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Combustion Properties of Calabrian Pine (*Pinus brutia* Ten.) Wood Treated with Vegetable Tanning Extracts and Boron Compounds

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Abstract: Specimens prepared from Calabrian pine (Pinus brutia Ten.) wood were treated with borate-supplemented aqueous solutions of Calabrian pine bark powder, acorn powder, sumach leaf powder and gall-nut powder. Then, fire test methods containing flame source, without flame source and glowing stages was performed according to the ASTM E 160-50 American Standard. The results showed that the lowest temperature (323 °C) for the flame source stage was recorded for specimens treated with mixtures of boric acid and borax (BA and Bx). In the without flame stage, the lowest temperature (404 °C) was recorded for specimens treated with acorn powder and (BA and Bx) mixture. In the glowing stage, the lowest temperature (107 °C) was obtained for specimens treated with sumach leaf powder and (BA and Bx) mixture. The test specimens treated with aqueous solutions of plant extracts yielded higher heat release rates for all stages. Weight loss was lowest for specimens treated with a boric acid and borax mixture (63.4%). Wood specimens treated with aqueous solutions of plant extracts yielded higher weight losses compared to the untreated control specimens. The wood specimens treated with aqueous solutions of plant extracts had a negative effect on fire parameters. However, the boric acid and borax mixture had significantly better fire retardant effectiveness over untreated and plant extracts that were applied as secondary treatments.

Key Words: Borax, Boric acid, Plant extracts, Fire parameters, Fire test

Bitkisel Sepi Maddeleri ve Borlu Bileşikler ile Muamele Edilen Kızılçam (*Pinus brutia* Ten.) Odununun Yanma Özellikleri

Özet: Bitkisel sepi maddelerinden kızılçam kabuğu, palamut meşesi, sumak yaprağı ve mazı meşesi meyvesinin tozlarının sulu çözeltileri ile muamele edilen kızılçam odunu deney örnekleri, ikincil olarak bor bileşikleri ile muamele edildikten sonra , alev kaynaklı, alev kaynaksız (kendi kendine yanma) ve kor hali yanma aşamalarını içeren ASTM E 160-50 standardında belirtilen esaslara göre, yanma deneylerine tabi tutulmuştur. Sonuç olarak; alev kaynaklı yanma aşamasında borik asit ve boraks karışımı ile muamele edilen örneklerde 323 °C, alev kaynaksız yanma aşamasında palamut meşesi tozu ve (borik asit ve boraks) karışımı ile muamele edilen örneklerde 404 °C ve kor hali yanma aşamasında sumak yaprağı tozu ve (borik asit ve boraks) ile muamele edilen örneklerde 107 °C ile en düşük sıcaklık dereceleri elde edilmiştir. Bitkisel sepi maddeleri ile muamele edilen örneklerde; alev kaynaklı, alev kaynaksız ve kor hali yanma aşamalarında yüksek sıcaklık değerleri ölçülmüştür. Yanma sonucu en düşük kütle kaybı; borik asit ve boraks karışımı ile muamele edilen örneklerde oluşmuştur (%63.4). Bitkisel sepi maddeleriyle muamele edilen örneklerde ağırlık kaybı oranları , muamelesiz kontrol örneklerine oranla, daha yüksek düzeyde gerçekleşmiştir. Bitkisel sepi maddeleri, genellikle yanma ile ilgili tüm parametreleri olumsuz yönde etkilemişlerdir. Bununla birlikte borlu bileşikler, bireysel olarak kullanımlarında ve bitkisel sepi maddeleri üzerine ikincil olarak uygulanmaları durumunda, yanma ile ilgili tüm parametreleri olumlu yönde iyileştirmiştir.

