

Effect of Boron Compounds on Physical, Mechanical, and Fire Properties of Injection molded Wood Plastic Composites

Nadir Ayırlmis (✉)¹, Turgay Akbulut¹, Turker Dunder¹, Robert H. White², Fatih Mengeloglu³
Zeki Candan¹, Umit Buyuksari¹, and Erkan Avci¹

¹**Nadir Ayırlmis**, Assoc. Prof.Dr.

¹**Turgay Akbulut**, Prof.Dr.

¹**Turker Dunder**, Assoc. Prof.Dr.

¹**Zeki Candan**, Research Assistant, Ph.D. Candidate

¹**Umit Buyuksari**, Research Assistant, Ph.D. Candidate

¹**Erkan Avci**, Research Assistant, Ph.D. Candidate

Department of Wood Mechanics and Technology, Forestry Faculty, Istanbul University

Bahcekoy, Sariyer, 34473 Istanbul, TURKEY

Tel.: +90 212 226-1100

Fax: +90 212 226-1113

(✉) Corresponding Author

E-mail: nadiray@istanbul.edu.tr

²**Robert H. White**, Ph.D.

Durability and Wood Protection Research Work Unit, Forest Service, Forest Products

Laboratory, United States Department of Agriculture, One Gifford Pinchot Drive, Madison,

WI 53726-2398 USA

Tel.: +1 608-231-9265

Fax.: +1 608-231-9303

³**Fatih Mengeloglu**, Assoc.Prof.Dr.

Department of Wood Mechanics and Technology, Forestry Faculty, Kahramanmaras Sutcu

Imam University, Kahramanmaras, Turkey

Tel.: +90 212 226-1100

Fax: +90 212 226-1113

Effect of Boron Compounds on Physical, Mechanical, and Fire Properties of Injection molded Wood Plastic Composites

Abstract

Physical, mechanical, and fire properties of the injection-molded wood flour/polypropylene composites (WPCs) incorporated with different levels of boron compounds, borax/boric acid (BX/BA) (0.5:0.5 wt %) and zinc borate (ZB) (4, 8, or 12 wt %) were investigated. The effect of the coupling agent loading (2, 4, or 6 wt %), maleic anhydride-grafted PP (MAPP), on the properties of the WPCs was also investigated. Incorporation of the fire retardants (FRs) into the WPCs decreased the physical and mechanical properties as compared to the WPCs without FR. The FR treatments had little impact on the fire performance of the WPCs in the cone calorimeter tests. The WPCs containing the ZB showed better water resistance and strength values in the bending, tensile, and izod impact than the WPCs containing the BX/BA. The SEM-EDS micrographs revealed that the outer surface of the wood fibers was surrounded by some crystalline deposits of the FRs. This resulted in the poor compatibility between the wood and polymer matrix in the WPCs containing the FRs. With the addition of the MAPP up to 4 wt %, the water resistance and mechanical properties of the WPCs containing FRs up to 8 wt % improved. But the further increasing of the MAPP (6 wt %) did not improve the properties of the WPCs containing FR (12 wt %).

Keywords: Boron compounds; Fire retardant; Injection molding; Polypropylene; Polyethylene, Strength; Water resistance; Wood plastic composite

1. Introduction

Wood plastic composites are a new group of polymeric composites that have recently been investigated. Using natural fibers such as wood flour, pulp fibers, and cellulose fibers as a reinforcement of these composites and their addition to thermoplastics such as polyethylene (high and low density), polypropylene, and PVC is mainly due to their advantages such as lower production cost and density, ease of preparation, lower energy requirements for processing, biodegradability, and wide ability over traditional reinforcing fibers like glass and carbon (Chaharmahali et al. 2010). Currently several commercial WPCs are manufactured for the residential construction industry, primarily as lumber for decking and railing systems. WPCs are resistant to moisture, insects, decay, and warping when compared with traditional pressure-treated lumber. WPCs are stiffer, exhibit less creep, and are more dimensionally stable than unfilled plastic lumber. In addition, WPCs offer a “wood” look and feel with minimum maintenance. Manufacturers are also introducing new applications for the furniture industry. Further expansion into the residential construction industry and development of applications for the furniture industry require an understanding of the fire performance of WPCs (Stark et al. 2010).

As organic materials, i.e. both polymers and wood, are sensitive to fire, improvement of fire retardancy of the composite materials has become important in order to comply with the safety requirements of the WPC products. The burning process is comprised of five fundamental steps, which are, heating, decomposition, ignition, combustion and propagation (Sain et al. 2004). Polymers employed in WPCs, burn and drip in case of fire leading to a very risky scenario. Thus, FR agents must be employed in order to improve fire behaviour. The fire performance of WPCs is not well understood, and there is little information regarding the effectiveness of various FRs in the public domain (Sain et al. 2004).

