and iron showed the best color stability. The values of Δa^* and Δb^* for both treated and untreated wood specimens suggested that the more yellowish color than reddish color resulting from irridiation.

In conclusion, both wood species treated with aqueous solutions of *Juglans regia* extract showed good decay resistance compared to untreated of both wood species. Color stability of wood specimens treated with aqueous solutions yielded higher color stability in some extent.

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Osman Goktas
Corresponding author
Department of Wood Science and Technology
Faculty of Technology
Mugla University
Mugla 48000
Turkey
Phone: +90 252 2111709
Fax: +90 252 223 8511
E-mail: ergun69@yahoo.com, ogoktas@mu.edu.tr

Ergun Baysal
Department of Wood Science and Technology
Faculty of Technology
Mugla University
Mugla 48000
Turkey

Ertan Ozen
Department of Wood Science and Technology
Faculty of Technology
Mugla University
Mugla 48000
Turkey
E-mail: eozen@mu.edu.tr

Ramazan Mammadov
Department of Biology
Faculty of Arts and Sciences
Mugla University
Mugla 48000
Turkey
E-mail: rmammad@yahoo.com

M. Emin Duru
Department of Chemistry
Faculty of Arts and Sciences
Mugla University
Mugla 48000
Turkey
E-mail: eminduru@hotmail.com