Anahtar Sözcükler: Boraks, Borik asit, Bitkisel sepi maddeleri, Yanma parametreleri, Yanma deneyi

Introduction

Wood and wood-based materials are mainly composed of carbon and hydrogen. For this reason, they are combustible (Ching-Mu and Wang, 1991). When heated, wood burns by producing flammable volatiles that may ignite. For wood to spontaneously combust, the temperature must be raised to 275 °C. However, if there is a flame source, it can become flammable at lower temperatures (Levan and Winandy, 1990; Yalinkilic et al., 1996; Yalinkilic et al., 1997a). For wood ignition; oxygen, O_2 flame source and flammable material are necessary. However, wood has excellent natural fire resistance as a result of its remarkably low thermal

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conductivity and the fact that wood char is formed when wood is burned. In order to reduce flammability and provide safety, wood is treated with fire-retardant chemicals. In other words, the combustibility of wood may be reduced with flame-retardants or fire-retardants (Nussbaum, 1988; Ellis and Rowell, 1989; Mitchell, 1993).

Borate chemicals offer substantial advantages for wood protection, providing fire resistance as well as efficacy against both fungi and insects, low cost, and ease of handling and treatment. There is growing interest in their low toxicity for mammals and environmental acceptability (Hafizoglu et al., 1994; Laks and Manning, 1995; Yalinkilic et al., 1997b; Yalinkilic et al., 1998a).

Boric acid (BA) and borax (Bx) are the most common boron compounds used in the wood preservation industry to obtain the benefit of their biological effectiveness and fire retardancy ability (Hafizoglu et al., 1994; Baysal, 1994). BA and Bx mixtures have some efficacy in retarding flame spread on wood surfaces. In addition to the usual char-forming catalytic effect, they have a rather low melting point and form glassy films when exposed to high temperatures in fires (Nussbaum, 1988).

Bx tends to reduce flame spread, but can promote smoldering or glowing. On the other hand, BA suppresses smoldering but has little effect on flame spread. Therefore, these compounds are normally used together (Yalinkilic et al., 1996; Baysal, 1994; Hafizoglu et al., 1994).

According to Baysal (1994), the weight loss and destruction time of Calabrian pine wood specimens treated with a BA acid and Bx mixture were much lower compared to untreated control specimens. Yalinkilic et al. (1996) reported that Douglas wood treated with a BA + Bx mixture (7:3; weight: weight) had higher fire-retardant properties than wood treated with either BA or Bx. Yalinkilic (2000) also reported that BA, Bx and their mixture (BA + Bx, 5:1, w/w) were added to a urea formaldehyde adhesive while manufacturing particleboard from waste tea leaves. Fire test results showed that whereas BA shortens the glowing time, while Bx extends it, the mixture of these boron compounds improved overall fire-retardant effectiveness.

Due to environmental considerations, CCA and creosote are used in a limited. PCP and many other biocides have also been prohibited in many European countries for a long time due to their detrimental effects on nature and on human health (Bozkurt et al., 1992). Therefore, in recent years, researchers have concentrated on the use of vegetable-based chemicals for wood protection. Some plant extracts contain tannin or have toxic effects against biotic agents, and they could be used to protect wood or wood-based objects from destructive organisms (Bozkurt and Göker, 1986; Schulta and Nicholas, 2000; Temiz, 2000).

Onuorah (2000) studied the ability of the heartwood extracts of two very durable tropical hardwood species (*Milicia excelsa* (Welw.) C.C. Berg Syn *Chlorophora excelsa* (Welw.) and *Erythrophelum suaveolens* (Guill & Perr.) Breanan Syn *Ertyrophelum guienensis* to suppress *Lenzites trabea* (brown rotter) and *Polyporus versicolor* (white rotter) attacks. In his work, air dried extracts were dissolved in 60% methanol and then impregnated into sapwood blocks of *Ceiba pentandra*. Treated blocks were exposed to *Lenzietes trabea* and *Polyporus versicolor* attack by soilblock tests. Only extract dosages of 48.056 and 96.11 kg m^{-3} were effective at in suppressing fungal attack.

Chang et al. (1998) reported the antifungal activities of α-cadinol, α-cedrol, hinokiol, sugiol, ferruginol, helioxanthin, savinin, and taiwanin C isolated from Taiwania heartwood. Of these, α-cadinol was found to possess the highest antifungal effectiveness. Kondo and Imamura (1998) had investigated the antifungal compounds in *Chamaecyparis obtusa* heartwood extracts using gas-liquid chromatography analysis. They deduced that the main antifungal compounds in *Chamaecyparis obtusa* were cadinane skeletal sesquiterpenoids.