The current trend is to use halogen-free FRs additives for WPCs due to ecological reasons. Intumescent FRs were developed to replace traditional halogen containing FRs answering the demands for low smoke, corrodibility, and concerns about toxicity for FR polymeric materials. Boron compounds are intumescent and halogen free FRs (Kurt and Mengeloglu 2011). Boron based flame retardants are generally char producers. The presence of boron can redirect decomposition to increase the production of carbon rather than carbon monoxide or carbon dioxide. By creating a surface layer of char, boron helps block oxygen from the surface and slows the escape of gases (Stark et al. 2010). Within the class of boron compounds, by far the most widely used is boric acid. Boric acid and sodium borate (borax) are the two FRs with the longest history, and are used primarily with cellulosic material. Zinc borate can be used as a fire retardant in PVC, polyolefins, elastomers, polyamides, epoxy resins. Zinc borate can function as a FR, smoke suppressant and anti-arcing agent in condensed phase.

Even though a lot of work has been reported on the fire properties of thermoplastics, effect of FRs on the technological and fire properties of the WF reinforced thermoplastic composites has not been extensively investigated. Recently, Ayilimis et al. (2011a and 2011b) investigated physical, mechanical, and fire properties of the flat-pressed WPC made from dry-blended wood flour (WF), various fire retardant (FR) powders, and polypropylene (PP) with maleic anhydride-grafted PP (2 wt %) formulations. The objective of our study was to determine the effects of the boron compounds on the physical, mechanical, and fire properties of the injection molded WPCs with and without coupling agent. The effect of the increased content of the coupling agent on the above mentioned properties of the WPCs containing boron compounds was also investigated. Table 1 shows the formulations used for the experimental WPCs.

2. Materials and methods

2.1. Materials

Wood particles were obtained from beech (*Fagus orientalis* Lipsky) lumbers by using laboratory type disc chipper with three knives. The moisture content of the wood particles, as determined by oven-dry weight, was found to be 40-50% prior to the treatment. The wood particles were processed by a rotary grinder (*Wiley type*) without adding additional water. The wood flour passing through a U.S. 35-mesh screen was retained by a U.S. 80-mesh. The PP ($T_m = 160^\circ\text{C}$, $\rho = 0.9 \text{ g/cm}^3$, MFI/ $230^\circ\text{C}/2.16 \text{ kg} = 6.5 \text{ g/10 min}$) produced by Petkim Petrochemical Co., Turkey, was used as the polymeric material. Maleic anhydride-grafted PP (MAPP-OPTIM-425; the reactive modifier maleic anhydride (MAH) content = 1 wt %) was supplied by Pluss Polymers Pvt. Ltd. in India.

Two FR systems (powder) were investigated:

1. A mixture of boric acid (BA) (H_3BO_3) ($\rho = 1.7 \text{ g/cm}^3$) and borax (BX) ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) ($\rho = 1.4 \text{ g/cm}^3$) (BA/BX, 1:1 by weight)
2. Zinc borate (ZB) ($3\text{ZnO} \cdot 2\text{B}_2\text{O}_3$) ($\rho = 2.8 \text{ g/cm}^3$)

Table 1 ~ Compositions of the injection molded WPC formulations.

WPC formulation	WPC composition			
	Fire retardant (FR) content	MAPP content	Wood flour (WF)	Polypropylene (PP)
	(wt %)	(wt %)	(wt %)	(wt %)
WF-40 (control)	-	-	40	60
BX/BA	4	-	38	58
WF-ZB	4	-	38	58
BX/BA	8	-	36	56
WF-ZB	8	-	36	56
BX/BA	12	-	34	54
WF-ZB	12	-	34	54
BX/BA	4	2	37	57
WF-ZB	4	2	37	57
BX/BA	8	4	34	54
WF-ZB	8	4	34	54
BX/BA	12	6	31	51
WF-ZB	12	6	31	51

MAPP: maleic anhydride-grafted PP. WF: wood flour. BX/BA (1:1): borax/boric acid, ZB: zinc borate.

2.2. Preparation of injection molded specimens

The WF was dried in a laboratory oven at 102°C for 24-h to a moisture content of 0-1% based on the oven-dry WF weight and then stored in a polyethylene bags. The WF, PP and MAPP granulates, and FR powder were processed in a 30-mm conical co-rotating twin-screw extruder (*Aysa Instrument Com*, Istanbul, Turkey) with a length-to-diameter (L/D) ratio of 30:1. The raw materials were fed into the main feed throat using a gravimetric feed system. The barrel temperatures of the extruder were controlled at 170, 180, 185, and 190°C for zones 1, 2, 3, and 4, respectively. The temperature of the extruder die was held at 200°C. The extruded strand passed through a water bath and was subsequently pelletized. These pellets were stored in a sealed container and then dried to the moisture content of 1-2% before the injection molding. The temperature used for injection molded specimens was 180-200°C from feed zone to die zone. The WPC specimens were injected at injection pressure between 45 and 50 kg/m² with cooling time about 30 s. Finally, the specimens were conditioned at a temperature of 23 °C and relative humidity (RH) of 50% according to ASTM D 618. The formulations of the composites are given in Table 2. Air-dry density values of the specimens varied from 1010 to 1070 kg/m³. Table 1 shows the raw material formulations used for the WPCs.

