

THERMAL ANALYSIS OF ORIENTAL BEECH SAWDUST TREATED WITH SOME COMMERCIAL WOOD PRESERVATIVES

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ABSTRACT

In this study, investigation of the thermal properties of Oriental beech (*Fagus orientalis*) sawdust treated with 0,25; 1 and 4,70% aqueous solutions of Adolit KD-5, Wolmanit CX- 8 and Tanalit-E were performed by using thermogravimetric analysis, differential-thermal analysis, and differential-thermal analysis under argon atmosphere.

Results were compared with the untreated wood (control). It was found that the treatment with Adolit KD-5, Wolmanit CX- 8 and Tanalit-E decreased the T_{max} (maximum degradation temperature) and increased residual char amount with respect to the control sample. Increases in the concentration of applied preservatives promote the char formation. It was found that the char content after pyrolysis experiment had good agreement with the boric acid amount in wood preservatives.

Keywords: Differential-thermogravimetry, *Fagus orientalis*, thermogravimetric analysis, differential-thermal analysis, residual char.

INTRODUCTION

Wood is an important renewable material with excellent physical and mechanical properties (Salca and Hiziroglu 2014). It has been extensively used for aesthetic, engineering, and structural applications (Bhat *et al.* 2010, Lesar *et al.* 2011). As wood is a natural organic material, it is subject to biological and non-biological factors such as insects like termites, decay fungi, mould, weathering, and fire in application fields. Thus, wood products require protection from these factors in order to provide reliable service. Wood protection technology generally involves the modification of wood products to improve protective properties. The most commonly used method of wood protection is chemical treatment which involves the impregnation of chemical substances into the wood (Poncsák *et al.* 2006, Ajuong and Pinion 2010). Preservative treatment is effective and can extend the service life of wood and wood products (Ahmed and Moren 2012). However, use of many of the effective poisonous chemicals is also questionable. Increased public concern on health and the environmental effects of many wood preservatives have emerged. The focus on copper-based preservatives has increased following concerns about environmental effects of chromium and arsenic and resulting restrictions on the use of chromated copper arsenate (CCA) (Freeman and McIntyre 2008). Chromated copper arsenate (CCA) has been used for wood preservation for more than 30 years but in the last decade the use of CCA has been restricted in Europe, North America, and Japan due to its toxicity against humans, animals, and environment (Palanti *et al.* 2011). Many of the alternative preservatives contain copper as their active ingredient against fungal decay. Copper compounds are very effective against numerous fungi and are the basis of numerous formulations of wood preservatives (Mourant *et al.* 2008). They

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also have advantages: it is relatively easy to create waterborne formulations; it is easy to analyze and determine penetration in wood, and copper slows photodegradation by UV radiation and water (Archer and Preston 2006). At present, the new generation of copper containing preservatives such as Tanalith-E (TN-E) and adolit-KD5 (AD KD-5) are being used in the forest products industry instead of CCA (Turkoglu *et al.* 2015). They are copper salts with others “co-formulating” substances, such as boron, azoles and organic compounds (Palanti *et al.* 2008). It is generally known that new copper containing wood preservatives protect wood against fungal decay and insects and prevent photodegradation. However, there are not enough systematic studies on fire retardancy of preservative treated wood. Most of the preservatives include copper and chromium ions which can promote glow combustion and they also include boric acid and its derivatives used in fire retardants. So, wood preservatives can have positive or adverse effects on thermal properties of woods. Fundamentally, wood has been used in house interiors, building or public transport constitutes a potential hazard for people in case of fire (Gao *et al.* 2005). Therefore, knowledge of the thermal degradation and fire performance of wood treated with these chemicals can be critical (White and Dietenberger 2001) and attract more attention than ever before in order to comply with the safety requirements (Lowden and Hull 2013, Jiang *et al.* 2015).

