ISSN impresa 0717-3644
ISSN online 0718-221X
Maderas. Ciencia y tecnología 21(1): 25 - 34, 2019
DOI: 10.4067/S0718-221X2019005000103

INVESTIGATION OF SOME SURFACE PROPERTIES AND THERMOGRAVIMETRIC ANALYSIS OF VENEER SHEETS TREATED WITH FIRE RETARDANTS

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ABSTRACT

This study investigated the effects of different fire retardant chemicals on surface and thermal properties of veneer sheets. Beech (*Fagus orientalis)*, alder (*Alnus glutinosa*), poplar (*Populus deltoides*) and scots pine (*Pinus sylvestris*) were chosen as wood species and zinc borate, borax, monoammonium phosphate and ammonium sulfate were chosen as fire retardant chemicals. The samples were impregnated by using the immersion method. Some surface properties such as colour measurements and surface roughness of the veneer sheets were conducted according to CIE L*a*b* system. Some thermal properties such as thermal conductivity of the veneer sheets were conducted according to standard and weight loss after combustion was determined by thermogravimetric analysis. Conforming to the results from the study, it was found that fire retardant chemicals increased the thermal conductivity and surface roughness of veneer sheets. Also, thermogravimetric analysis experiments showed that all of the fire retardant chemicals decreased the loss in weights.

Keywords: Colour measurement, plywood, surface roughness, thermal conductivity, urea formaldehyde.

INTRODUCTION

Wood and wood based panels have long been used as materials in the construction industry because they have a great durability, high strength and versatility (Stevens *et al.* 2006). Plywood, a wood based product, is one of the most important building and furniture materials (Fateh *et al.* 2013). Compared with solid wood, the chief advantages of plywood are that the properties along the length of the panel are more nearly equal to properties along the width, there is greater resistance to splitting, and the form permits many applications where large sheets are desirable (Aydin and Colakoglu 2008). There are some unfavorable characteristics of the plywood similar to wood and other wood-based composite panels. It can be combusted easily, and this is one of the undesired characteristics of plywood (Ozkaya *et al.* 2007). The flammability and combustibility properties of such a solid material can be reduced by several recommended treatments (Fateh *et al.* 2013). The treatment with fire retardant chemicals is the most effective process to protect wood and wood based products from fire is. Therefore, use of fire retardant chemicals has been increased. It has also risen due to awareness of environmental protection and consumer safety, requirement standards which have been raised accordingly. Moreover, capability and properties of fire retardant chemicals such as being harmless to human, animals, and plants with less release of smoke and toxic gases when burned are important parameters for consumers to select a fire retardant chemical. It was also shown the fire retardant chemicals have an effect on other physical, mechanical and some technological properties of the materials treated with them. Inorganic based fire retardant chemicals are extensively used in forest industry because they have both good thermal stability, less release of smoke, less corrosive toxic gases and less strength loss (He *et al.* 2014, Yao *et al.* 2012).

Thermal conductivity is the flow of thermal energy through a unit thickness of a material under a temperature gradient and is expressed by the coefficient of thermal conductivity (Kollman and Cote 1968). It is a very important parameter in determining heat transfer rate and is required for development of drying models in industrial operations such as adhesive cure rate (Kol and Altun 2009). Also, it must be known when choosing the insulation materials to attenuate fluctuation in the outdoor environment

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Received: 04.10.2017 Accepted: 22.07.2018

which maintains an indoor temperature that is independent of outdoor temperature fluctuations. The materials used for insulation must have good warmth-keeping properties such as lower thermal diffusivity to provide sufficient protection from severe temperature changes (Kawasaki and Kawai 2006). Wood materials possess superior thermal conductivity properties compared to other building materials due to its porous structure (Gu and Zink-Sharp 2005, Kruger and Adriazola 2010). They are one of the preferred materials in many applications such as construction industry, refrigerators, automobile industry, and in the manufacture of barrels, because of their low thermal conductivity and high resistance (Gu and Zink-Sharp 2005). Thermal conductivity of wood materials varies according to wood type, direction of wood fiber, resin type, and additive members used in the manufacture of wood composite panels (Kamke and Zylkowoski 1989).