Digrak et al. (1999) studied the antimicrobial activities of valex (the extract of valonia), as well as those of mimosa bark, gallnut powder, *Salvia aucheri* Bentham var. *aucheri* and *Phlomis bourgei* Boiss. extracts. Their results indicated that mimosa bark extracts had the greatest antibacterial activity, and they were followed extracts valex, gallnut powders, *Salvia aucheri* var. *aucheri* and *Phlomis bourgei*. Furthermore, it was found that gallnut powders and the extracts of mimosa bark contained high amounts of tannins and had antifungal qualities.

The objective of the present work was to determine the fire performance of Calabrian pine wood treated with a BA and Bx mixture and then treated secondarily with some plant extracts. We also aimed to determine the fire properties of wood treated with aqueous solutions of plant extracts that are well known for their antimicrobial and antifungal properties. The tannin contents of the plant extracts used in this study was 34% for brutia pine bark powder, 68-70% for acorn powder, 64% for sumach leaf powder and 50-70% for gall-nut powder (Bozkurt and Göker, 1986; Baytop, 1999).

Materials and Methods

Preparation of test specimens

Calabrian pine wood specimens were cut to 13 x 13 x 76 mm (radial by tangential by longitudinal) for fire testing according to ASTM E 160-50. (1975). All specimens were oven-dried at 103 ± 2 °C before and after treatment. The plant extracts utilized in this study were obtained from different regions in Anatolia. Valex was obtained from valonia oak (*Quercus macrolepis* Ky-Q. *aegilops* L.) grown in the Muğla-Aydın region, brutia bark powder was obtained from Calabrian pine grown in Muğla region, and gallnut powder was obtained from *Quercus infectoria* grown near Kozanlı (Adana). Lastly, sumach leaf powder was collected from Kahramanmaraş.

Preparation of the extracts and chemicals solution

Two groups of chemicals were chosen to prepare aqueous solutions (Table 1):

1. Borates: Aqueous solutions of BA and Bx were mixed by weight (7/3; w/w) and dissolved in distilled water to a concentration of 7%, which is the suggested ratio for increasing the fire resistance of wood (Hafizoglu et al., 1994; Baysal, 2003). A magnetic stirrer was used to accelerate the dissolving.
2. Plant extracts: Calabrian pine powder (designated "bark powder"), acorn powder, sumach leaf powder and gall-nut powder were dissolved in distilled in water to a concentration of 4%, which is the suggested ratio to protect wood from a number of wood destroying organisms (Sen, 2001; Temiz, 2000). The solutions were heated to 80 °C.

Treatment method

The test specimens were impregnated with aqueous solutions of borate and plant extracts according to ASTM D 1413-76. Treatment solutions were prepared the day before impregnation for homogenizing. A vacuum

Table 1. Chemicals used in treatments.

Group No.	Impregnation chemicals and concentration of solutions	pH*	Density of solutions (gml^{-1})
1	Control**	-	-
2	BA + Bx (7%)***	7.20	1.020
3	Acorn powder (4%)	4.81	1.10
4	1. Acorn powder (4%) 2. BA + Bx (7%)	-	-
5	Gall-nut powder (4%)	4.84	1.035
6	1. Gall-nutpowder 4%) 2. BA + Bx(7%)	-	-
7	Bark powder (4%)	4.38	1.10
8	1. Bark powder (4%) 2. BA + Bx (7%)	-	-
9	Sumach leaf powder (4%)	5.47	1.085
10	1. Sumach leaf powder (4%) 2. BA + Bx (7%)	-	-

* at 25 °C
** Untreated wood specimens
*** BA: Boric acid; Bx: Borax

desiccator used for the impregnation process was connected to a vacuum pump through a vacuum trap. The wood specimens were subjected to 30 min of vacuum at 760 mmHg followed by 30 min of diffusion in the treatment solutions. Three replicates, one of which contains 24 specimens, were used for each variation.