2.3. Determination of physical properties

The thickness swelling (TS) and water absorption (WA) tests after 2-h of boiling in water were carried out according to ASTM D 570 specifications. The test specimens were in the form of a disk 50.8 mm in diameter and 3.2 mm in thickness. The conditioned specimens were entirely immersed for 2-h in a container of boiling distilled water. At the end of the immersion time, the specimens were removed from boiling water and cooled in distilled water maintained at room temperature. After 15 min, the specimens were removed from the water, one at a time, all surface water removed with a dry cloth, and the specimens weighed to the

nearest 0.01 g and measured to the nearest 0.001 mm immediately. Ten replicate specimens were tested for each WPC formulation.

2.4. Determination of mechanical properties

The flexural properties of the specimens with dimensions of 127 mm x 12.7 mm x 3.2 (thickness) mm, modulus of rupture (MOR) and modulus of elasticity (MOE), were measured in three-point bend tests using a standard material testing system (Zwick Roel Z010) at a crosshead speed of 2.8 mm/min in accordance with ASTM D 790. Tensile strength of the specimens (dogbone shape (type III)) were tested with a crosshead speed of 5 mm/min in accordance with ASTM D 638 using a Zwick Roel Z010. Five replicate specimens were tested for tensile and flexural properties of each WPC formulation. The izod pendulum impact resistance (notched) were performed according to ASTM D 256 using a Zwick Roel HIT5.5P impact testing machine. The notches were made using a Polytest notching cutter by Ray-Ran test equipment. Ten replicate specimens were tested for the izod pendulum impact resistance of each WPC formulation

2.5. Fire performance

Heat release measurements were conducted in accordance with ASTM E 1354. Specimens were 100 mm x 100 mm and specimen thickness was 10 mm. Specimens were conditioned at 23°C and 50% RH prior to the testing. The cone calorimeter tests were conducted in the horizontal orientation with the conical radiant electric heater set at a heat flux level of 50 kW/m². The specimens were tested in the optional retainer frame but without the wire grid over the test specimen. Ignitability was determined by using a 4 seconds criteria for sustained ignition for observing the time for sustained ignition of the specimen. Two replicate specimens were tested for each type of specimen.

3. Results and discussion

3.1. Water resistance

The TS and WA values of the WPC specimens are presented in Table 2. Compared to the control specimens, the water resistance of the specimens without the MAPP decreased with increasing FR content. The TS and WA values of the control specimens were lower than those of the specimens containing the FRs due to higher hygroscopicity resulting from use of the FRs (Kurt and Mengelöglu 2011). Wood treated with inorganic FRs is usually more hygroscopic than untreated wood. In particular, boron compounds and phosphates may have diverse effects on the hygroscopicity of wood (LeVan and Winandy 1990). An increase in the TS and WA values of the WPCs containing the ZB and BX/BA can be attributed to the new adsorption sites formed by these chemicals. The specimens containing the ZB showed higher water resistance than the specimens containing the BX/BA. When the BX/BA and ZB contents increased from 4 to 12 wt %, the average TS values of the un-coupled specimens increased by 13-39% and 7-32% compared to the control specimens, respectively. Similar results were also found for the WA values. The TS and WA values of the ZB and BX/BA treatments without MAPP at the higher contents were always higher than the lower contents.

The water resistance of the WPCs containing the FR was improved by increasing content of the MAPP which was the MAPP. At the same content (wt %) of the FR, the TS and WA values of the coupled specimens was lower than the un-coupled specimens, even lower than the control specimens. The addition of MAPP up to 4 wt % had more effect on the water resistance and mechanical properties of the WPCs compared with the FRs up to 8 wt %. With the addition of the MAPP up to 4 wt %, the water resistance and mechanical properties of the WPCs containing FRs (up to 8 wt %) improved. This result was expected because the strong interfacial bonding between the WF and the PP caused by the MAPP decreased the TS and WA of the un-coupled WPCs containing the FRs (Figs. 1 and 2).

Table 2. ~ Physical and mechanical properties the WPCs at different loading levels of the fire retardant and coupling agent.