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 (Liodakis *et al.* 2003, Tsujiyama and Miyamori 2000). Thermal decomposition characteristics of wood components are different because of differences in their chemical structure. Hemicellulose, cellulose, and lignin decompose in distinctive temperature range. Hemicellulose decomposes easily with respect to other components of wood. It decomposes at temperature between 200-280°C (Sinha *et al.* 2000). In temperature range between 250-300°C, the other main components of the wood products (lignin and cellulose) degrade (Salman *et al.* 2014). It was reported that the wooden materials generally separate into three stages during the pyrolysis. First stage is removal of volatile parts below 200°C; second stage is degradation of hemicelluloses, celluloses, and lignin between 200-378°C; and third stage is conversion of nonvolatile and noncombustible part into tar and char, up to 600°C. Wood is exposed to thermal degradation reactions under the effect of increased temperatures using differential thermal analysis (DTA) and thermogravimetry (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). The temperatures at which decomposition reactions of wood start and the changes in sample weight with the reactions can be followed (Yorulmaz and Atimtay 2009). The thermal conversion process is generally conducted in a chamber in one of the three ways; pyrolysis, gasification, and combustion or incineration (Chandrasekaran and Hopke 2012, Helsen and Van den Bulck 2005). Pyrolysis is defined as the thermal degradation of carbonaceous materials in the absence of oxygen and is a possible thermochemical conversion (Di Blasi 2008, Chandrasekaran and Hopke 2012). The mechanism of fire retardant efficiency can be well understood by studying the pyrolysis process of wood treated with fire retardants. Using the fire-retardant chemicals can cause pyrolysis reactions to form more char and water, and therefore reduce the temperature of the pyrolysis as well as reduce the yield of the flammable gases (Yunchu *et al.* 2000). Thermogravimetric analysis studies were carried out by several researchers about thermal degradation of wood treated with fire retardant chemicals (Yunchu *et al.* 2000, Baysal 2002, Jiang *et al.* 2010, Tomak *et al.* 2012, Jiang *et al.* 2015). Deka *et al.* (2002) investigated the resistant capacity of a chemically treated hard wood, *Anthocephalus cadamba* (Roxb) Miq. to thermal degradation using TGA and DTG techniques. They found that treated wood was thermally more stable than the untreated wood. Wu *et al.* (2014) researched flame retardancy and thermal degradation behavior of red gum wood treated with hydrated magnesium chloride. They reported that treatment with hydrated magnesium chloride decreased flame intensity and heat release rate, and reduced smoke concentration and gas yield. Uner *et al.* (2016) investigated thermal behaviour of borate treated Oriental beech wood. They found that borate treatment decreased the T_{max} (maximum degradation temperature) and increased residual char amount. Higher concentration levels of borates resulted in higher char content of Oriental beech wood specimens. Wang *et al.* (2004) studied thermal characteristics of basswood (*Tilia amuresis*) impregnated with boric acid. They found that treatment of wood with boric acid resulted in increased weight loss at lower temperatures and decreased weight loss at higher temperatures.

Wood products are commonly used materials in daily life. Generally, wood products are used as its treated form because of durability and cosmetic concern. Most chemicals used as preservatives and

coatings affect the thermal behavior of wood. So, it is crucial to have knowledge about the effect of heat on treated woods. Thermal behavior of wood is crucial because of safety and statutory regulations so, the research's have to be performed for each chemical used as preservatives or another purposes. Thermal behaviour of Oriental beech (*Fagus orientalis*) wood treated with aqueous solutions of Adolith KD 5, Wolmanit CX 8 and Tanalith -E have not been investigated yet in literature. The aim of this study is to evaluate the thermal behavior of Oriental beech wood treated with 0,25%, 1%, and 4.70% aqueous solutions of Adolith KD 5, Wolmanit CX 8 (WCX-8), and Tanalith-E by thermogravimetry analysis (TGA), differential thermogravimetry (DTG), and differential thermal analysis (DTA).

MATERIALS AND METHODS

Materials

Sapwood of Oriental beech (*Fagus orientalis* L.) timber free of knots, excessive cross-grain, and other obvious defects was obtained from Yucel Wood Products Industry, located in Mugla, the South West region of Turkey. Oriental beech timber 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. Diameter of obtained particles was smaller than the 288 μm . AD KD-5, WCX-8, and TN-E were used as impregnation chemicals. Before impregnation, the samples 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%, 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. Samples were collected by filtration and the treated wood samples were subsequently dried at 60°C until they had the unchangeable weight. Similar impregnation procedure of wood flour and wood samples is described in TG and DTA studies on fire retardant treated wood by Jiang *et al.* (2010) and Yunchu *et al.* (2000). The treated wood samples were then moisture conditioned for two weeks at 20°C and 65% relative humidity.

Thermal analysis

Thermogravimetry analysis (TGA), differential thermogravimetry (DTG), and differential thermal analysis (DTA) were carried out under argon at a heating rate of 10°C/min and a purge rate of 50 mL/min using a LABSYS TG-DTA analyzer (France). The temperature was heated from the room temperature to 600°C. During the heating and pyrolysis of about 10 mg of sample, 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 DISCUSSIONS

Commercial wood preservatives include numerous chemicals that affect the final properties of the woods. The composition of AD KD-5, WCX-8, and TN-E were listed in Table 1.

Table 1. Composition of the commercial wood preservatives.

