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FIRE RESISTANCE PERFORMANCE OF WOOD MATERIALS COLORED WITH ECO-FRIENDLY POMEGRANATE SKIN (*PUNICA GRANATUM*) EXTRACTS

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ABSTRACT

The main goal of this study was to determine fire resistance properties of wood treated with pomegranate extract and mordant mixes. According to that wood materials Scotch pine (*Pinus sylvestris* L.), Oriental beech (*Fagus orientalis* Lipsky) were chosen. Aluminum sulphate ($KAl_2(SO_4)_3.18H_2O$) copper sulphate ($CuSO_2.5H_2O$) and vinegar were used as mordant agent and a synthetic dye was used for comparison. Ultrasonic assisted method were used for extractionthe plant dyestuff from pomegranate skin (*Punica granatum*) and then applied to wood blocks by immersion (classic) and immersion ultrasonic assisted methods. The combustion test was realized according to ASTM-E 69-02 (2002) standard. The mass losses release of gasses (CO, O_2) and the temperature differences of samples were detected for each 30 seconds during combustion. The results showed that the aluminum sulphate mixes were showed the best results on all tests. Unfortunately test performances are not enough to retard the fire effect on the wood materials. Eco-friendly natural colorant might be developed to use them as fire retardant.

KEYWORDS: Natural colorant, pomegranate, fire resistance, combustion, beech wood, pine wood.

INTRODUCTION

Wood material has unique properties and structural characteristic, hence are irreplaceable for decoration and furniture. On the other part due to organic structure, it needs to protect against water absorption, pests, fungal effects and inflammableness properties. Paint, varnish, and wood preservatives are often used to prevent these negative effects on wood material. However, during the preservation of wood by chemical means and in terms of environmental health, drawbacks have emerged in recent years (Kurtoglu 1988). Typically, protection for wood must have toxic effects against pests. Exposure to contamination indoors causes adverse effects on human health, and so this has become a subject of careful scrutiny for society. There has been a recent increase in the use of natural dyes in cloth, wool, cotton, food, arıd cosmetic products, and various studies have been made on dyeing (Ozen et al. 2014a).

Recently many natural dyes were mentioned in many scientific studies. Ozen et al. (2014a) investigated the colorability of wood materials with pomegranate skin and black mulberry extracts; they reported that the extracts can be used to color wood materials. Ozen et al. (2014b) reported that the madder root extracts and mordant mixes could be an eco-friendly alternative for wood preservation. Yang and Clausen (2007, Ozen et al. 2014b) investigated antifungal effect of seven essential oils; derived-ajowan, dill weed, Egyptian geranium, lemongrass, rosemary, tea tree, and thyme, and their results indicated that these natural extracts had antibacterial and antifungal activity. Schultz and Nicholas (2002, Ozen et al. 2014b) reported that heartwood extracts have fungicidal and antioxidant properties and they could be used as wood preservatives. Tascioglu et al. (2013, Ozen et al. 2014b), reported that commercial mimosa and quebracho extracts can be utilized as alternative to impregnation chemicals against wood decay fungi in door applications. Goktas et al. (2008) investigated the antifungal activities and color stability under ultraviolet (UV) exposure of wood treated with aqueous solutions of *Juglans regia* extracts; they reported effectiveness decay resistance of the extracts. Goktas et al. (2010) investigated the antifungal property of poisonous plant extracts from *Muscari neglectum* Cuss. and *Gynandririssi syrinchium*; they reported that the extracts had antifungal effect. Baysal et al. 2007, Goktas et al. 2010) investigated fire resistance of Douglas fir treated with borates and natural extractives; they reported that natural extractives did not improve fire resistance of the samples.

From early antiquity, pomegranate has been cultivated throughout the Mediterranean and North African regions, including Central Saharan oases, for its valuable fruit. Considering the locations and context of pomegranate representations and archaeobotanical evidence, this fruit has held a long-time position as a luxury food (Bruni et al. 2011, Bosi et al. 2009). At the Villa Rustica in Oplontis, over a ton of carbonized pomegranates, dated around the time of the Roman empire, were discovered (Bruni et al. 2011, Jashemski and Meyer 2002). Several parts of the plant were used as both a tanning agent and dye. The dried fruit rind yields a yellow dye which can be used for dyeing clothes, making a hair dye, or even as a mordant (Bruni et al. 2011, Dastur 1964).