WPC type ^a	Fire retardant content	Coupling agent (MAPP) ^b content	Physical properties ^c			Mechanical properties ^c			
			Density	Thickness swelling	Water absorption	Modulus of rupture	Modulus of elasticity	Tensile strength	Impact resistance (notched)
	wt %	wt %	kg/m ³	%	%	MPa	MPa	MPa	J/m
WF-40 (control)	-	-	1030 (20)	1.31 (0.06)	1.60 (0.10)	68.8 (6.9)	8609.5 (150.2)	26.3 (0.6)	25.7 (1.7)
WF-BX/BA	4	-	1050 (15)	1.48 (0.07)	1.83 (0.13)	64.2 (8.0)	8845.3 (261.3)	24.8 (0.7)	23.9 (2.7)
WF-ZB			1010 (20)	1.40 (0.07)	1.74 (0.15)	65.6 (7.7)	8914.8 (344.1)	25.4 (2.2)	24.6 (2.5)
WF-BX/BA	8	-	1060 (20)	1.67 (0.10)	1.98 (0.12)	62.5 (6.9)	9003.8 (336.1)	23.2 (0.1)	22.5 (1.9)
WF-ZB			1040 (30)	1.51 (0.09)	1.86 (0.17)	64.2 (9.4)	9244.6 (157.5)	24.7 (0.7)	22.8 (1.3)
WF-BX/BA	12	-	1050 (20)	1.82 (0.11)	2.20 (0.24)	58.5 (5.72)	9314.3 (205.2)	21.6 (0.2)	20.4 (5.2)
WF-ZB			1010 (10)	1.73 (0.13)	2.05 (0.20)	61.0 (7.3)	9488.4 (110.7)	23.5 (1.7)	21.6 (1.7)
WF-BX/BA	4	2	1060 (10)	1.24 (0.05)	1.48 (0.14)	70.3 (5.6)	9085.3 (124.4)	27.0 (0.5)	26.2 (1.3)
WF-ZB			1040 (30)	1.14 (0.06)	1.30 (0.17)	69.5 (7.3)	9287.5 (320.5)	28.8 (3.2)	26.9 (1.0)
WF-BX/BA	8	4	1020 (25)	1.04 (0.10)	1.24 (0.10)	71.8 (6.5)	9375.8 (280.5)	28.8 (0.7)	27.8 (1.4)
WF-ZB			1050 (10)	0.93 (0.06)	1.13 (0.09)	73.6 (7.1)	9784.2 (158.7)	30.6 (0.7)	29.3 (2.1)
WF-BX/BA	12	6	1070 (30)	1.17 (0.09)	1.35 (0.11)	69.7 (6.1)	9556.7 (267.3)	28.1 (0.2)	27.2 (1.8)
WF-ZB			1050 (20)	1.06 (0.05)	1.22 (0.08)	72.2 (5.6)	9843.7 (313.6)	29.5 (1.6)	28.8 (1.9)

^a WF: wood flour, BX/BA (1:1): borax/boric acid, ZB: zinc borate.^b MAPP: maleic anhydride-grafted PP.^c Values are means and standard deviations in parentheses.

The coupling agents improve the quality of adhesion between plastics and wood particles to reduce the gaps in the interfacial region and to block the hydrophilic groups (Ayrilmis and Jarusombuti 2011). The anhydride groups in the MAPP enter into an esterification reaction with the surface hydroxyl groups of wood fibers and covalently bond to the hydroxyl groups. With the decreasing hydroxyl groups on the WF surface, hydrogen-bonding sites for water molecules decreased on the WPC surface and this resulted in a lower TS and WA value. However, the water resistance and mechanical properties of the WPCs decreased when the MAPP and FR contents increased from 4 to 6 wt % and 8 to 12 wt %, respectively. This was attributed to the MAPP at 6 wt % could not suppress the negative effect of the FRs at 12 wt % due to the higher contamination of the wood surface by crystalline deposits of FRs (Table 2).

Different volume percentages of the ZB and BX/BA used in the WPC specimens were also responsible for the differences in the TS and WA values of ZB and BX/BA treatments. This was mainly attributed to the different densities of the FRs used in the experiments. For this reason, the volume percentages of the FRs in the WPCs differed from each other. The volume percentage of the specimens containing the BX/BA in the WPC was higher than that of the specimens containing the ZB due to the fact that the ZB had higher density than the BX and BA. It was estimated that the interfacial bonding between the functional polar groups of the WF and MAPP was decreased by increasing volume percentage of the FR powder. The contamination of the wood surface resulted in poor compatibility between the WF and polymer matrix since some of the material stays as a powder on the outer surface of the WF (Ayrilmis et al. 2011b). The poor compatibility between the WF and polymer matrix allows for easier moisture intrusion as compared to the WPCs without FRs. As a result of the poor compatibility, the water resistance of the WPC specimens containing the FRs decreased (Fig. 1). However, the TS and WA values of the FR-treated specimens containing the MAPP was

lower than those of the FR-treated specimens without MAPP at all levels of the FR content (Fig. 2).