Weight percent gain (WPG) (%, w/w) due to chemical load was calculated via the following equation:

$$\text{WPG (\%w/w)} = \frac{W_{of} - W_{oi}}{W_{oi}}$$

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

Fire test method

Fire tests for the Calabrian pine wood specimens were performed according to ASTM E 160-50. Specimens were conditioned at 27 ± 2 °C and 30-35% relative humidity to the targeted equilibrium moisture content of 7% prior to fire testing. The 24 specimens were arrenged into 12 layers shaped like a square prism (Figure). The heating flame was connected to an LPG tank controlled by a sensitive bar-gauged valve. The flame was balanced a the standard height before the fire test samples' frame was put in position. Then, fire testing was performed with a flame source, without a flame source and at the glowing stage according to ASTM E 160-50. Temperatures were recorded at the combustion column by thermocouples at 15, 30 and 30 for combustion with a flame source, without a flame source and at glowing stage respectively.

Evaluations of fire test results

Fire test results were evaluated by a computerized statistical program composed of analysis of variance and by following Duncan tests at a 95% confidence level. Statistical evaluations were performed on homogeneity groups (HG), in which different letters reflected statistical significance.

Results and Discussion

Weight loss

The weight losses of Calabrian pine specimens are given in Table 2. In terms of combustion weight losses of the treated specimens, borates exhibited their well-known fire-retardant effect. The lowest weight losses were recorded for the Calabrian pine specimens treated with a BA and Bx mixture (63.4%). Wood specimens treated with a secondary (BA + Bx) mixture with plant extracts reduced weight loss up to 15-20% compared to those treated with plant extracts alone. Test specimens treated with aqueous solutions of plant extracts yielded

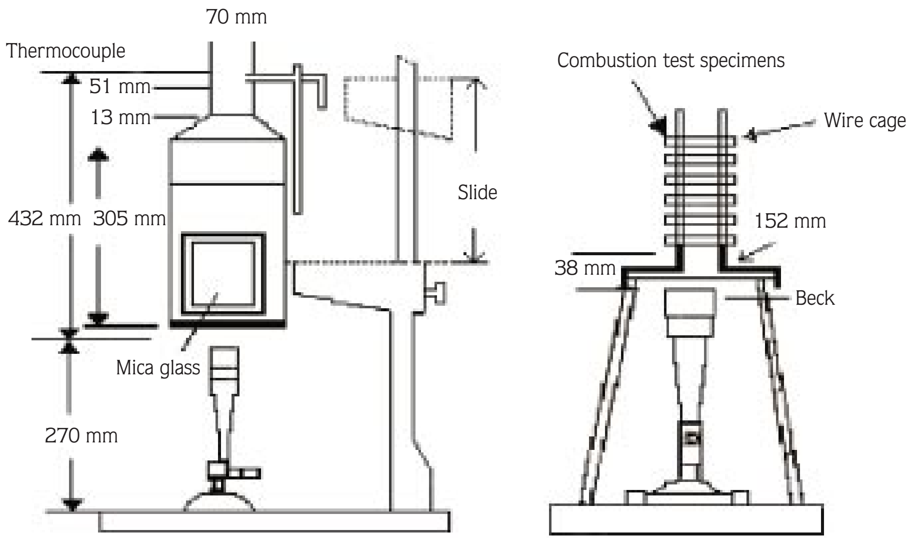

Figure. Combustion test apparatus.

Table 2. The weight losses of Calabrian pine wood resulted from the fire test. Three replication were made for each group. Small letters given as superscript over total retention and weight loss values represent homogeneity groups (HG) obtained by statistical analysis with similar letters reflecting statistical insignificance at the 95% confidence level. SD: Standard deviation.