The studies about thermal degradation of solid wood, wood based products or their by-products are necessary for construction fire safety to under-stand the comportments of those materials in case of fire. Thermogravimetric analysis (TGA) is a traditional method to investigate thermal degradation by determining the characteristics of weight loss in different heating rates and atmosphere conditions (Fateh *et al*. 2013). TGA can be tested with milligram-sized samples. Milligram size samples of materials can be rapidly manufactured and allow to regulate several formulations with fire retardant chemicals to be studied for degradation before full manufacture of the material is undertaken. TGA measurements can provide the pyrolysis rates and mechanisms and char yield in nitrogen at heating rates from 5 to 200°C/min (Hagen *et al.* 2009).

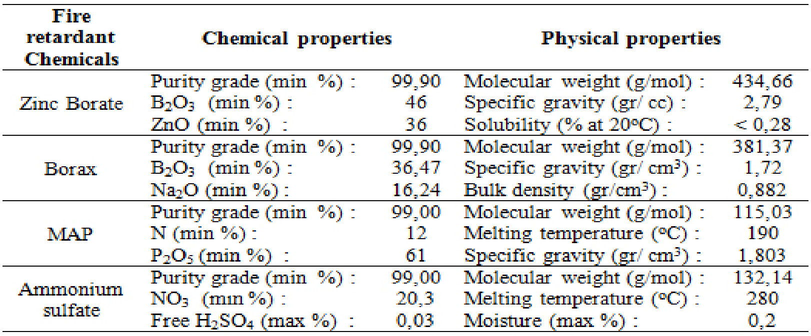
Environmental factors and treatment conditions such as UV light, heat, moisture, alkali or acids during steaming, preservative treatment, drying or finishing can cause changes in properties of wood and wood based products (Aydin and Colakoglu 2005, Burtin *et al.* 1998). These changes are generally physical and chemical changes which especially bring about a loss of original colour and surface fibers, and graying (Yildiz *et al.* 2013). Colour is a basic visual property which can be distinguished easily by consumers for wood and wood based products. Colour measurement is one of the quality control tests. The colour harmony of constituents is significant when assembling individual pieces into final products (Aydin and Colakoglu 2005, Resch *et al.* 2000). Recently, market requirements have been demanding that producers supply lumber and panel which is attractive, and of specified, uniform and replicable colour (Yeo and Smith 2003). Also, colour of material can cause a change in price of wood (Ishiguri *et al.* 2003). Surface roughness of veneers plays an important role in plywood manufacture. Cross grain, annual ring width, rays, knots, reaction wood, ratio of early wood and late wood, pre-treatment and peeling conditions, such as knife angle, are some of the raw material and production parameters influencing roughness of veneer. Control of veneer surface in plywood production is essential to maintain plywood quality (Ayrilmis *et al.* 2006).

Although there are lots of studies in literature about fire retardant chemicals, effects of fire retardant chemicals on thermal and surface properties of wood and wood based panels are not comprehensively investigated. The aim of this study is to increase the knowledge of effects of fire retardant chemicals on thermal and surface properties of veneer sheets. For this aim, four fire retardant chemicals and four wood species widely used in the plywood industry were used and the technological test methods thermal conductivity, TGA, surface roughness and colour measurement were applied.

MATERIALS AND METHODS

The wood species widely used in the plywood industry in Turkey were chosen as material. Beech *(Fagus orientalis Lipsky)*, alder *(Alnus glutinosa* subsp. *barbata)* and poplar (*Populus deltoides* I-77/51 clone) were obtained as hardwood and Scots pine (*Pinus sylvestris* L.) was obtained as softwood. Zinc borate, borax, monoammonium phosphate (MAP) and ammonium sulfate were used as fire retardant chemicals**.** The chemical and physical properties of the fire retardant chemicals used are given in Table 1 (these properties were supplied from the producers).

Table 1: Properties of fire retardant chemicals.