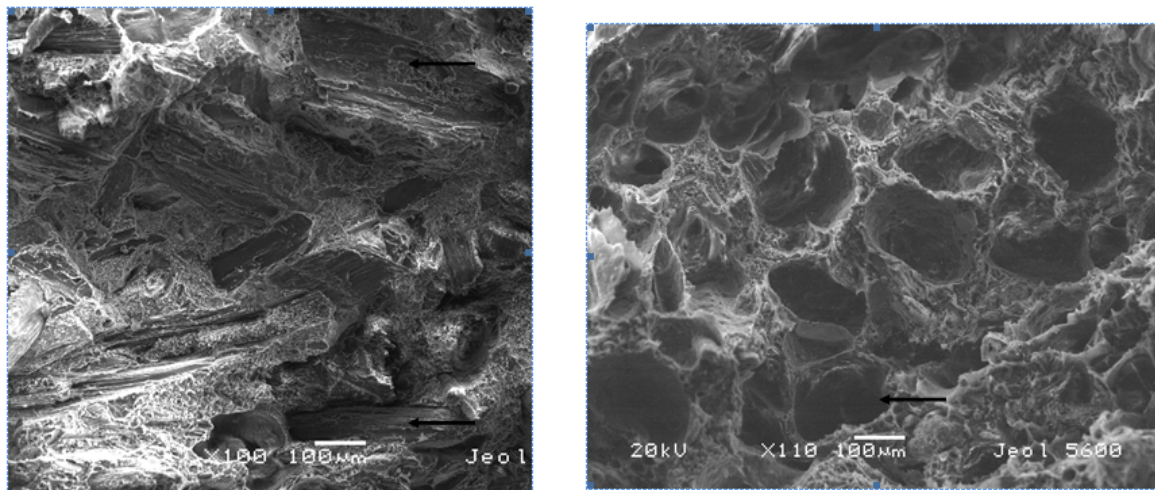


Figure 1. ~ *Poor compatibility between the wood fiber and polymer matrix of the un-coupled WPC specimen containing 8 wt % fire retardant.*

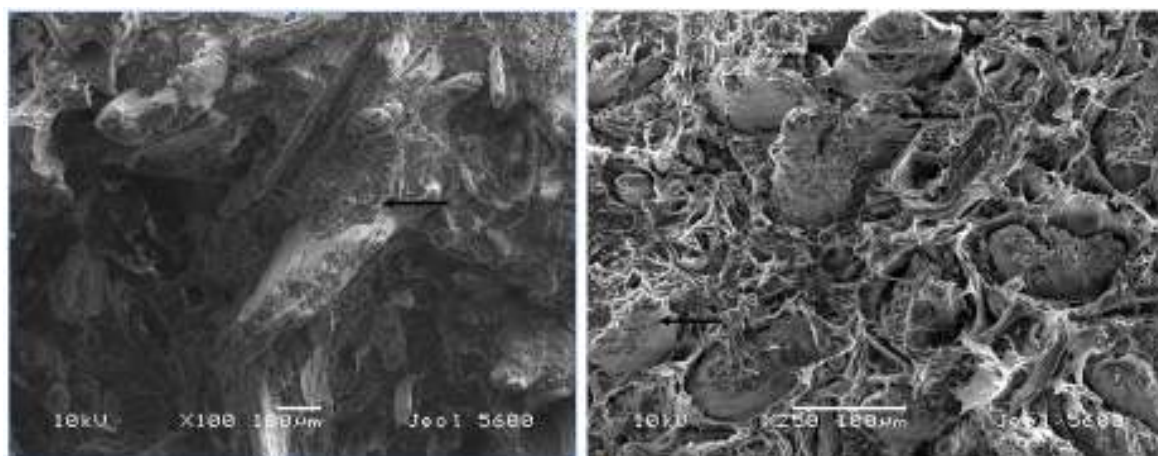


Figure 2. ~ *Strong compatibility between the wood fiber and polymer matrix of the 4% wt MAPP coupled WPC specimen containing 8 wt % fire retardant.*

Decreasing polymer content as a function of increasing FR content in the WPC was also responsible for the lower water resistance of the specimens. This was mainly attributed to the hydrophobic character of the PP because of its being devoid of functional polar groups such as hydroxyls in the molecular and thus chemically inactive. For example, the PP content of

the control specimens was 60 wt % while it was 54 wt % for the un-coupled specimens containing 12 wt % FR (Table 1). The PP can crystallize on the WF and thereby wrapping WF better and leaving less exposed the wood on the WPC surface. The TS and WA of the specimens increased with increasing FR content in the WPC due to the fact that a larger share of the particle surface (WF and FR powder) was insufficiently bonded and protected by the plastic component.

3.2. Mechanical properties

The mechanical properties of all WPC formulations are presented in Table 2. The un-coupled control specimens had the highest MOR, followed by the un-coupled specimens containing the ZB and BX/BA, respectively. The MOR of the un-coupled specimens containing the BX/BA and ZB as a function of increasing FR content decreased by 7-15% and 5-11% compared to the control specimens while the MOE increased by 3-8% and 4-10% increased, respectively. This finding was consistent with previous studies (Ayrilmis et al. 2011a and 2011b, Kurt and Mengeloglu 2011). It should be noted that the control group contained more polymer compared to other groups. Although the ratio of the polymer matrix to the WF was fixed as 1.5-1.6 in the formulations, the groups with FRs reduced the actual amount of the polymer in the formulation. It was estimated that decreasing polymer content caused an increase in the MOE. The flexural properties of the coupled WPCs containing the FRs were better than un-coupled WPCs containing the FRs. Furthermore, the MOR and MOE of the specimens having an increased MAPP content increased with increasing FR content, even higher than the un-coupled control specimens. This finding revealed that the MAPP had more effect on the flexural properties of the WPCs than the FRs. The negative effect of the boron compounds on the MOR, tensile strength, and impact resistance of the WPCs was suppressed by increasing coupling.

