

**THERMAL CHARACTERISTICS OF ORIENTAL BEECH
WOOD TREATED WITH SOME LEACHING RESISTANT
BORATES**

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ABSTRACT

It was aimed to investigate thermogravimetric analysis (TGA), differential-thermogravimetric (DTG), and differential-thermal analysis (DTA) of Oriental beech (*Fagus orientalis* L.) wood treated with some leaching resistant borates such as sodium tetraphenylborate (STPB) and phenylboronic acid (PBA). In this study; 0.25, 1.00, and 4.70 % aqueous solutions of borates were prepared.

Results of the study indicated that pyrolysis occurred in three stages for STPB and PBA treated Oriental beech wood. Generally, while concentrations of the STPB and PBA increased, T_i and T_{max} values of STPB and PBA treated wood specimens decreased. Residual char contents of STPB and PBA treated wood specimens for all concentrations were higher than untreated control specimen.

KEYWORDS: Thermal characteristics, leaching resistant, borates, Oriental beech, residual char, pyrolysis.

INTRODUCTION

Wood is a sustainable and environmentally friendly natural material widely used as a construction material (Tomak et al. 2012, Trisna and Hiziroglu 2013, Turkoglu et al. 2015). However, wood is much more easily degraded by environmental agents, including fire, biological organisms, water, and light (Kiguchi and Evans 1998). These problems can be partially overcome by impregnation of the wood (Tomak et al. 2011). Still today, there is a general afraid in the public opinion about the impact of wood preservation formulations on the environment and human health. Boron-based systems are candidates for the future range of preservative formulations with lower environmental and health impacts (Caldeira 2010). Boron compounds have been used for wood preservation since the early twentieth century because of easily applicable, biologically active, flame retardant and, more importantly environmentally safe preservatives (Williams 1990, Lloyd 1993, Laks and Manning 1994, Baysal and Yalinkilic 2005). Boron wood preservatives have several great advantages for application as wood preservatives including a broad spectrum of activity against insects and fungi, low mammalian toxicity, low volatility, and they are colorless and odorless (Murphy 1990, Yalinkilic et al. 1999). Thus, borates have played an ever-increasing role in the preservation of wood worldwide (Freeman et al. 2009) and wood preservation researchers have refocused on boron in the last two decades (Obanda 2008). Recent studies more focused on improvements to the application methods and inclusion of borates in remedial treatments. Researches continue to develop borate formulations that have increased resistance to leaching while maintaining biocidal efficacy (Robinson et al. 2005). It is also well known that boron compounds work efficiently as fire retardant chemicals for cellulosic materials. Fire-retardant chemicals drastically reduce the rate at which flames travel across the wood surface, thereby reducing the capacity of the wood against fire (Levan and Tran 1990, Levan and Winandy 1990). The fire retardant effectiveness of boron compounds is a physical mechanism achieved by the formation of a coating or protective layer on the wood surface at high temperature, and by forming glassy films that may inhibit mass transfer of combustible gases (Levan and Tran 1990, Wang et al. 2004). Despite the many advantages of boron compounds, they have limited utility in outdoor applications because they are easily leached out from impregnated wood when exposed to water or rain (Yalinkilic 2000). For this reason, combination of boron compounds with other wood preservatives has been gained popularity (Temiz and Yildiz 2002). In literature, several strategies have been proposed to limit leaching of borates for increasing applicability of the borates. Common strategies of decreasing leachability of borates are using organo boron compounds and precipitation of organo-borate salts. It was reported that the aromatic boronic acids decreased leaching from wood by means of interaction with aromatic parts of lignin and restriction of access of water. Phenylboronic acid (PBA) is the most common studied ones among the aromatic boronic acids (Obanda et al. 2008). It has recently proved promising for leaching resistant boron compound (Liu et al. 1994; Yalinkilic et al. 1997a, b, 1998; Manning et al. 1997). In the case of precipitation of organo-borate salts, organo borate compound, which forms an insoluble complex, is applied. The most common example of this application is using sodium tetraphenylborate (STPB). Chemical structures of both of these compounds are illustrated in Fig. 1.