Group No.	Treatment Chemicals	WPG (Mean ± SD)	Weight Losses (Mean ± SD)
1	Control	-	88.3 ± 3.4^{a}
2	BA + Bx*	2.85 ± 0.87^{d}	63.4 ± 5.6^{c}
3	Acorn powder	5.27 ± 3.39^{b}	91.1 ± 2.2^{a}
4	1. Acorn powder 2. BA + Bx	2.25 ± 0.77^{d}	80.3 ± 6.9^{b}
5	Gall-nut powder	7.24 ± 3.12^{a}	92.0 ± 6.5^{a}
6	1. Gall-nut powder 2. BA + Bx	4.87 ± 1.69^{bc}	78.8 ± 4.7^{b}
7	Bark powder	3.57 ± 0.85^{bcd}	88.9 ± 9.2^{a}
8	1. Bark powder 2. BA + Bx	4.72 ± 2.12^{bc}	75.3 ± 5.1^{b}
9	Sumach leaf powder	3.76 ± 1.58^{bcd}	92.7 ± 2.8^{a}
10	1. Sumach leaf powder 2. BA + Bx	3.31 ± 1.82^{cd}	79.6 ± 7.7^{b}

* BA: Boric acid; Bx: Borax

higher weight losses compared to the untreated control specimens. Therefore, it can be concluded at this stage that plant extracts have no fire–retardant effect but require borate supplementation.

Yalinkilic et al. (1998b) reported that Douglas fir (*Pseudotsuga menziesii* (Mirb.) Franco) specimens treated with a BA + Bx mixture (7:3; w/w) lost around 58% of their weight during combustion. Temiz (2000) reported that *Alnus glutinosa* specimens treated with similar chemicals lost about 60% of their weight. According to Baysal (1994), the weight losses of Calabrian pine (*Pinus brutia* Ten.) specimens impregnated with BA (5.5% conc.) were approximately 68%.

Temperatures in the combustion column

Table 3 contains the recorded temperatures in degrees Celcius obtained with and without flame source (TFS and TWFS respectively) and at the glowing stage (GS).

BA and Bx treatment alone and plant extract treatment followed by supplementary borate treatment had lower heat release rates for almost all the combustion stages, indicating the fire-retardant effects of borates on combustion. The lowest heat release at the combustion stage with a flame source was obtained at 323 °C for specimens impregnated with the BA + Bx mixture. The lowest heat release of the combustion without a flame source was recorded at 404 °C for specimens impregnated with acorn powder and treated secondarily with the BA + Bx mixture and the lowest heat release was encountered at the glowing stage at 107 °C for specimens impregnated with sumach leaf powder and treated secondarily with the BA + Bx mixture. We recorded lower temperatures indicating the flame retardancy effect of borates on combustion when combined with the plant extracts. As seen in Table 3, wood specimens treated with sumach leaf powders had the highest heat release at an average of 747 °C during combustion stage. This effect might be due to the potential ignition effect of sumach leaf powder in the combustion stage. The plant extracts release considerable heat when used alone. For instance, sumach leaf powder specimens were almost 300 °C higher during combustion with the flame source when compared to untreated control specimens (Table 3).

Table 3. Temperature records at during flame combustion (TFS), without flame source (WTFS) and glowing stage (GS).

Group No.	Treatment chemicals	TFS (°C) (Mean ± SD)	WTFS (°C) (Mean ± SD)	GS (°C) (Mean ± SD)
1	Control	454 ± 156^{cde}	718 ± 74^{abc}	226 ± 94^{a}
2	BA + Bx	323 ± 126e	492 ± 308^{d}	134 ± 37^{bc}
3	Acorn powder	655 ± 208^{ab}	786 ± 233^{a}	189 ± 85^{ab}
4	1. Acorn powder 2. BA+Bx	438 ± 170^{cde}	404 ± 266^{d}	124 ± 36^{bc}
5	Gall-nut powders	660 ± 184^{ab}	738 ± 105^{ab}	211 ± 130^{a}
6	1. Gall-nut powder 2. BA + Bx	523 ± 161^{bc}	559 ± 207^{bcd}	130 ± 53^{bc}
7	Bark powder	581 ± 134^{bc}	598 ± 73^{abcd}	196 ± 112^{ab}
8	1. Bark powder 2. BA + Bx	353 ± 127de	524 ± 279^{cd}	135 ± 30^{bc}
9	Sumach leaf Powder	747 ± 203^{a}	781 ± 201^{a}	199 ± 90^{ab}
10	1. Sumach leaf powder 2. BA + Bx	493 ± 173^{cd}	450 ± 308^{d}	107 ± 31^{c}

Note: For abbreviations refer to Table 2.