The tensile strength of the specimens without MAPP were decreased by increasing FR content compared to the control WPCs. The tensile strength of the un-coupled specimens containing BX/BA and ZB decreased by 6-18% and 3-11%, respectively, compared to the control specimens. Decreases in the tensile strength of the filled plastics with the addition of FRs has reported by some workers (Sain et al. 2004, Horn et al. 2000, Chiu and Wang 1998). Evaluation of the un-coupled WPC specimens with the FR using SEM-EDS micrographs revealed that outer surface of the wood fibers was surrounded by some crystalline deposits of the FRs, thereby increasing surface area of the solids within the WPC and reducing the bonding efficiency of the polymer (Fig. 3). This resulted in the contamination of the wood surface by the presence of loosely adhering crystalline deposits of FRs and was a cause of poor compatibility between the WF and polymer matrix in the WPC. Such an effect was clearly observed in the tensile test of the specimens. The SEM micrographs visually describe the above findings. The WF were found un-bound in the Fig. 3 when no coupling agent was used. However, the interaction between the PP and WF contaminated by the FRs improved with the incorporation of the MAPP. The different volumetric percentages of the ZB and BX/BA in the WPC also affect the tensile strength of the WPCs. Increment in the volumetric content of the FR negatively affect interface bonding between the wood flour and the polymer matrix due to the fact that the contamination of the wood surface is increased by increment in the volumetric content of the FR (Ayrilmis 2011b.)

When the cross sections of the tensile test specimens after failure were observed in the SEM, Fig. 2 showed the strong bonding between the polar wood and non-polar PP matrix using the coupling agent (the MAPP). By closely examining these micrographs, it was observed that WF contaminated by the FRs was strongly bonded to the PP and had to be broken in order to fracturing the WPC bar (Fig. 2).

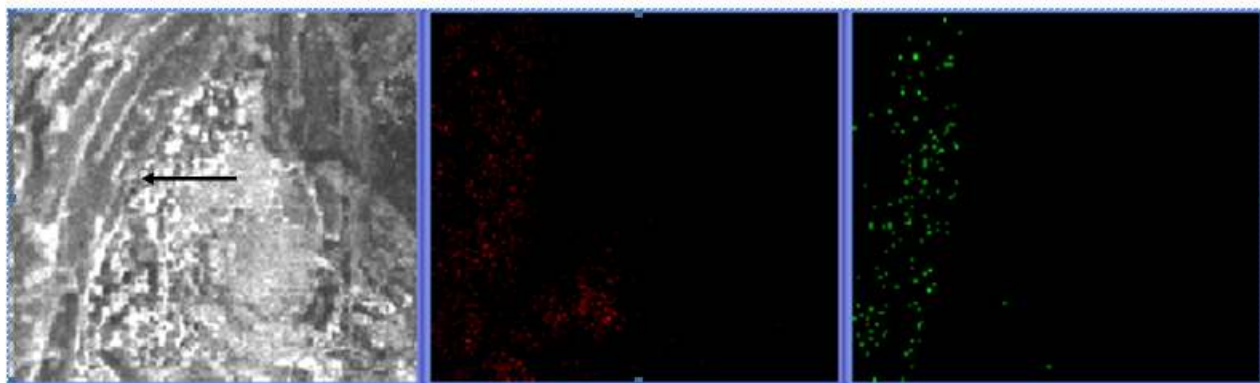


Figure 3. ~ Typical elementary chemical analysis using a scanning electron microscope (SEM) equipped with an Energy Dispersive Spectrometer (EDS) on the cross section of the tensile strength un-coupled WPC specimen containing zinc borate (ZB) after the failure. The SEM/EDS micrographs show that the outer surface of the wood fibers was surrounded by some crystalline deposits of the boron compounds. This results in the poor compatibility (the arrow's way on the SEM micrograph) between the wood fiber and polymer matrix. The EDS micrographs: red color shows the element Boron (B) element and green color shows element Zinc (Zn).

Decreasing polymer content of the WPCs as a function of the increasing FR content could be responsible for the lower tensile strength since the FR content increases and thus the amount of the plastic, as the adhesive, decreases. The mechanical properties of the specimens containing the ZB was higher than those of the specimens BX/BA due to the higher volume percentage of the ZB as compared to the BX/BA.