Fire retardant Chemicals	Chemical properties		Physical properties	
Zinc Borate	Purity grade (min %) :	99,90	Molecular weight (g/mol) :	434,66
	B_2O_3 (min %) :	46	Specific gravity (gr/ cc) :	2,79
	ZnO (min %) :	36	Solubility (% at 20°C) :	< 0,28
Borax	Purity grade (min %) :	99,90	Molecular weight (g/mol) :	381,37
	B_2O_3 (min %) :	36,47	Specific gravity (gr/ cm^3) :	1,72
	Na_2O (min %) :	16,24	Bulk density (gr/cm^3) :	0,882
MAP	Purity grade (min %) :	99,00	Molecular weight (g/mol) :	115,03
	N (min %) :	12	Melting temperature (°C) :	190
	P_2O_5 (min %) :	61	Specific gravity (gr/ cm^3) :	1,803
Ammonium sulfate	Purity grade (min %) :	99,00	Molecular weight (g/mol) :	132,14
	NO_3 (min %) :	20,3	Melting temperature (°C) :	280
	Free H_2SO_4 (max %) :	0,03	Moisture (max %) :	0,2

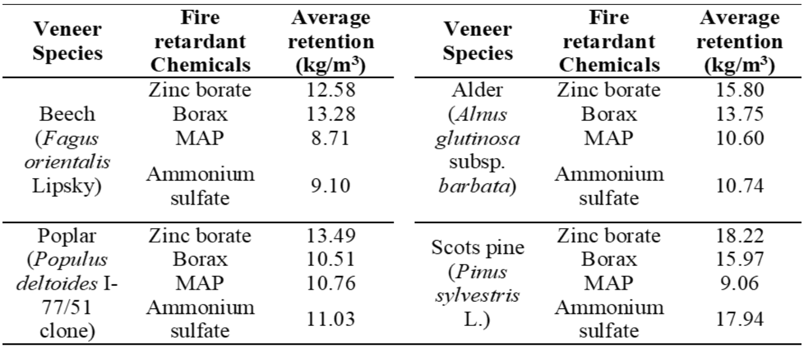
Manufacturing and treatment of veneer sheets

In this experimental study, rotary cut veneer sheets with dimensions of 0,2 ×50 × 50 cm thickness were obtained from freshly cut logs. While the alder and poplar veneers were manufactured from freshly cut logs, beech and Scots pine logs were steamed for 12 h before veneer production. The horizontal opening between knife and nosebar was 85 % of the veneer thickness, and the vertical opening was 0,5 mm in rotary cutting process. The veneers were then dried to 6-8 % moisture content with a veneer dryer. After drying, veneer sheets were treated with some fire retardant chemicals. For this aim, 5 % aqueous solutions of zinc borate, borax, monoammonium phosphate (MAP) and ammonium sulfate were used. The veneers were subjected to re-drying process at 110°C and then conditioned to 5-7 % moisture content again after they were immersed in the fire retardant solutions for 20 minutes. Twenty samples from each group were used to determine the level of retention. The retention level for each treatment solution was calculated with the following Equation 1, and they were presented in Table 2.

$$R = \frac{GxC}{V} x10 \; kg/m^3 \quad (1)$$

where; R is the retention level (kg/m^3), G is the treatment solution absorbed by the sample in grams, C is the preservative or preservative solution in 100 g treatment solution, V is volume of sample in cm^3.

Table 2: Retention levels of fire retardant chemicals.



Veneer Species	Fire retardant Chemicals	Average retention (kg/m^3)	Veneer Species	Fire retardant Chemicals	Average retention (kg/m^3)
Beech (*Fagus orientalis* Lipsky)	Zinc borate	12.58	Alder (*Alnus glutinosa* subsp. *barbata*)	Zinc borate	15.80
	Borax	13.28		Borax	13.75
	MAP	8.71		MAP	10.60
	Ammonium sulfate	9.10		Ammonium sulfate	10.74
Poplar (*Populus deltoides* I-77/51 clone)	Zinc borate	13.49	Scots pine (*Pinus sylvestris* L.)	Zinc borate	18.22
	Borax	10.51		Borax	15.97
	MAP	10.76		MAP	9.06
	Ammonium sulfate	11.03		Ammonium sulfate	17.94

METHODS

Colour measurement

The colour measurements were carried out using a Minolta CM- 2600d spectrophotometer, equipped with an integrating sphere according to the CIE L*a*b* system (HAL 1996). Three samples from each group were used to determine the change of colour. Measurements were made over an 8 mm diameter spot with 10^{o} observer angle. The colour coordinates of each sample were measured before and after treatment of the veneer sheets at the same point. The ΔL^*, Δa^*, and Δb^* colour coordinates, therefore total colour change (ΔE^*) occurring in the specimens due to the treatment, were calculated on the tangential surface of replicates by comparing them to their initial values.

