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RESEARCH PAPER

The Reaction to Fire of Some Chemicals Treated Pine Wood Product Surface*

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Abstract: Wooden materials have been extensively used for furniture, outdoor and indoor cladding, buildings, terrace, fence, garden furniture and interior decoration and to decorate the roofline of houses. However, wood which is used outdoors or in areas exposed to moisture needs to be protected against wood-destroying organisms and to be shielded from water, marine pests, fungi, fire and weather conditions. Untreated wood materials have no resistance to the spread of fire and many buildings which constructed from wood based materials need to fire resistance. It is possible that the wooden material ensures very durable and resistant against physical effects by surface applications such as wood preservative paint and acrylic resin-based varnish. The application of fire retardant chemicals can also provide to satisfy regulatory requirements for wood products.

In this study, titanium dioxide and antimony trioxide were applied on pine (*Pinus sylvestris L.*) solid wood material to determine durability of reaction to fire using by oxygen index test technique (ASTM D 2863-6) and real fire test. These chemicals were added to the wood preservative paint which is a commercial product as concentrations of 2%, 5% and 10% for titanium dioxide and 2% and 5% for antimony trioxide. The effects on color change of their surfaces, brightness and surface roughness measurements, water absorption and thickness swelling of this wood material were also carried out. The results obtained were analyzed statistically and compared with the related standards. Addition of these chemicals to used wood preservative paint showed positive effect on the fire properties of the pine wooden surface.

Keywords: Antimony trioxide, Fire retardant, Titanium dioxide, Wood preservative paint.

Yüzeyi Bazı Kimyasallar ile Muamele Edilen Çam Odunun Yanma Performansı

Öz: Ahşap malzemeler, mobilya, dış ve iç cephe kaplamaları, binalar, teras, çit, bahçe mobilyaları, iç dekorasyonda ve evlerin çatı hattını dekore etmek için yaygın olarak kullanılmaktadır. Bununla birlikte, açık havada veya neme maruz kalan alanlarda kullanılan odun odunu tahrip eden organizmalara karşı, sudan, deniz zararlılarından, yangın ve hava koşullarından korunmalıdır. İşlem görmemiş ahşap malzemelerin yangının yayılmasına karşı direnci yoktur. Ahşap esaslı malzemelerden yapılmış birçok binanın yangına dayanıklı olması gerekir. Ahşap malzemenin, ahşap koruyucu boya ve akrilik reçine bazlı vernik gibi yüzey uygulamalarında fiziksel etkilere karşı çok dayanıklı ve dayanıklı olması mümkündür. Yangın geciktirici kimyasalların uygulanması, ahşap ürünler için yasal gereklilikleri karşılamayı da sağlayabilir.

Bu çalışmada, sınırlayıcı oksijen indeksi testi (ASTM D 2863-6) ve gerçek yanma testi kullanılarak yangına karşı dayanıklılığın belirlenmesi amacıyla çam (Pinus sylvestris L.) odunu malzemesine titanyum dioksit ve antimon trioksit uygulanmıştır. Bu kimyasallar, titanyum dioksit için %2, %5 ve %10 ve antimon trioksit için %2 ve %5 konsantrasyonlarında ticari bir ürün olarak ahşap koruyucu boyaya eklenmiştir. Ahşap yüzeylerdeki renk değişimleri, parlaklık ve yüzey pürüzlülüğü ölçümleri, su absorpsiyonu ve kalınlığına şişme üzerine etkileri araştırılmıştır. Elde edilen sonuçlar istatistiksel olarak analiz edilmiş olup ilgili standartlarla karşılaştırılmıştır. Bu kimyasalların ahşap koruyucu boyaya eklenmesi, çam odun yüzeyinin yanma özellikleri üzerinde olumlu etki göstermiştir.

Anahtar sözcükler: Antimon trioksit, yangın geciktirici, titanyum dioksit, ahşap koruyucu boya.