The specimens containing the FR showed lower notched impact strength as compared to the control specimens (Table 2). For example, when the BX/BA and ZB contents increased from 4 to 12 wt %, the impact resistance of the un-coupled specimens decreased by 7-21% and 4-16%, respectively, compared to the control specimens. This was mainly attributed to the poor compatibility between the WF and polymer matrix due to the contamination of the wood surface by the presence of loosely adhering crystalline deposits of FRs (Fig. 3). The specimens containing the ZB had higher impact resistance than the specimens containing the BX/BA. The impact resistance of the specimens having an increased MAPP decreased with increasing FR content.

3.3. Fire properties

The cone calorimeter results for the WPC formulations are presented in Table 3. In the cone calorimeter tests; the heat release rate, mass loss rate, and specific extinction area were measured as a function of time. The observation of time for sustained ignition (TSI, s) was also recorded. From the curves of heat release rate, the recorded observations included the initial peak heat release rate (PHRR, kW/m²) and the heat release rates averaged over 300 s (AHRR-300, kW/m²) after sustained ignition. The average effective heat of combustion (AEHOC, MJ/kg) was calculated from the total heat released and the total mass loss. The mass loss rate data was averaged for the duration of 10 to 90% of the ultimate mass loss (AMLR10-90). Obscuration of a laser beam in the exhaust duct was recorded as a measure of the visible smoke development from the burning specimen. Average specific extinction area (ASEA, m²/kg) was computed from smoke obscuration data for duration of the test. The results (Table 3) indicated that the treatments provided little, if any, improvements in the fire performance.

Linear regression analysis was conducted on the test results as a function of treatment level. The overall results were consistent with the conclusion that the treatments were ineffective. Some linear regression analysis of the combined BX/BA and BX/BA/MAPP data indicated significance levels less than 0.10 for the regression model. The linear regression analysis of the combined BX/BA and BX/BA/MAPP data suggested the BX/BA treatment might have reduced the AMLR (R^2 of 21%) but increased the AEHOC (R^2 of 28%). In the case of the ZB treatments, the similar analysis suggested the treatment might have decreased AMLR (R^2 of 38%), TSI (R^2 of 25%), PHRR (R^2 of 28%), and AHRR-300 (R^2 of 28%). Increased AEHOC and decreased TSI are not desirable effects on fire performance. In the case of PHRR, the suggested reduction from the linear regression of the ZB data was only 40

Table 3. Cone calorimeter results for the WPCs at different loading levels of the fire retardant and coupling agent.

WPC type ^a	Fire retardant content	Coupling agent (MAPP) ^b content	Cone calorimeter results ^c						
			Time to Sustained Ignition	Peak Heat Release Rate	Avg. Heat Release Rate 300s	Total Heat Released	Avg. Mass Loss Rate (10%-90%)	Avg. Effective Heat of Combustion	Avg. Specific Extinction Area
	wt %	wt %	s	kW/m ²	kW/m ²	MJ/m ²	g/m ² s	MJ/kg	m ² /kg
WF-40 (control)	-	-	23.6	510	373	312	10.8	31.0	420
WF-BX/BA	4	-	19.8	513	371	245	12.7	31.9	472
WF-ZB			20.2	530	403	335	11.1	32.7	514
WF-BX/BA	8	-	20.7	510	384	323	10.1	33.4	508
WF-ZB			21.2	479	342	311	9.2	31.7	480
WF-BX/BA	12	-	20.9	504	364	295	9.6	33.0	436
WF-ZB			20.2	474	334	315	9.0	33.2	477
WF-BX/BA	4	2	20.1	465	382	318	10.9	33.0	474
WF-ZB			20.4	518	393	317	9.9	33.9	493
WF-BX/BA	8	4	20.3	507	377	310	9.9	34.1	509
WF-ZB			20.9	499	357	314	9.6	33.4	468
WF-BX/BA	12	6	22.3	495	384	306	10.0	34.0	545
WF-ZB			20.1	495	367	310	9.8	33.2	488

^a WF: wood flour, BX/BA (1:1): borax/boric acid, ZB: zinc borate.

^b MAPP: maleic anhydride-grafted PP.

^c Values are means of two replicates.

kW/m^2 for a change from no treatment to 12 wt % treatment level which is not much given a PHRR of 510 kW/m^2 for the untreated control specimen.

The WF and plastic content were evenly adjusted to accommodate the 4%, 8%, and 12% addition of the FR chemical. Each FR treatment level included specimens with and without a coupling agent (MAPP). For specimens with the coupling agent, the loading level of the coupling agent was 50 wt % of the loading level of the FR chemical. The addition of the coupling agent had little apparent effect on the fire test results. The improvements for 12% ZB over the untreated 40% wt. WF, 60% wt PP. were not as great as in the earlier studies in which the WF was 50% wt. (Stark et al. 2010, Ayrilmis 2011a). In the earlier studies, the addition of FR chemical reduced the polymer content only, whereas in the current study, the addition of FR chemical was accommodated by equal reductions in wood and polymer contents. Thus, the improved results for the ZB in the two earlier studies reflected the added reductions in the polymer content. Further investigation is needed to determine whether any processing factors affected the effectiveness of the chemical treatments.