In this study colorants extracted from pomegranate skin (*Punica granatum*) were used as colorant for wood materials. The objectives of this study were to develop an eco-friendly, natural wood dye extracted from pomegranate (*Punica granatum*) instead of synthetic dyes and to determine its fire resistance performance.

MATERIAL AND METHODS

Wood specimens were cut from randomly selected first grade oriental beech (*Fagus orientalis* Lipsky), Scotch pine (*Pinus sylvestris* L.), in conformity with the ASTM-E69-02 (2002) standards in sample size (9.5x19x1016 mm) (longitudinal x radial x tangential directions). All specimens were oven-dried temperature of 103±2°C before and after treatment. Aluminum sulphate ($KAl_2(SO_4)_3.18H_2O$) and copper sulphate ($CuSO_2.5H_2O$) were supplied from Kimetsan Co. and vinegar was purchased from Fersan Co.. The synthetic dye used to compare natural and synhetic; woodtex – wood colorant was provided from Kayalarkimya Co.

Preparation of dyestuff and mordant mixes

The Pomegranate skin (*Punica granatum*) was obtained from region Hatay, Turkey. Dried pomegranate skin material was extracted with demineralized water in an ultrasonic bath (Elmasonic X-tra 150 H). Ultrasound-assisted extraction was considered as an efficient method for extracting bioactive compounds from *Solvia officinalis* (Salisova et al. 1997) and *Hibiscus tiliaceus* L. flowers (Melecchi et al. 2002), antioxidants from *Rosmarinus officinalis* (Albu et al. 2004), and triterpenoids and steroids from *Chresta* spp. (Schinor et al. 2004). The use of ultrasound as an adjunct to conventional extraction provides qualitatively acceptable tools from *Amaranthus caudatus* seeds but much more economically, more quickly and using equipment commonly available (Bruni et al. 2002, Wang and Weller 2006).

Pomegranate skin extracts dissolved distilled water to concentration 5 % which is the suggested ratio of wood protection against biologic destructive organisms (Yeniocak 2013). Extraction performed in an ultrasonic bath depends on parameters 180 min. time, 45°C temperature and 180 W sonic powers. At the end of extraction water loss owing to evaporation was added into the extract.

Aqueous solutions of pomegranate skin extracts were mordanted by admix aluminum sulphate ($KAl_2(SO_4)_3.18H_2O$) 5 %, copper sulphate ($CuSO_2.5H_2O$) 5 %, and grape vinegar 10 % in order to stabilize the color of dyes extracted, to ensure it to hang on the applied material (to increase retention amount), and to create color options (Ozen et al. 2014b).

Treatment procedure

The test specimens were treated with pomegranate skin extracts and mordant mixes. In the treatment ultrasonic-assisted dipping and conventional dipping method were used. Treatment processes was performed in two methods classic and ultrasonic-assisted dipping (300 W sonic power) in time 60 minutes and 45ºC temperature. The weight percent gain (WPG) (%,) due to immersion load was calculated from the following Eq. (Ozen et al. 2014b) :

$$WPG = \frac{W_{of} - W_{oi}}{W_{oi}} \times 100 \qquad (\%)$$

where: W_{oi} - oven-dried weight (g) of a wood sample before dipping,
W_{of} - oven-dried weight (g) of a wood sample after dipping.

After impregnation wood samples were conditioned under 20 ± 2°C and 65 ±3 % relative humidity until they reached aninvariable weight before the fire resistance tests (Ozen et al. 2014b).

Implementation of test

The fire resistance test was performed according to the ASTM E-69-02 (2002) method. In order to measure temperature variation and concentration of the released gasses (O_2, CO),

Testo 350 M and XL flue gas analyzers were used. The probe was inserted into the first hole from the top of the fire tube (Yapıcı et al. 2011).

Statistical analysis

To specify the effects of natural extract sand mordant mixes on combustion multi-variance analysis and for pair wise comparisons Duncan test's was practiced.

RESULTS AND DISCUSSION

Means extracts retentions, computed depends on gross retention data and concentration, of impregnated wood specimens were given in Tab. 1. When wood species which impregnated by pomegranate skin extracts compared, the highest retention were specified with beech 7.31 % on pomegranate extract and alum mixes group impregnated with ultrasonic method and lowest retention were 4.62 % on pomegranate extract and vinegar mixes group treated with classic method. According to results; retention rate of pomegranate extracts maintained at least had two times more. Heterogeneous texture, density and porosity values of wood species used play an important role on retention values (Ozen et al. 2014b, Aydin and Colakoglu 2003). On the other side particle of extracts are responsible for penetration efficiency to the retentions rate (Ozen et al. 2014a, b).