Wood preservatives	Component (amount %)
AD KD-5	Copper(II)hydroxidecarbonate 20,33% Didecylpolyoxoethylammoniumborate 10% Boric acid 8% Alpha-iso-Tridecyl-omega-hydroxy-polyglycolether 1-2,5%
WCX-8	Bis-(N-cyclohexyldiazoniumdioxy)-copper 2,8% Copper hydroxide carbonate 13,04% Boric acid 4,0% 2-Aminoethanol 20-40%
TN-E	Copper carbonate 20,5% w/w 2-aminoethanol < 20% w/w Boric acid < 5% w/w Tebuconazole < 20% w/w Organic acid < 5%w/w Polyethyleneamine < 20% w/w Surfactant < 5%w/w

The TG curves, first derivative of TG curves (DTG) and DTA curves of Oriental beech wood treated with (0,25%; 1% and 4,70%) AD KD-5, WCX-8, and TN-E are shown in Figure 1. The obtained results were compared with the non-treated Oriental beech wood called as control. The temperature of the initial weight loss of pyrolysis (T_i), maximum degradation temperature (T_{max}) and residual char content are given in Table 2.

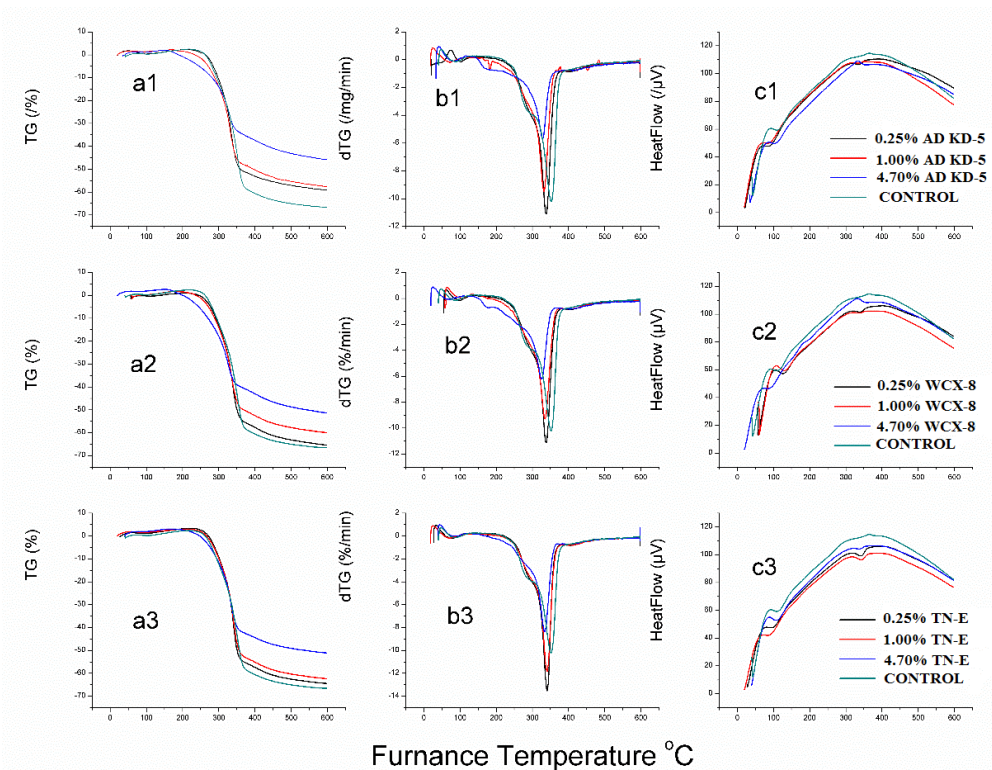


Figure 1. TG curves (a1, a2, a3), first derivative of TG curves (TGA) (b1, b2 and b3) and DTA (c1, c2, c3) curves of (1) AD KD-5, (2) WCX-8 and (3) TN-E impregnated Oriental beech wood with different concentrations (0,25%; 1 % and 4,70%).

As seen in Figure 1, there was no significant weight loss observed in TG and DTG curves for the control sample below 200°C. At nearly 110°C, endothermic peak corresponds to evaporation of physically bound water was observed from the DTA curves. Sharp decrease observed in TG curves between 231-350°C for the untreated sample could be related to the degradation of cellulose, hemicelluloses, and lignin. As mentioned before, hemicellulose decomposed easily with respect to the other components. Pyrolysis of hemicellulose results in formation of CO, CO₂, condensable vapors, and organic acids between 200-280°C (Sinha *et al.* 2000). Organic acids especially acetic acid formation acts as depolymerization catalyst and increase the decomposition of polysaccharides (Brosse *et al.* 2010, Esteves and Pereira 2008). Until the temperature reached 370°C, other components continued to degrade. At higher temperature, charring reaction occurred. As seen in Table 2, residual char content and T_{max} values were calculated as 33% and 353°C respectively for the untreated sample.