4. Conclusions

The following general conclusions can be drawn from the study provided in the paper:

1. Compared to the control WPC, incorporation of the boron compounds, the ZB and BX/BAFR, decreased the water resistance and mechanical properties of the WPCs without the MAPP, except for the MOE. The MOE of all WPCs with and without MAPP increased with increasing FR content. The WPCs containing ZB had higher water resistance and mechanical properties than the WPCs containing BX/BA.
2. Use of the coupling agent up to 4 wt % had more effect on the water resistance and mechanical properties of the WPCs than the use of the FRs up to 8 wt % because the TS and

WA values of the specimens having an increased content of the MAPP decreased with increasing the FR content. A similar trend was also observed for the mechanical properties. However, the water resistance and mechanical properties of the coupled WPCs decreased when the MAPP and FR contents increased from 4/6 and 8/12 wt %, respectively, except for the MOE. This was attributed to the MAPP could not suppress the negative effect of the FRs at 12 wt % due to the higher contamination of the wood surface by crystalline deposits of FRs.

3. The SEM-EDS micrograph revealed that outer surface of the wood fibers was surrounded by some crystalline deposits of the FRs. This resulted in the poor compatibility between the wood flour and polymer matrix in the WPCs containing the FRs.

4. The results from the cone calorimeter tests indicated that the ZB and BX/BA treatments provided little, if any, improvements in the fire performance. It is noted that in this study, the increased FR treatments was accommodated by reducing the percentage WF as well as the percentage PP.

Acknowledgement

This work has been supported by the Research Fund of Istanbul University, Istanbul, Turkey. Project No: 2396. Its support is gratefully acknowledged. The authors would like to thank United States Department of Agriculture, Forest Service, Forest Products Laboratory for the cone calorimetry tests.

ASTM Standards

ASTM International (2010) Standard test method for water absorption of plastics, ASTM D570 - 98(2010)e1, West Conshohocken, PA.

ASTM International (2008) Standard practice for conditioning plastics for testing, ASTM D618-08.

ASTM International (2010) Standard test methods for flexural properties of unreinforced and reinforced plastics and electrical insulating materials. ASTM D790-10.

ASTM International (2010) Standard test method for tensile properties of plastics. ASTM D638-10.

ASTM International (2010) Standard test methods for determining the izod pendulum impact resistance of plastics. ASTM D256-10.

ASTM International (2008) Standard test method for heat and visible smoke release rates for materials and products using an oxygen consumption calorimeter. ASTM E 1354-08a.

Literature Cited

Ayrilmis, N. and S. Jarusombuti. 2011. Flat-pressed wood plastic composite as an alternative to conventional wood-based panels. J. Compos. Mater. 45:103–112.

Ayrilmis, N., J.T. Benthien, H. Thoemen, and R.H. White. 2011a. Effects of fire retardants on physical, mechanical and fire properties of flat-pressed WPCs. Eur. J. Wood Prod., in press (*Doi:10.1007/s00107-011-0541-3*).

Ayrilmis, N., J.T. Benthien, H. Thoemen, and R.H. White. 2011b. Properties of flat-pressed wood Plastic composites containing fire retardants. J. Appl. Polym. Sci., in press.

Chaharmahali, M., J. Mirbagheri, M. Tajvidi, S.K. Najafi, and Y. Mirbagheri, Y. 2010. Mechanical and physical properties of wood-plastic composite panels. J. Reinf. Plast. Compos. 29:310-319.

Chiu, S.H. and W.K. Wang. 1998. The dynamic flammability and toxicity of magnesium hydroxide filled intumescent fire retardant polypropylene. J. Appl. Polym. Sci. 67:989-995

Kurt, R. and F. Mengeloglu. 2011. Utilization of boron compounds as synergists with ammonium polyphosphate for flame retardant wood-polymer composites. Turk. J. Agric. For., in press (*Doi:10.3906/tar-0910-508*).

Levan, S.L. and J.E. Winandy. 1990. Effects of fire-retardant treatments on wood strength: a review, Wood and Fiber Science 22(1):113-131.

Sain, M., S.H. Park, F. Suhara, and S. Law. 2004. Flame retardant and mechanical properties of natural fibre-PP composites containing magnesium hydroxide. Polym. Degrad. Stabil. 83:363-367.

Stark, N.M., S.A. Mueller, R.H. White, and T.A. Osswald. 2010. Evaluation of various fire retardants for use in wood flour–polyethylene composites. *Polym Degrad. Stabil.* 95:1903-1910.

Horn, W.E., Jr. 2000. Inorganic Hydroxides and Hydroxycarbonates: Their Function and Use as Flame-Retardant Additives, in *Fire Retardancy of Polymeric Materials*, Ed. A. F. Grand and C. A. Wilkie, Marcel Dekker, New York, pp. 285-352.

In: Proceedings of the 11th international conference on wood and biofiber plastic composites & nanotechnology in wood composites symposium. 2011 May 16-17; Madison, WI Madison, WI: Forest Products Society: 21 pages