Surface roughness

A fine stylus-type profilometer, Mitutoyo Surftest SJ-301 Surface Roughness Tester was used for the surface roughness test. Rz parameter (mean of the 10-point height of irregularities) was used to evaluate surface roughness of the samples according to DIN 4768 (1990). Ten samples at dimensions of 300×300×2 mm were used for each test group to evaluate surface roughness. Measurements were taken perpendicular to the grain.

Thermal conductivity

The thermal conductivity of plywood was determined according to ASTM C-518 (2004). Two specimens with the dimensions of 300×300×2 mm were used for each group. The Lasercomp Fox-314 Heat Flow Meter shown in Figure 1 was used for the determination of thermal conductivity.

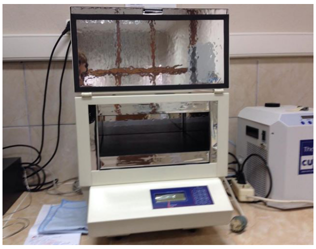


Figure 1: Lasercomp Fox-314 heat flow meter.

Thermogravimetric analyzes (TGA)

The thermogravimetric analysis measurements were carried out using a TG/DTA 6300 (Seiko Instruments Inc. - SII NanoTechnology Inc., Japan). Before measurements, the samples of each group were powdered. About 5-10 mg of the wood powder was weighed in a platinum cup and scanned from room temperature (20°C) to 800°C at a heating rate of 20°C/min. The samples were measured in an atmosphere of nitrogen. The maximum thermal degradation rate was calculated as mass (%) at peak temperature divided by peak temperature.

RESULTS AND DISCUSSION

Surface properties of veneer sheets

The Rz (mean of the 10-point height of irregularities) and the ΔL^*, Δa^*, and Δb^* colour coordinates, therefore total colour change (ΔE^*) occurring in the specimens due to the treatment, were calculated and these values are shown in Table 3.

Table 3: Surface properties changes of veneer sheets with treated different fire retardant chemicals.

Veneer species	Fire retardants	Rz	ΔL*	Δa*	Δb*	ΔE*
Beech (*Fagus orientalis* Lipsky)	Untreated control	90.28	-	-	-	-
	Zinc borate	112.07	2.29	-1.30	-4.56	5.27
	Borax	121.43	-11.57	5.25	9.53	15.87
	MAP	107.20	-4.55	4.43	9.67	11.57
	Ammonium sulfate	103.20	-4.83	4.62	10.26	12.23
Alder (*Alnus glutinosa* subsp. *barbata*)	Untreated control	70.92	-	-	-	-
	Zinc borate	82.28	0.37	-0.64	-12.87	12.9
	Borax	99.19	-12.27	2.73	0.28	14
	MAP	88.10	-9.47	2.61	-1.72	10.03
	Ammonium sulfate	94.50	-4.11	2.87	2.39	5.57
Poplar (*Populus deltoides* I-77/51 clone)	Untreated control	85.91	-	-	-	-
	Zinc borate	96.56	7.05	-3.28	-9.71	12.43
	Borax	126.26	1.46	-1.76	-0.87	2.48
	MAP	105.34	-4.90	3.09	7.69	9.67
	Ammonium sulfate	120.33	3.79	-2.27	0.66	4.48
Scots pine (*Pinus sylvestris* L.)	Untreated control	76.60	-	-	-	-
	Zinc borate	148.57	4.97	-5.52	-8.39	11.32
	Borax	91.35	-4.72	0.51	1.21	8.36
	MAP	111.07	-3.89	2.35	3.32	5.65
	Ammonium sulfate	95.65	-5.39	2.81	3.98	7.30