Tab. 1: Values retentions of wood species (%).

Extracts	Treatment method	Scotch pine		Beech	
		WPG (%)	St. devin.	WPG (%)	St. devin.
Control (Non mordant)	Ultrasonic	4.84	0.72	5.70	0.55
	Classic	5.31	0.89	5.33	0.03
Pomegranate + Alum	Ultrasonic	4.90	0.12	7.31	1.13
	Classic	5.10	0.12	6.62	1.01
Pomegranate + Copper	Ultrasonic	5.23	0.12	5.78	0.46
	Classic	5.58	0.58	5.60	0.17
Pomegranate + Vinegar	Ultrasonic	4.62	0.06	5.62	0.06
	Classic	5.44	1.43	5.32	0.59
Synthetic	Classic	1.70	0.08	2.42	0.17

Results of fire test

The average values based on coloration pomegranate extracts and mordant mixes are given in Tab. 2.

According to the test results (Tab. 2) mass losses rate recorded between 75.1 and 99.4 %. The highest mass losses rate observed from beech wood species (99.4 %) colored with copper sulphate mixes and treated by classic method. The lowest mass losses rate determined from Scoth pine species (75.1 %) colored with aluminum sulphate treated by ultrasonic method. The aluminum sulphate and copper sulphate has increased the combustion resistance on Scotch pine species. These results were similar with; Baysal et al. (2007 and Baysal 2002) reported that mass losses of Scotch pine wood specimens treated with aqueous solutions of the natural extractives were around 85-94 %. Also, Baysal et al. (2007) found that mass losses of Douglas fir wood species treated with aqueous solution of pine bark powder, acorn powder, sumac leaf powder and gall-nut powder were 85.20, 94.29, 90.72 and 90.97 % respectively.

As a result of combustion test the reduction of O_2 concentration was measured between 20.4 and 20.9 %. Reduction of O_2 was not showed any important modification depending on mordants, wood species or treatment method. The proportion of oxygen in air is normally 21 %. Pomegranate and mordant mixes were shown to be not effective as fire retardants.

Tab. 2: The average values of combustion test.

Extracts	Wood type	Treatment method	Temp. (°C)	CO (ppm)	O_2 (%)	Mass losses rate (%)
Control (Non mordant)	Scotch pine	Ultrasonic	610.0 (3.95)	450.5 (3.59)	20.9 (0.02)	94.7 (1.08)
		Classic	579.0 (3.11)	452.0 (6.62)	20.9 (0.01)	97.4 (0.87)
	beech	Ultrasonic	605.5 (6.77)	763.0(3.85)	20.5 (0.05)	98.1 (0.49)
		Classic	671.0 (4.15)	707.0 (5.80)	20.4 (0.02)	95.5 (0.68)
Pomegranate + alum	Scotch pine	Ultrasonic	598.5 (2.44)	280.5 (5.22)	20.9 (0.04)	75.1 (1.29)
		Classic	620.0 (3.25)	288.0 (6.55)	20.9 (0.00)	77.9 (1.73)
	beech	Ultrasonic	529.0 (6.60)	485.5 (4.89)	20.6 (0.04)	98.9 (0.72)
		Classic	647.0 (5.77)	592.5 (3.27)	20.6 (0.00)	95.5 (0.36)
Pomegranate + copper	Scotch pine	Ultrasonic	574.5 (3.37)	526.0 (6.96)	20.9 (0.07)	79.8 (0.81)
		Classic	599.5 (4.92)	517.5 (8.35)	20.8 (0.04)	83.8 (1.64)
	beech	Ultrasonic	650.0 (5.27)	906.5 (2.13)	20.4 (0.05)	95.1 (0.86)
		Classic	645.5 (8.66)	824.0 (2.55)	20.6 (0.00)	99.4 (0.36)
Pomegranate + vinegar	Scotch pine	Ultrasonic	627.5 (6.70)	472.5 (8.29)	20.8 (0.01)	84.7 (1.05)
		Classic	566.5 (4.57)	459.0 (7.71)	20.9 (0.00)	80.8 (1.48)
	beech	Ultrasonic	676.5 (7.32	741.0 (2.24)	20.5 (0.06)	98.8 (0.30)
		Classic	692.5 (4.45)	884.0 (3.08)	20.5 (0.01)	98.7 (0.44)
Synthetic	Scotch pine	Classic	689.0 (2.47)	712.5 (9.81)	20.9 (0.04)	99.2 (0.24)
	beech		711.5 (3.67)	644.5 (5.19)	20.9 (0.01)	98.5 (0.01)

(Mean of 5 replicates, numbers in parenthesis are standard deviations).