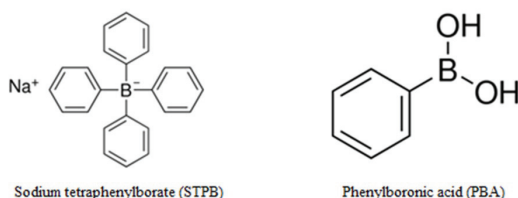


Fig. 1: Chemical structures of STPB and PBA.

The investigation was carried out by Yalinkilic (2000) about the fungal and termiticidal efficacy of PBA-impregnated Sugi (*Cryptomeria japonica* D. Don) wood and tested in terms of boron leachability from treated Sugi wood. Decay test results of the study showed that PBA-treated Sugi wood specimens were resistant to both decay fungi before and after leaching. Moreover, PBA-treated wood specimens gave perfect termiticidal performance before and after leaching. Ion chromatography analysis of hot water extract of impregnated Sugi wood before and after leaching showed that PBA is considerably resistant to water leaching. Lin et al. (2001) studied the double impregnation of Japanese cedar (*Cryptomeria japonica*) by tetraphenylborate sodium salt with potassium chloride or tetramethylammonium bromide to form insoluble salts of tetraphenylborate tetramethylammonium or tetraphenylborate potassium in order to reduce the leachability of boron compounds in wood. The reduction of boron retention in impregnated wood specimens after the water-leaching test was very small, showing the good fixation of boron compounds in wood. In addition, this treatment indicated an excellent decay resistance against both *C. versicolor* and *T. palustris*. However there are not enough systematic studies on fire retardant that limit or eliminate the potential for these chemicals. Therefore, knowledge of the thermal degradation and fire performance of wood treated with these chemicals can be critical. Various methods have been developed for evaluating the effectiveness of fire retardant treated wood and the most common of them; thermal analysis is a simple, convenient, reproducible and fast method for evaluating the pyrolysis and flame retardants under air or inert gas flow (Tomak et al. 2012, Liodakis et al. 2003, Tsujiyama and Miyamori 2000). Wood is exposed to thermal degradation reactions under the effect of increased temperatures using differential thermal analysis (DTA) and thermogravimetric (TG) techniques at heating rates 20 and 30°C·min⁻¹ in temperature range 30–650°C (Kiziltas et al. 2011, Gao et al. 2004, Wielage et al. 1999). Jin et al. (2014) investigated fire resistance of Southern pine impregnated with boron-containing fire-retardant by TG analysis. The results showed that boron-containing fire-retardant improved Southern pine fire retardant performance, the mass loss rate decreased, the char yield and the peak temperature of maximum mass loss increased. Ozdemir and Tutus (2013) studied thermogravimetric analysis (TGA) of HDP panels impregnated with some aqueous solutions of boric acid and borax. The results of TGA revealed that weight losses when using borates were lower than the untreated control wood. Increasing the concentration ratio resulted in a decrease in combustion amount.

The aim of this study was to evaluate the thermal behavior of Oriental beech (*Fagus orientalis* L.) wood treated with 0.25, 1.00, and 4.70 % aqueous solutions of sodium tetraphenylborate (STPB) and phenylboronic acid (PBA) wood preservatives by thermogravimetric analysis (TGA), differential thermogravimetric (DTG), and differential thermal analysis (DTA).

MATERIALS AND METHODS

Materials

Sodium tetraphenylborate (STPB) and phenylboronic acid (PBA) were obtained from chemical materials suppliers. Sapwood of Oriental beech (*Fagus orientalis* L.) timber free of knots, excessive cross-grain was machined into narrow strips. The strips were carefully chosen for having the same annual ring and then cut into small pieces prior to milling. Wood flour was prepared by grinding the small wood pieces in a Wiley mill with a 50 meshes. Before impregnation, the specimens were conditioned at 65 % relative humidity and 20°C for two weeks.