According to Table 3, the fire retardant chemicals increased the surface roughness of the veneer sheets. The highest values of surface roughness were found for beech, alder and poplar veneers treated with borax and scots pine veneers treated with zinc borate. Ayrilmis *et al.* (2006) reported that fire retardant chemicals increased the surface roughness of veneer sheets. Dundar *et al.* (2008) also found that surface roughness of LVLs made from fire-retardant treated veneers was significantly increased as compared to untreated control specimens and this difference increased with increasing drying temperature. The second drying process performed after the impregnation may also have contributed to increase in surface roughness. Aydin (2004) indicated that the effects of fire retardant chemicals on surface roughness varied according to steaming pre-treatment and veneer drying temperature.

Positive and negative values show a tendency of wood surface to become reddish and greenish for Δa^*, and yellowish and bluish for Δb^*, respectively (HAL 1996). Treatment with zinc borate caused surfaces to become greenish and bluish in the all of veneer sheets. Borax, monoammonium phosphate and ammonium sulfate caused surfaces to become reddish and yellowish both for beech and scots pine veneers. In literature, Scots pine showed the yellowish and reddish hues due to impregnation (Simsek and Baysal 2012). In the alder veneers, borax and ammonium sulfate caused surfaces to become reddish and yellowish while monoammonium phosphate caused surfaces to become reddish and bluish. Borax caused surfaces to become greenish and bluish while monoammonium phosphate caused surfaces to become reddish and yellowish in the poplar veneers. Also, ammonium sulfate caused surfaces to become greenish and yellowish. Negative lightness stability (ΔL^*) indicates a tendency of a wood surface to become darker (HAL 1996). Treatment with borax, monoammonium phosphate and ammonium sulfate caused surfaces to become darker in the all of veneer sheets except poplar. Simsek and Baysal (2012) found that borate treatments darkened both beech and Scots pine surfaces. In the poplar veneers, only monoammonium phosphate caused surfaces to become darker. The lowest darkening ($-\Delta L^*$) and total colour change (ΔE^*) were observed with borax for beech and alder veneers. Aydin and Colakoglu (2005) stated that the reason for this could be the water-soluble extractive contents of beech and alder wood. In the poplar veneers, these values were obtained from monoammonium phosphate and zinc borate, respectively. They were obtained from ammonium sulfate and zinc borate in the Scots pine veneers, respectively.

Thermal properties of veneer sheets

Thermal conductivity

According to wood species and fire retardant chemicals, the thermal conductivity mean values of veneer sheets are given in Figure 2.

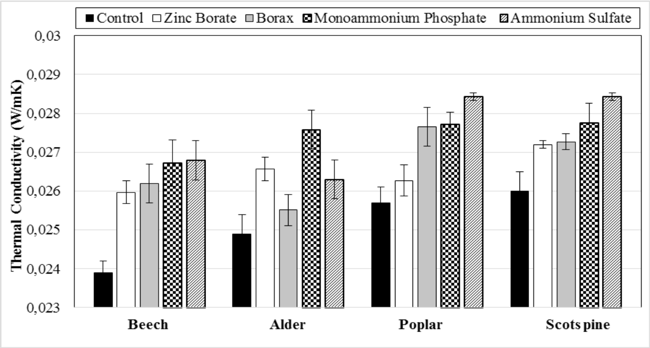


Figure 2: Thermal conductivity changes of the veneer sheets.

As can be seen from Figure 2, the highest values of thermal conductivity were obtained with ammonium sulfate for beech, poplar and scots pine veneers and monoammonium phosphate for alder veneers. When all test groups analyzed, untreated (control) groups were found to have the lowest thermal conductivity values. In the past studies, it was stated that thermal conductivity values of veneer sheets increased with impregnation, because the air in the cell lumen is replaced with a material having a better thermal conductivity (Kol *et al.* 2008). Another study suggests that fire retardants cause thermal conductivity to increase so that heat is absorbed by the chemical to prevent the wood surface from igniting (Rowell and Levan-Green 2005). Ozdemir *et al.* (2013) also found that thermal conductivity of high density fiberboard increased with using fire retardant chemicals and when the concentration of these chemicals increased, the thermal conductivity values increased. The differences among the thermal conductivity values in the study groups were very small. The reason for this is the low retention levels. Uysal *et al.* (2008) stated in their study that thermal conductivity values increased when retention levels were increased.