Table 2. The temperature of the initial weight loss of pyrolysis (T_i), maximum degradation temperature (T_{max}) and Residual char of AD KD-5, WCX-8, and TN-E impregnated Oriental beech wood.

	Concentration	T _i (°C)	Residual Char Content %	T _{max} (°C)
CONTROL	-	231	33	353
AD KD-5	0,25%	229	41	332
	1,00%	207	42	330
	4,70%	182	54	327
TN-E	0,25%	230	36	339
	1,00%	226	38	339
	4,70%	212	49	331
WCX-8	0,25%	216	35	336
	1,00%	207	40	333
	4,70%	165	49	330

TG, DTG and DTA curves of AD KD-5 WCX-8, and TN-E impregnated Oriental Beach wood with various concentration are seen in Figure (a1, b1 and c1); (a2, b2 and c2) and (a3, b3 and c3) respectively. Below 200°C, no observable changes could be seen in TG curves of all commercial wood preservative treated samples but as same in DTG curves of untreated wood sample endothermic peaks at nearly 80-110°C were observed in figure c1, c2 and c3 for all concentrations, and for all treated wood samples, exothermic peaks between 340-350°C were seen. These peaks arisen from the transition of amorphous B₂O₃. Although crystalline boronoxide melt at 450°C, it was reported that the amorphous boron oxide soften at 325°C (Sevim *et al.* 2006). In Table 1, the composition of wood preservatives is listed. From the table, it is easily seen that there are small amounts of boric acid in AD KD-5, WCX-8, and TN-E. As seen in DTG curves (Figure b1, b2 and b3) degradation rates decreased with the increasing concentration of AD KD-5, WCX-8, and TN-E. T_i, residual char content, and T_{max} values of AD KD-5, WCX-8, and TN-E treated wood samples are listed in Table 2. As seen in the Table, initial degradation temperature decreased with the increasing of applied concentrations. Residual char content of all treated wood sample were higher than untreated wood sample and residual char content of treated wood sample increased with increasing applied preservative concentration. T_{max} values were found

inversely proportional with the increasing concentration of AD KD-5, WCX-8, and TN-E.

As seen in Table 2, proportional with the boric acid content of wood preservatives, residual char contents of 4,70% AD KD-5, WCX-8, and TN-E impregnated wood sample were found 54, 49, and 49% respectively. Commercial wood preservative includes Cu and pesticides for protecting wood from fungus and other pest. They also included boric acid which is also known as flame retardant. Presences of borates caused formation of a protective layer and protects the wood from high temperatures. Because of these reasons, the char amount of treated Oriental beech wood increased with respect to untreated sample. It was thought that the residual char content was in good agreement with the boric acid amount in wood preservatives. It was reported that the inorganic salts change the pyrolysis process depending on the salt type and substrate. Metals, metal salts or metal complexes in the formulations of wood preservatives applied on woods increase char yield (Tomak *et al.* 2012).

CONCLUSIONS

Pyrolysis of Oriental beech sawdust treated with 0,25; 1 and 4,70% aqueous solutions of AD KD-5, WCX-8, and TN-E was monitored by thermogravimetric analysis under argon atmosphere in this study. Thermogravimetric analysis (TG), differential thermogravimetric analysis (DTG), and differential thermal analysis (DTA) were discussed in this article.

Our results showed that increase in applied concentration of boric acid containing wood preservatives decreased the T_{max} (maximum degradation temperature) and increased residual char amount. Residual char contents of the treated wood sample were higher than untreated wood sample. The highest residual char content was found for the sample treated with 4,70% AD KD-5 among the all treated wood sample. Residual char content of preservative treated wood sample at fixed concentration of 4,70% ranked as in the following order: AD KD-5 > WCX-8 and TN-E > Control. It was also found that T_i values was lower than the untreated wood sample.

In this work, we focused on thermal characteristics of Oriental beech sawdust samples and effects of preservatives on structural constituents during the heat treatment. So, homogeneity of the wood samples was important because of the reliability of the result. It is clear that the desired homogeneity could not be achieved when the solid wood is used. Thermal characteristic of preservative treated solid wood should be tested for actual effects of heat on wood products. The thermo-gravimetric analysis is fast, efficient and effective way to test the performance of the chemicals against fire. But still combustion tests and/ or limited oxygen index test (LOI) should be done for better understanding of behavior of chemicals against the fire. For the further investigations, gas products of pyrolysis should be investigated for better understanding.

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