According to Tab. 3, the highest temperature value (692.5°C) was observed in beech wood species colored with pomegranate + vinegar mixes treated by classic method. The lowest value (529.0°C) was found in beech wood samples with pomegranate + alum mixes treated by classic method. Pomegranate extract might be potential ignition effect in combustion. Because pomegranate extractives have high heat release when used alone on both of wood species. These values showed similarity with previous studies.

Result showed that; mordant mixes and control group showed lesser temperature value compared to synthetic. It was seen that pomegranate extract and mordant mixes decreased the temperature on combustion compared to that of synthetic dye samples.

The highest value of CO concentration (906.5 ppm) was observed in beech wood samples colored with pomegranate + alum mixes; the lowest value of CO concentration was determined in Scotch pine wood with applied pomegranate + copper mixes (280.5 ppm) treated by ultrasonic method. The results are shown as different homogenous groups in Tab. 3. The results linked with these values are shown in Figs. 1 and 2.

Tab. 3: Duncan test results (p ≤ 0.05).

Wood Type	Extracts	Temp. (°C)		CO (ppm)		O_2 (%)		Mass losses rate (%)	
		Mean	HG	Mean	HG	Mean	HG	Mean	HG
Scoth Pine	Control (Non mordant)	594.50	a	451.25	bc	20.94	b	96.08	de
	Pomegranate + Alum	609.25	b	284.25	a	20.97	b	76.54	a
	Pomegranate + Copper	587.00	a	521.75	cd	20.90	b	81.83	b
	Pomegranate + Vinegar	597.00	a	435.75	b	20.90	b	82.81	bc
	Synthetic	689.00	d	712.50	g	20.92	b	99.26	gh
Beech	Control (Non mordant)	638.25	c	735.00	gh	20.52	a	96.84	de
	Pomegranate + Alum	588.00	a	539.00	cd	20.60	a	97.25	ef
	Pomegranate + Copper	647.75	c	865.25	i	20.51	a	97.34	ef
	Pomegranate + Vinegar	684.50	d	812.50	ı	20.55	a	98.79	fg
	Synthetic	711.50	e	644.50	f	20.91	b	98.57	fg

Multi variance analyze was applied for to determined which factor were effect the test results. Multi variance analyzes of mass losses, CO, temperature and O_2 are given in Tabs. 4, 5, 6 and 7 respectively.

The effects of mordant agent, wood species, interaction of wood species - mordant agent, on mass losses data were evaluated for combustion and specified statistically significant (Tab. 4). The lower mass losses obtained from pomegranate + aluminum sulphate mixes on the Scotch pine species.

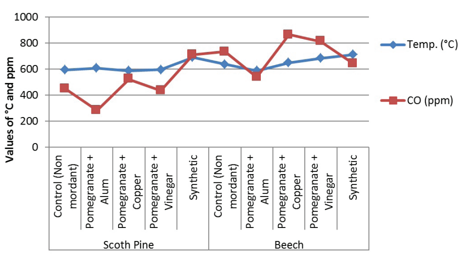

Fig. 1: Average values of temperature and Co.

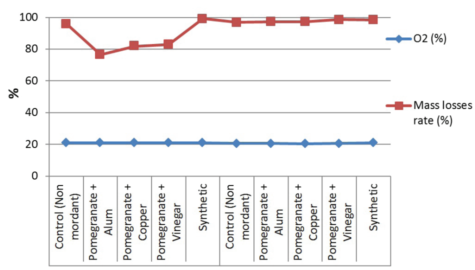

Fig. 2: Average values of O2 and mass losses rate.

Tab. 4: Multi variance analyze of mass losses.