INTRODUCTION

Wood is renewable material, easily workable, compatible with its outstanding physical and mechanical properties. Since it is used flats, window and door, furniture, ceilings and floors, stairs, benches, its preservation is important significantly. But the use of wood can be restricted by safety regulations concerned with its ignitability and flame spreading characteristics. The application of a suitable fire retardant system can prevent these problems, thus can be used in many areas of use (Russell et al., 2004).

The resistance of wood to burning and flame spread have been researched for many years. A wood consists of carbon and hydrogen which make it highly combustible. A fuel, an oxidizer and a source of heat are three main elements in a combustion reaction. The combustion of wood occurs by the pyrolysis of the cellulose and its reaction to oxygen. When the temperature is increased, the pyrolysis starts. The combustion stops if any of the elements is removed (Pabelina et al., 2012). Many fire retardant techniques include surface treatment with fire retardant chemicals such as fire resisting coatings and pressure impregnations of chemical solutions into wood (White, 1999).

Fire retardants are mainly based on organic phosphorus, halogens, and metallic oxides. A fire retardant such as phosphorus, aluminum, antimony, chlorides, bromides, and boron-containing compounds can act in physically or chemically. It can cause a charred layer of carbon to form on the surface (Demir et al., 2005). The fire retardant chemicals act as by conversion of volatile gases to on flammable gases, promotion of char formation, forming an intumescent foam barrier at the surface, free radical termination in the gaseous phase, and occurring a glaze barrier at the surface (Ötsman & Tsantaridis, 2016). The fire retardants can affect the reaction to fire properties, however this effect is lower for the fully developed fire. When intumescent paints are used the time for start of charring can be delay and the fire resistance can be increased. The fact is that fire retardants cannot make wood non-combustible. The fire performance of fire retardant treated the virgin wood products may degrade depending on time in outdoor applications. The fire retardant chemicals can be removed in the wood surface when the wood surface is exposed to high humidity (Nussbaum, 1988; Östman et al., 2001; Ötsman & Tsantaridis, 2016). Mineral fillers such as titanium dioxide for intumescent fire retardant coating have attracted much attention in recent years. Titanium dioxide (TiO₂) can be also used in coating industry as pigments. Some researchers were found that the presence of rutile type TiO_2 could improve the fire resistance of the coating by enhancing char formation (Li et al., 2015; Lam et al., 2011). Antimony trioxide is usually used as a synergistic energy to improve fire retardancy (Giúdice & Benítez, 2001).

Pine wood which is a type of softwood grown in many varieties in world has a uniform texture, finishes well,

and east to work. It is usually light yellow in color and also some resistance against shrinkage, sharping and swelling (Zhong et al., 2013). It is considered that these properties will be improved by the use of some chemicals treated wood preservative paint. The wood preservatives contain pentachlorophenol or creosote in oil, water-borne salt treatments, copper, zinc, chromium, arsenic, and other compounds can protect wood against attack by fungi, bacteria, and insects (Thamasson et al., 2006).

There is a need to search new fire retardant treated wood products with improved long term durability of the reaction to fire performance in many usage areas, recently. The main aim of the study is to obtain an improved fire performance of pine wood surface. In this study, the effects on the flammability (the limiting oxygen index (LOI) and real fire test) of pine wood surface with applied of titanium dioxide and antimony trioxide were investigated owing to the synergy effect of wood preservative paint and these chemicals. The surface roughness, color change, brightness and physical properties of the pine wood surfaces were also measured in according to the relative standards.

MATERIAL and METHODS

Materials: Pinus sylvestris L. solid wood surface was used for improving its flammability properties in this study. The wood preservative paint was obtained from Polisan, Turkey. Titanium dioxide and antimony trioxide were purchased from Sigma-Aldrich for this study. The purity grades of these chemicals are above 99,99%. Table 1 and 2 show average size of chemicals and sample codes, respectively.