Thermogravimetric analyzes (TGA)

The temperature of the initial weight loss of pyrolysis (T_i), maximum degradation temperature (T_{max}), weight loss at maximum degradation temperature (W_{max}) and residual char at 800°C are given in Table 4. Figure 3 also show thermogravimetry curves according to wood species.

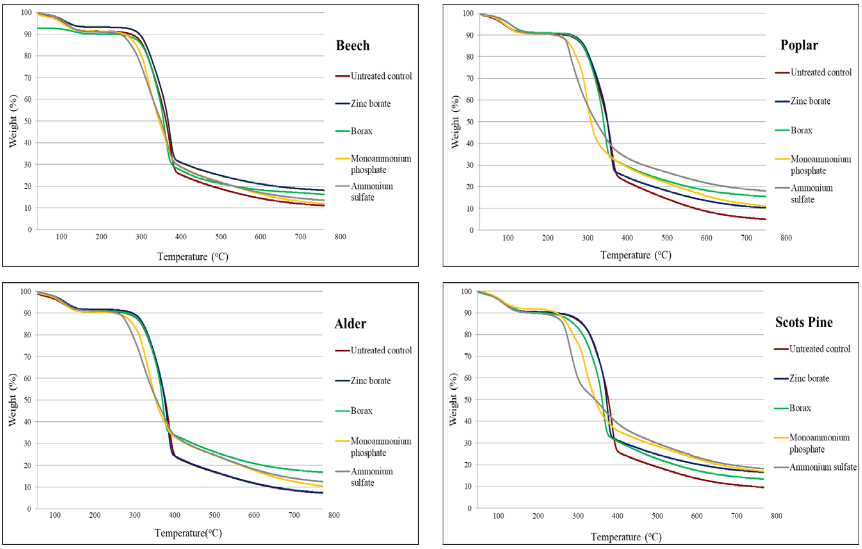


Figure 3: Thermogravimetric curves of veneers treated fire retardant chemicals.

Table 4: The results of thermal degradation of the veneers treated the fire retardant chemicals.

Veneer species	Fire Retardant Chemicals	Ti (°C)	Tmax (°C)	W max (%)	Residual char (%)
Beech (*Fagus orientalis* Lipsky)	Untreated control	229.5	368.9	62.24	11.09
	Zinc Borate	231.1	368	57.79	18.13
	Borax	228.7	351.4	57.1	16.2
	MAP	227.9	361.8	62.45	12.03
	Ammonium sulfate	227.6	361.7	61.4	13.56
Alder (*Alnus glutinosa* subsp. *barbata*)	Untreated control	233.9	370.3	64.15	7.25
	Zinc Borate	235.5	365.3	63.23	7.36
	Borax	233.4	349.7	54.29	16.73
	MAP	180.1	311.1	40.49	10.44
	Ammonium sulfate	178.7	358.2	58.75	12.47
Poplar (*Populus deltoides* I-77/51 clone)	Untreated control	231.4	370.4	64.63	5.02
	Zinc Borate	230.6	363.6	62.49	10.22
	Borax	234.5	347.8	55.24	15.54
	MAP	194.1	289.9	39.93	10.96
	Ammonium sulfate	184.7	238.7	19.41	18.12
Scots pine (*Pinus sylvestris* L.)	Untreated control	180.7	376.6	62.47	9.54
	Zinc Borate	177.5	360.5	57.1	16.47
	Borax	178.8	348.6	55.67	13.35
	MAP	180	290.5	33.63	16.97
	Ammonium sulfate	178.8	245.5	26.95	18.1

The sharp decrease in thermogravimetric curves for the untreated control groups was seen between 180-380°C due to degradation of cellulose, hemicelluloses and lignin. Sinha *et al.* (2000) reported that the hemicelluloses decomposed easily to form gaseous products (CO, CO_2, condensable vapors, and etc.) between 200-280°C. Degradation of the hemicelluloses also causes the organic acid especially acetic acid formation. Acetic acid formation accelerates the decomposition of polysaccharides by means of acting as depolymerization catalyst (Brosse *et al.* 2010, Esteves and Pereira 2008). In temperature range between 250-300°C, the other main components of the wood products (lignin and cellulose) are degraded and gas, tar and char are produced during the pyrolysis (Pétrissans *et al.* 2014). In this study, untreated control groups gave similar results.