Impregnation method

Aqueous solutions of the wood preservatives having concentration of 0.25, 1.00, and 4.70 % were prepared using distilled water for the impregnation procedure. The wood flour approximately 100 g was immersed in the solutions at 60°C for 2 h. The treated wood specimens were subsequently dried at 60°C until they had the unchangeable weight. Similar impregnation procedure of wood flour and wood specimens are described in TG and DTA studies on fire retardant treated wood by Jiang et al. (2010) and Yunchu et al. (2000). The treated wood specimens were then moisture conditioned for two weeks at 20°C and 65 % relative humidity.

Thermal analysis

Differential thermal analysis (DTA) and thermogravimetric (TG) were carried out under argon at a heating rate of 10°C·min⁻¹ and a purge rate of 50 mL·min⁻¹. The temperature was heated from the room temperature to 600°C. During the heating and pyrolysis of about 10 mg of wood specimen, the weight loss was monitored continuously. Onset and inflection temperatures of the pyrolysis were recorded by the analyzer for each treatment group. The rate of weight loss as a function of time was derived from TG curve resulting in a derivative TG curve.

RESULTS AND DISCUSSION

Thermal degradation of wood could be evaluated into three stages. In first stage, below 200°C, the volatile components (physically bound water and essential oils) of the wood were eliminated. Second stage could be described as main degradation stages. In this stages hemicelluloses, celluloses, and lignin are degraded simultaneously between 200-378°C. In third stage, remaining (non-volatile and non-combustible) part of the wood was carbonized and it was converted into tar and char, up to 600°C (Beall and Eickner 1970, Tomak et al. 2012). The thermal properties of Oriental beech wood treated with (0.25, 1.00, and 4.70 %) STPB and PBA were investigated by TG, DTG, and DTA under argon atmosphere with heating rate of 10°C·min⁻¹. Obtained results were compared with the non-treated Oriental beech wood called as control. Important parameters, the temperature of the initial weight loss of pyrolysis (T_i), the maximum degradation temperature (T_{max}), and Residual char are given in Tab. 1. Figs. 2 and 3 illustrate the TG curves, first derivative of TG curves (DTG) and DTA curves of STPB and PBA treated Oriental beech wood, respectively.

Tab. 1: T_i (the temperature of the initial weight loss of pyrolysis), T_{max} (maximum degradation temperature) and residual char of STPB and PBA treated Oriental beech wood.

Chemicals	Concentration (%)	T_i (°C)	Residual char content (%)	T_{max} (°C)
Control	-	231	33	353
Sodium tetrabphenylborate	0.25%	213	39	335
	1.00%	178	41	320
	4.70%	176	43	297
Phenylboronic acid	0.25%	211	41	327
	1.00%	204	48	327
	4.70%	185	48	274

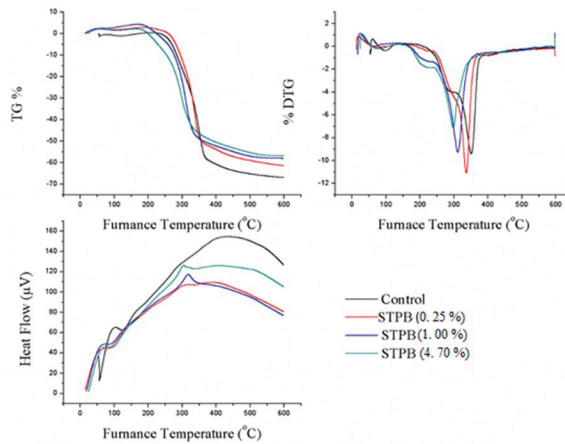


Fig. 2: TG curves, first derivative of TG curves (DTG) and DTA curves of STPB treated Oriental beech wood with different concentrations (0.25, 1.00, and 4.70 %).