Table 1. Average	size	of o	chemicals.
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Chemical type	Average size (nm)
Titanium dioxide	30≥
Antimony trioxide	30≥

Table 2.	Composi	tion of	fire	retardant	coatings,	%	by	volume	on
wood pre	eservative	paint							

Sample Code	2%	5%	10%	
Control	-	-	-	
WPP	-	-	-	
A1	х	-	-	
A2	-	х	-	
A10	-	-	х	
T1	х	-	-	
T2	-	х	-	

WPP: Wood preservative paint; A: Antimony trioxide; T: Titanium dioxide.

Preparation of wood surfaces: Firstly, the wood preservative paint is applied onto the pine wood surface. Amounts of fire retardant chemicals were changed in proportion to amounts of titanium dioxide (2% and 5%) and which of antimony trioxide (2%, 5% and 10%). Secondly, they were mixed for 5 minutes with the wood preservative

paint, and then applied by a brush onto solid pine surface. Once again was painted by the wood preservative paint after the wood surfaces were dried for 12 hours.

Fire properties: The limiting oxygen index (LOI) test method was determined using a Dynisco Limiting Oxygen Index Chamber according to ASTM D 2863 to measure the minimum oxygen concentration. Four samples with the dimensions of 10 mmx15mmx5 mm were used for each group. All wood samples were placed in vertical glass column and then adjusted oxygen and nitrogen flow. The LOI test determines minimum concentration of oxygen and nitrogen flowing upward in a test column. The five samples for each group were tested.

The real fire test was also performed. The sample dimensions were the same as the dimensions of the LOI sample. All samples were burned to the mark in mm/min. The blowtorch was kept at a distance of 10 cm from the wood surface. The flame was applied for 5 seconds. The flame extinguishing time was recorded after the flame source cut off. Four samples for each group were tested.

Color measurements: All surfaces were removed from dust and dirt to minimize the risk of variation of color values by differences in surface structure. The color measurement was carried out according to CIE L*a*b* method by parameters L*a*b* and ΔE * with a PCE-TCD 100 (CM10P Color Meter). Measurements were realized on both sides of wood samples. The arithmetic mean of these measurements was calculated for each sample. The coordinates L* (lightness or black-white relation), a* (coordinate red-green), b* (coordinate yellow-blue), measured on unpainted and painted solid pine wood were used to determine overall color change ΔE * by using the CIE L*a*b* color measuring system.

Surface roughness: The residual particles remaining after painting were removed by cleaning the surface. Surface roughness of painted or unpainted samples was measured with a profilometer (Mitutoyo SJ-210). Equipment has stylus with 5μ radius and 90° contact angle running at speed of 0.5 mm/s. A total of 25 random measurements with a span of 15 mm were taken from the surface. Mean peak to valley height (R_z) which is well accepted roughness parameter was used an indicator of the surface quality of wood samples. The measurements were performed in different areas, along two different perpendicular directions (longitudinal and tangential).

Surface brightness: The brightness measurements were measured by PCE-GM 100. Measurements were done with a gloss meter which measured at 20°, 60°, and 80° as parallel and perpendicular to the fibers for each wood surface before and after painting.

Physical testing: The thickness swelling (TS) and water absorption (WA) were determined after 2h, 24h, 48h, 72h, 96h and 120h soaking in water in accordance with EN 317.

Statistical Analysis: The statistical analysis was carried out with using SPSS 21.0 statistical package software. The results were statistically tested with the one-way analysis of variance. The significance (P<0.05) between the samples was compared with Duncan homogeneity groups. Each value is an average of 6 specimens and the values in the parentheses are standard deviations.