Factors	Sum of squares	Degrees of of freedom	Mean square	F-value	P-value*
A: Wood species	2805.55	1	2805.55	129.69	0.000*
B: Mordant agent	778.58	3	259.52	11.99	0.000*
C: Treatment method	3.77	1	3.773	0.17	0.678^{NS}
Interaction A*B	896.89	3	298.96	13.82	0.000*
Interaction A*C	13.48	1	13.48	0.62	0.433^{NS}
Interaction B*C	81.36	3	27.12	1.25	0.300^{NS}
Interaction A*B*C	68.35	3	22.78	1.05	0.377^{NS}

Error	1038.34	48	21.63		
Total	534931.69	64			

NS: not significant.

Tab. 5: Multi variance analyze of CO.

Factors	Sum of squares	Degrees of freedom	Mean square	F-value	P-value*
A: Wood species	1509826.56	1	1509826.56	364.58	0.000*
B: Mordant agent	931960.59	3	310653.53	75.01	0.000*
C: Treatment method	2510.06	2	1255.03	0.30	0.739^{NS}
Interaction A*B	24731.18	3	8243.72	1.99	0.123^{NS}
Interaction A*C	3875.06	1	3875.06	0.93	0.336^{NS}
Interaction B*C	39247.68	6	6541.28	1.57	0.165^{NS}
Interaction A*B*C	39299.68	3	13099.89	3.163	0.029^{NS}
Error	298167.00	72	4141.20		
Total	30676710.00	96			

NS: not significant.

The effects of mordant agent and wood species on CO data for combustion found to be statistically significant (Tab. 5).

Highest increase value of CO concentration evaluated in the experiment of pomegranate + copper sulphate mixes on beech wood samples. Lowest value of CO concentration was founded in Scotch pine species that had been treated with pomegranate + aluminum sulphate mixes.

Tab. 6: Multi variance analyze of temperature.

Factors	Sum of squares	Degrees of freedom	Mean square	F-value	P-value*
A: Wood species	29155.56	1	29155.56	13.56	0.000*
B: Mordant agent	6396.25	3	2132.08	0.99	0.401^{NS}
C: Treatment method	6622.68	2	3311.34	1.54	0.221^{NS}
Interaction A*B	25694.18	3	8564.72	3.98	0.011^{NS}
Interaction A*C	14460.06	1	14460.06	6.72	0.011^{NS}
Interaction B*C	28011.56	6	4668.59	2.17	0.050^{NS}
Interaction A*B*C	10963.68	3	3654.56	1.69	0.174^{NS}
Error	154782.00	72	2149.75		
Total	36154160.00	96			

NS: not significant.

The effects of wood species on temperature data were evaluated for combustion and found to be statistically significant (Tab. 6).

Recent studies have shown in the first 4 minutes, the first stage of the experiment, combustion occurred in all the samples nearly at the same time (Bruni et al. 2011), as our result show. The maximum mass losses rate (76.54 %) was evaluated on Scotch pine species treated with pomegranate + aluminum sulphate mixes.

Tab. 7: Multi variance analyze of O_2.

Factors	Sum of squares	Degrees of freedom	Mean square	F-value	P-value*
A: Wood species	2.31	1	2.31	1563.42	0.000*
B: Mordant agent	0.07	3	0.02	16.65	0.000*
C: Treatment method	0.03	2	0.01	10.49	0.000*
Interaction A*B	0.01	3	0.00	2.65	0.000*
Interaction A*C	0.00	1	0.00	0.42	0.000*
Interaction B*C	0.05	6	0.00	6.57	0.000*
Interaction A*B*C	0.08	3	0.02	19.11	0.000*
Error	0.10	72	0.00		
Total	41550.20	96			

NS: not significant.

The effects of mordant agent, treatment method, wood species, and all interactions on O_2 values were observed for combustion and found to be statistically significant (Tab. 7).

CONCLUSIONS

Fire resistance properties of wood species treated with pomegranate skin and mordant mixes extracts were studied.

Overall the Scotch pine species treated with pomegranate + alum and pomegranate + copper mixes were showed highest fire resistance compared to synthetic dye.

Generally aluminum sulphate mixes were showed the best results on all tests. Pomegranate and mordant mixes were showed some fire resistance. Unfortunately, these performances are not enough to retard the fire effect on the wood materials.

In conclusion, depending on literature results natural extractives may be preferred in use for protection of wood materials against destructive organism. According to that, eco-friendly natural extractives might be developed also for use them as fire retardant instead of synthetic dyes.

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