There are many theories that proposed to explain the mechanism of fire retardancy of chemicals. These theories are classified into five categories: Barrier theories, thermal theories, dilution by noncombustible gases theories, free radical trap theories and increased char/reduced volatiles theories. Generally, fire retardancy mechanism of the chemicals could operate by the combination of these theories (LeVan 1984). The most acceptable theory which is called "chemical theory" suggests that the retardant chemicals operate by altering the pyrolysis of wood, increasing of char amount and causing the formation of less volatile and combustible vapors (LeVan and Winandy 1990). Also, according to Kollman and Cote (1968) all known flame retardant chemicals increased charring of wood at relatively low temperatures, thus improving thermal insulation. In Table 4, it was seen that all of the fire retardant chemicals gave higher char amounts than untreated control panels. As stated by NIIR (2017), fire retardant chemicals lower the temperature at which pyrolysis occurs, directing the degradation pathway toward more char production and fewer volatiles. The pyrolysis reactions generally occur at low temperatures for the treated wood compared to the untreated wood (LeVan and Winandy 1990, Holmes 1977). According to the temperature of the initial weight loss of pyrolysis (T_i) and maximum degradation temperature (T_{max}) in Table 4, the pyrolysis of untreated veneer sheets generally began at higher temperatures than for treated veneer sheets.

The highest residual char amounts were obtained from ammonium sulfate for poplar and Scots pine; zinc borate for beech; borax for alder,. Boron materials as fire retardant chemicals were investigated by several researchers during the last decades (Tomak *et al.* 2012, Tondi *et al.* 2014, Salman *et al.* 2014, Yuksel *et al.* 2014, Istek *et al.* 2013, Roth *et al.* 2007, Jin *et al.* 2014, Qu *et al.* 2011). Parallel to the previous studies, our results showed that the impregnation of boron materials (Zinc borate and borax) enhanced the resistance against fire (Uner *et al*. 2016). Use of borates promotes the formation of CO and decrease CO_2 amount during the pyrolysis (Uysal and Ozciftci 2004). Gao *et al*. (2006) stated that boron material is protected with a physical mechanism; the melted boron material results in a film layer which inhibits the access of oxygen and temperature to material.

CONCLUSIONS

Increased surface roughness values of the veneer sheets were obtained after fire retardant treatment. Among the fire retardant chemicals used in the study, boron treated groups exhibited the highest surface roughness values. Surface roughness of veneer sheets is extremely important on the product quality. Also, the fire retardant chemicals caused colour changes on surface of veneer sheets. It can significantly affect the wettability of the surface, the quality of gluing, and adhesion in plywood manufacture. Therefore, the effect of fire retardant chemicals on the surface roughness should be considered during manufacturing.

It was observed that fire retardant chemicals increased the thermal conductivity values of the veneer sheets when compared to the control groups of each wood species. Among the fire retardant chemicals used in the study, ammonium treated test groups gave the highest thermal conductivity values compared to boron treated groups. Fire retardant chemicals may increase the thermal conductivity of the wood to dissipate the heat from the surface faster than it is supplied by igniting source, or they may affect chemical and physical changes so that heat is absorbed by the chemical, preventing the wood surface from igniting (NIIR 2017). The increase of thermal conductivity can provide a significant advantage especially in the use of conductivity required areas such as wood buildings. According to TGA results, all fire retardant chemicals increased residual char amounts. Among the fire retardant chemicals used in the study, boron treated groups gave the highest residual char amounts for beech and alder. For poplar and Scots pine, highest residual char was observed in ammonium sulfate treated groups.

ACKNOWLEDGEMENTS

Some results of this study were presented in Third National Furniture Congress held by Selçuk University, Konya, 2015. The authors acknowledge the partial financial support of this study by TUBITAK (The Scientific and Technical Research Council of Turkey) (Project No: 112O819).

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