Baysal (1994) reported that the heat release for Calabrian pine wood treated with boric acid was 165.5 °C at the combustion stage, 290.5 °C for the without combustion stage and 118.0 °C for the glowing stage. Moreover, according to Hafizogflu et al. (1994) the heat release for treated Douglas fir wood (*Pseudotsuga menziesii* (Mirb.) Franco) was 233.5 °C at the combustion stage, 270.5 °C for the without combustion stage and 149.5 °C for the glowing stage.

Duration of combustion without flame source and glowing

Almost all plant extracts gave unfavorable results in terms of duration of combustion without flame source and glowing (Table 4). These results are consistent with earlier findings (Temiz, 2000; Baysal 2003). Gall-nut powder was the worst treatment chemical for lengthening combustion after flame source removal (872 sec). The BA + Bx treatment shortened the duration without flame source and at glowing stage. Le Van and Winandy (1990) reported that both boron compounds had different effects on flame retardancy, as Bx lengthen the time of glowing while BA increased smoke generation. Therefore, it seems both chemicals played a role as fire-retardants in this study, while it is evident that these chemicals had an inhibitory effect on duration of combustion without flame just as they shorten without combustion and glowing time (Table 4). As for the deflection stage, which can be an indicator of constructional failure during combustion or time until constructional failure begins, the longest time was obtained with the specimens treated with acorn powder solutions followed by BA + Bx treatment for specimens treated with the BA + Bx mixture. Plant extracts with secondary borate treatment performed better than the other applications.

Conclusion

This study examined some limited combustion properties of wood specimens treated with borates or plant extracts and borates supplemented with plant extracts. Double treatments of wood with borates and plant extracts were performed to benefit from their potential cumulative protection in terms of biological resistance and fire retardancy at the same time.

Plant extract type was also found to have a profound effect on the combustion behavior of treated wood. They drastically increased the weight loss of wood after combustion, while only borates reduced such losses by a considerable extent as well as reducing the temperature profiles of the released heat during combustion after

Table 4. Duration without flame source (DWFS), after glowing (DGL) and to deflection (DT).

Group No.	Treatment chemicals (Mean ± SD)	DWFS (s) (Mean ± SD)	DGL (s) (Mean ± SD)	DT(s)
1	Control	565 ± 17.4[d]	604 ± 34.4[b]	294 ± 18.9[e]
2	BA + Bx	188 ± 34.1[h]	385 ± 58.8[e]	803 ± 20.6[a]
3	Acorn powder	872 ± 47.6[a]	540 ± 47.8[c]	186 ± 18.2[f]
4	1. Acorn powder 1. BA + Bx	338 ± 33.3[f]	393 ± 27.9e	714 ± 37.2[b]
5	Gall-nut powder	369 ± 49.2[e]	609 ± 19.3[b]	90 ± 16.1[g]
6	1. Gall-nut powder 2. BA + Bx	156 ± 20.5[i]	517 ± 25.5[c]	347 ± 23.9[d]
7	Bark powder	665 ± 19.7[c]	751 ± 65.8[a]	98 ± 9.7[g]
8	1. Bark powder 2. BA + Bx	211 ± 13.4[h]	540 ± 47.8[c]	355 ± 15.3[d]
9	Sumach leaf powder	788 ± 37.4[b]	605 ± 40.1[b]	181 ± 33.7f[e]
10	1. Sumach leaf powder 2. BA + Bx	270 ± 15.5g	476 ± 28.8d	570 ± 40.1[c]

Note: For abbreviations refer to Table 2.

flame source removal and at glowing stage. In addition, specimens treated with aqueous solutions of plant extracts had poorer fire parameters. However, the fire resistive properties of wood specimens treated with a secondary BA + Bx mixture over plant extracts were significantly higher.

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