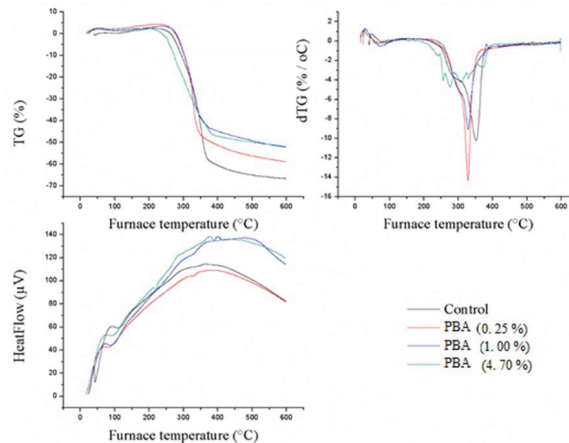


Fig. 3: TG curves, first derivative of TG curves (TGA) and DTA curves of PBA treated Oriental beech wood with different concentrations (0.25, 1.00, and 4.70 %).

In parallel with the general consideration, for the untreated Oriental beech wood, there were three different steps observed in TG curve. First step was below 231°C, second step was between 231 to 367°C and third step was between 367 to 600°C. In first stage, there was no considerable mass loss could be observed in spectrums. In this stage, only at 110°C small endothermic peak could correspond to evaporation of physically bounded water was observed in DTA curves. Main mass loss was observed for the untreated wood specimen in second stages. Nearly 57 % of initial weight of untreated sample decreased in this stage. Decrease in weight during this stage arises from the thermal decomposition of cellulose, hemicelluloses, and lignin. Cellulose, hemicelluloses, and lignin are main building blocks of woods. Hemicellulose is the most heat sensitive one among them. In literature, it was reported that the hemicelluloses thermally degraded between 200–280°C. It decomposed into CO, CO₂, condensable vapors, and organic acids (Sinha et al. 2000). Organic acids especially acetic acid formation act as catalyst which improve the degradation of polysaccharides (Brosse et al. 2010, Esteves and Pereira 2008). At temperatures between 250 to 367°C, the other components (lignin and celluloses) were decomposed. In literature, it was reported that the lignin and celluloses are decomposed between the temperatures 250 to 350°C and produce tar and char during thermal degradation in inert atmosphere (Pétrissans et al. 2014). At 600°C, char content of the untreated wood specimen was 33 % of initial weight, initial degradation temperature was found as 231°C and T_{max} of the untreated wood specimen was 353°C.

Besides the wood products are flammable, they are also subjected to biological attacks and negative effects of environmental conditions. To prevent wood products from fire, fungus, termites, and other insects, chemicals called as wood preservatives are used (Ajuong and Pinion 2010). Borates are good candidate for the protection of wood against the fire and biological attacks. Borates have several advantages such as improving flame retardancy, providing sufficient preservation against fungus and termites, having low toxicity. Although having all these advantages, borates could diffuse through the environment from the treated wood with the rainfall (Simsek et al. 2010). Fig. 2 shows the TG curves, DTG curves, and DTA curves of 0.25, 1.00, and 4.70 % STPB treated Oriental beech wood. Pyrolysis of wood specimens occurred in three stages. As same for the untreated wood specimen, there was no significant mass loss observed for the treated wood specimens in first stages. Only in DTA curve nearly at 100°C, endothermic peak related with the dehydration of wood was seen. As seen in Figures, for the STPB treated wood specimens, boundaries of the stages were different. Both T_{max} and T_i values of the wood specimens were decreased with the increasing of applied concentration of STPB. T_{max} values of the 0.25, 1.00, and 4.70 % STPB treated wood specimens were found as 335, 320, and 297, respectively and T_i values of the wood specimens were found as 213, 178, and 176°C in same order. Char content of the wood specimen was found as 39, 41, and 43 % of initial weight of 0.25, 1.00, and 4.70 % STPB treated wood specimens, respectively. Char content of the wood specimens were proportional with the increasing concentration of STPB. In DTA curves of 1.00 and 4.70 % STPB treated Oriental beech wood specimen exothermic peak was observed nearly at 315°C. This peak was related with the decomposition of STPB. Same observation for STPB was considered in air atmosphere in literature (Ninan and Nair 1976). In literature, we could not find enough data related with thermal gravimetric analysis of STPB treated woods. Thermogravimetric pyrolysis of ammonium and alkali metal-TPB was studied by Wendlandt (1956). It was found that the pyrolysis of alkali metal tetraphenylborates starts at different temperature and the temperature was related with the ionic radius of the metals.