RESULTS and DISCUSSION

Reaction to fire properties: The results of LOI and real fire test are shown in Table 3. LOI levels of the samples were in the range of 26,5-27 for pine wood and wood preservative paint. The LOI levels of antimony trioxide and titanium dioxide with samples were ranging from 28,5 to 30. A maximum LOI level was obtained with A10 sample including antimony trioxide of 10%. Antimony trioxide is a well-known pigment for the interference on flame spreading velocity. It shows an adequate fire-retardant behavior during the fire spreading reaction. But it is not effective it by itself. It should be used in combination with halogenated organic compounds. The antimony trioxide increases smoke production since the char analysis shows that about 80-95% of antimony volatilizes. It also decreases concern for the toxicity of antimony oxide (Giúdice & Benítez, 2001). According to ISO 4589, control and WPP samples were located in "Limited fire retardant material" class while A1, A10, T1 and T2 were classified in "Fire retardant material" class, as seen in Table 4. For real fire test, the effect of A10 and T2 was important to decrease the flame spreading. The fire retardant chemicals can act combining one or more mechanisms. Titanium dioxide shows a significant physical activity generated by reducing the concentration of the organic part which divides the material in isolated pieces by occupation of pores and amorphous polymer regions. As a result of which a higher amount of heat is required to attain the pyrolysis temperature (Giúdice & Benítez, 2001). Hashim et al., (2009) reported that lignocellulosic material reduced heat release rate and increased charring rate. When compared with char weight of all samples, a minimum value was obtained with T2 sample, followed by A10 sample.

Table 3. LOI and real fire test results of samples.

Sample ID	LOI	After flame time	After glow time	Weight loss	Smoking
•		(sec.)	(sec.)	(%)	
Control	26,5	60	60	1,689	+
WPP	~27	60	29	0,799	
A1	28,5	60	17	0,759	
A2	-	60	4	0,750	
A10	~30	60	0	0,729	
T1	28,5	60	20	1,124	+
T2	28,5	60	10	0,558	+

LOI level	Classification of fire	Chemical
≤23	Flammable material	
24-28	Limited fire retardant material	Control, WPP
29-35	Fire retardant material	A1, A10, T1, T2



Figure 1. The images of samples after LOI test.

Brightness and Color Properties: The brightness (80°) values were increased by usage of antimony trioxide and titanium dioxide according to that of pine wood. This is clearly seen Figure 2. The brightness values of the samples were increased when ratio of antimony trioxide increased from 2% to 10% for antimony trioxide. Similar results were also seen in 2% and 5% ratios of titanium dioxide. The highest values of the brightness were obtained from A10 samples. No significant difference was found between ΔE values of samples with antimony trioxide and titanium dioxide with samples based on Duncan test as displayed in Table 5.

Table 5. Statistical analysis results and homogeneity groups of the brightness, ΔE and roughness test results.

Somula designation	Brightness			AF	Dra (um)	
Sample designation	20°	60°	80°	- AL	кz (μш)	
Control	0,24a*	1,76a	69,66a	2,80a	8,90b	
Control	(0,05)**	(0,59)	(4,43)	(1,49)	(1,51)	
W/DD	4,96ab	24,32b	80,08b	2,30a	6,02ab	
wrr	(2,15)	(5,01)	(2,61)	(1,04)	(3,26)	
4.1	9,25bc	38,36cd	93,03c	2,38a	5,90ab	
AI	(5,75)	(13,24)	(1,16)	(1,48)	(4,02)	
10	9,75bc	41,73d	98,02d	2,44a	5,51ab	
A2	(1,24)	(3,85)	(2,84)	(1,26)	(3,90)	
110	9,17bc	38,95cd	112,74f***	2,40a	2,78a	
A10	(2,59)	(5,55)	(3,74)	(0,53)	(1,53)	
7 1	11,37c	39,62d	107,81e***	3,32a	4,10a	
11	(8,59)	(10,91)	(2,21)	(0,31)	(1,34)	
T 2	7,15bc	29,64bc	110,61ef***	2,48a	3,98a	
12	(2,97)	(7,53)	(3,33)	(1,16)	(1,93)	
Groups with same letter	s in column indi	cate that there	s no statistical dif	ference (p <	0.05) between	

the samples according to Duncan's multiply range tests which were performed separately for each group.