Fig. 3 shows the TG curves; DTG curves and DTA curves of 0.25, 1.00, and 4.70 % PBA treated Oriental beech wood. In Tab. 1, T_i , T_{max} and % residual char content values of PBA

treated wood specimens were listed. Initial degradation temperatures (T_i) decreased with the increasing of the PBA concentration and T_{max} values decreased with the increasing of PBA concentration, too. Percent of char content at 600°C of the treated wood specimens are higher than the untreated wood specimen. Other important observation was seen in Tab. 1 that the percent of residual char content of 1.00 and 4.70 % PBA treated wood specimens were found as same. It could be said that the higher than the 1.00 % of PBA impregnation did not improve the protection against the heat. In Fig. 3, it was seen that the degradation took place in three stages. In first stage, there was no significant changes could be observed as same for the STPB treated and untreated wood specimen. And like other wood specimens, only in DTA curves endothermic peaks could be related with desorption of physically bound water were seen for the PBA treated wood specimens. Main weight loss for the wood specimens were observed in second stage between temperatures 230-380°C. As seen In DTG curves, for the 4.70 % PBA treated wood specimen, multiple degradation steps were seen. These multiple steps might be related with the decomposition of PBA and reactions of pyrolysis products of PBA. In literature, PBA was also used as carbohydrate receptors and it was employed in electrochemical and optical sugar sensors (Egawa et al. 2011, 2014). PBA reacted with carbohydrates OH- groups and formed boronate esters with them (Pappin et al. 2012). In our study from the obtained results, it was thought that chemically bound PBA was more stable than the unbounded ones. So, for the 4.70 % PBA loaded wood specimens, multiple degradation steps might arise from the degradation of unbound PBA. Thermo-degradation of PBA and its salt was studied in literature by Fields and Doyle (1974). They concluded that the thermal degradation of PBA and its derivatives in air and in nitrogen atmosphere took place through a degradation of series of intermediate compounds to the anhydrous metaborate salt (Fields and Doyle 1974). Chemical compounds that containing boron generally employed on surface treatment of woods as fire retardant. In literature, fire retardancy properties of borates were investigated by several researchers (Istek et al. 2013, Jin et al. 2014, Roth et al. 2007, Salman et al. 2014, Tomak et al. 2012, Tondi et al. 2014, Yuksel et al. 2014). Mostly, borates melt at lower temperature and form glassy B_2O_3 films on surface of the wood during the pyrolysis. This film insulates the woody product and protects wood from the heat, and this protective layer decreases the amount of combustible vapor generation during the pyrolysis. All of these phenomena promote the charring reactions. In our study, char content of STPB and PBA treated wood specimens were higher than untreated wood specimens. So, it could be said that the STPB and PBA treatment promoted the resistance of Oriental beech wood against to heat.

CONCLUSIONS

In this study, thermogravimetric analysis (TG), differential thermogravimetric (DTG), and differential thermal analysis (DTA) techniques were performed for investigation of thermal behavior of Oriental beech wood treated with 0.25, 1.00, and 4.70 % aqueous solutions STPB and PBA under inert atmosphere. Leaching of these compounds is limited and they could be considered as the solutions of fixation problem of borates. Despite the leaching of these compounds from the wood specimens and physical properties of STPB and PBA treated discussed in literature (Obanda et al. 2008), thermal properties of STPB and PBA treated wood were not fully investigated. So, our study may help to fill this gap in literature.

Our results showed that T_i and T_{max} values of untreated control specimen were higher compared to STPB and PBA treated Oriental beech wood specimens. Moreover, in general, T_i

and T_{\max} values of STPB and PBA treated Oriental beech wood specimens decreased with the increasing of concentrations of the both borates. According to our results, residual char contents of STPB and PBA treated wood specimens were higher than that of control specimen. Residual char content of the wood specimens were found as 39,41 and 43 % of initial weight for the 0.25, 1.00, and 4.70 % STPB treated wood specimens respectively and 41, 48, and 48 % of initial weight for the PBA treated wood specimens in same order with STPB. For the further investigations, gas products of pyrolysis should be investigated for better understanding.

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