** The values in the parentheses are standard deviations.

*** Because UV filter is not used, the results are more than a hundred.

Figure 3 shows ΔE values of all samples. There were no apparent differences between the WPP and antimony trioxide for ΔE . As you can see Table 6, all samples are taken part into so little differences group. It was observed that ΔE is not significantly changed by the addition of 5 wt % of antimony trioxide ratios. However, the addition of antimony trioxide content above 5 wt%, ΔE decreased gradually. ΔE was found to be 2,38-2,44 for WPP with applied by 10% antimony trioxide, and 2,48-3,32 for WPP with applied by 5% titanium dioxide. While titanium dioxide ratios increased, ΔE increased slightly. The reason of this is that titanium dioxide improves brightness and smoothness (Stoneburner, 2014).



Figure 2. Brightness values (80°) of the samples.



Figure 3. ΔE values of the samples.

Table 6. Color ΔE differences (Heidelberg, 2008)

0-1	Unseen differences
1-2	So little differences
2-3,5	Intermediate differences
3,5-5	Apparent differences
>5	Obvious differences

Surface roughness: Table 5 and Figure 4 indicates R_z values of the samples. The highest R_z value of 8,90 μ m was found for control sample. Rz values of WPP samples with added antimony trioxide were decreased ranging from 2% to 10%. Rz values of WPP samples with added titanium dioxide were also reduced ranging from 2% to 5%. The minimum R_z value was found for A10 samples while the control had the highest R_z value which was indicated by statistical test. Both WPP samples with antimony trioxide and titanium dioxide for the minimum Rz value resulted in 2,78 µm and 3,98 µm, respectively. The quality of coating applied to surface of wood which being a nonhomogeneous material is affected by several factors such as roughness, porosity, species, density and interaction between coating and the substrate (Cheng & Sun et al., 2006; Ozdemir et al., 2015). The component of used fire retardants chemicals would be considered one of the mentioned factors influencing roughness as well as bonding between wood preservative paint and the pine wood surface in this study.



Figure 4. R_z values of the samples.

Physical properties: The water absorption and thickness swelling results of samples after immersion in distilled water for 120 hours are given in Figure 5 and Figure 6. It was seen that the increasing of water immersion time resulted in WA and TS values to increase for all samples. While the water absorption and thickness swelling of control sample were higher than those of painted samples. The usage of antimony trioxide and titanium dioxide decreased WA and TS. The minimum value for WA was achieved from T2 sample. It is known that fire retardant chemicals prevent the samples to intake water by penetrating to the lignocellulosic material (Donmez et al., 2016). Some researchers have reported that fire retardants such as zinc borate, aluminum trihydrate, aluminum trihydroxide, sodium aluminate) reduced water absorption and thickness swelling (Gnatowski & Burnaby, 2005; Hashim et al., 2009; Donmez et al., 2016).



Figure 5. Long-term water absorption of the samples



Figure 6. Long-term thickness swelling of the samples.

CONCLUSIONS

Consequently, the LOI results showed that pine wood surface painted with antimony trioxide and titanium dioxide had affirmative effects in comparison with control and WPP. A1, A10, T1 and T2 were in class of "fire retardant material" according to ASTM D 2863. Using A10 as a fire retardant exhibited the best result in terms of fire performance. As the increase in ratio of used chemicals for real fire test, it was concluded that the char weight of the samples was decreased. The long-term thickness swelling and water absorption values of painted with the chemicals have given better results than control. The ΔE of samples painted by using antimony trioxide and titanium dioxide did not change significantly. Increasing titanium dioxide has little improved the ΔE . The brightness value of samples increased while increasing ratio of chemicals. However, Rz values of the samples painted using antimony trioxide and titanium dioxide had a lower in comparison with control and WPP. With an increase in antimony trioxide and titanium dioxide loading, the R_z values decreased slightly.

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