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## Utilization of boron compounds as synergists with ammonium polyphosphate for flame retardant wood-polymer composites

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**Abstract:** The investigation consisted of utilizing boron compounds (BCs) as a synergist with ammonium polyphosphate (APP) in manufacturing flame retardant wood-polymer composites (WPCs). Flame retardants (FRs) at various ratios (APP, boric acid (BA), borax (BX), zinc borate and BA/BX mixtures) were used to determine the physical, mechanical, and burning rate properties of WPCs manufactured from wood flour, polypropylene, and maleic anhydride polypropylene. The results showed that the addition of FRs reduced all mechanical properties of WPCs except for impact strength. However, this reduction was lower for samples having BCs in FRs formulation. Similar burning rate results were achieved in WPCs having FRs. The combination of BA/BX with APP is thought to have overcome the negative effect of APP on strength properties through the formation of boron phosphate between APP and BA. BCs, used as the synergist, improved the mechanical and FRs performance of WPCs. This study showed that using BCs as synergists with APP has potential as a relatively inexpensive halogen-free FR alternative for the industry.

**Key words:** Ammonium polyphosphate, boron, boron phosphate, flame retardants, polypropylene, wood-polymer composite

### Bor bileşiklerinin sinerjist olarak amonyum polifosfat ile alev geciktirici odun-polymer kompozitleri için değerlendirilmesi

**Özet:** Bor bileşiklerinin (BB) sinerjist olarak amonyum polifosfat ile alev geciktirici odun-polymer kompozitleri (OPK) üretiminde değerlendirilmesi incelenmiştir. Odun unu, polipropilen, maleik anhidritli polipropilen ve farklı oranlardaki alev geciktiricilerden (AG) (APP, borik asit (BA), boraks (BS), çinko borat (ÇB) ve BA/BS karışımları) üretilen OPK'ların; fiziksel, mekanik ve yanma hızı özellikleri test edilmiştir. Sonuçlar AG ilavesinin, şok direnci dışında bütün mekanik özellikleri düşürdüğü, fakat AG formülasyonunda BB içeren örneklerdeki düşüşün daha az olduğunu göstermiştir. AG'li OPK benzer yanma özellikleri sağlamıştır. BA/BS ve APP kombinasyonlarında APP'nin direnç üzerindeki negatif etkisinin üstesinden gelmesinin, BA-APP arasındaki boronfosfat oluşumuyla sağlandığına inanılmaktadır. BB'lerinin sinerjist olarak kullanımı, OPK'nın mekanik ve yanma hızı performanslarını iyileştirmiştir. Bu çalışma, BB sinerjist olarak APP ile kullanımının, endüstri için nispeten ucuz halojensiz AG olarak iyi bir potansiyele sahip olduğunu göstermiştir.

**Anahtar sözcükler:** Amonyum polifosfat, bor, boronfosfat, alev geciktirici, polipropilen, odun-polimer kompozit

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## Introduction

The term wood-polymer composites (WPCs) covers a wide range of composite materials using polymers ranging from polypropylene (PP) to polyvinyl acetate (PVA) and fillers ranging from wood flour to flax (Mali et al. 2003). WPCs can be defined as a material consisting of polymers, lignocellulosic fibers, and additives (lubricants, coupling agents, pigments, antioxidants, UV stabilizers, antimicrobial agents, etc.). WPCs are manufactured using a high volume process such as extrusion, compression, or injection molding (Klyosov 2007). The properties of WPC can be designed to meet the product requirements of various wood polymers. Additives can improve specific properties of WPCs (Tangram 2002).

Wood flour and other natural fibers are used as reinforcing fillers in WPCs due to their low cost, high strength, low density, renewability, and biodegradability (Mengeloğlu and Matuana 2001; Clemons 2002; Pendelton et al. 2002). Additional advantages include abundance in the environment, recyclability, low energy need, sustainability, and low abrasion properties (Rowell et al. 1997; Mengeloğlu et al. 2000). The serious environmental problems caused by growing consumption of synthetic polymers derived from petroleum emphasize the need to maximize the use of renewable resources (Qui et al. 2006).

WPC components—wood and polymers—are flammable. Flame spread, fuel contribution, and smoke development are of great importance in wood-based materials (Suchsland and Woodson 1986). Complying with the safety requirements of the wood-fiber composite products necessitates the improvement of flame retardancy of composite materials (Sain et al. 2004). Efforts to expand the use of wood-based products in institutional and commercial structures may require treatment with flame retardants (LeVan 1984). The WPC can be made flame resistant by loading it with flame retardant (FR) components or employing PVC (or other low flammable polymers) as a base polymer. Using PVC is not considered a viable alternative due to its hydrogen chloride (toxic and volatile strong acid) release (Klyosov 2007).

Many FRs are available for polymers in the market, including phosphate/phosphorus containing chemicals (Green 2000; Chiang and Hu 2001), monoammonium phosphate, diammonium phosphate (Green 2000; Horrocks 2001), ammonium polyphosphate, expandable graphite (Schartel et al. 2003), ammonium sulfate, zinc chloride, magnesium hydroxide, aluminum trihydrate, calcium sulfate, potassium carbonate, etc.

The current trend is to use halogen-free FRs for ecological reasons (Mali et al. 2003). 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 (Chiang and Hu 2001).

Ammonium polyphosphate (APP) and boron compounds are intumescent and halogen free FRs. They retard flame by producing carbonaceous foam protecting the underlying material from temperature increase (Hafizoğlu et al. 1994; Clariant 2008a). Boron compounds can be a viable option as synergists reducing the strength loss and improving FR properties of WPCs if used with commonly available FRs for the WPCs industry.

APP is a well known phosphorus FR, accounting for about 20% of FRs in the industry (without polyolefins) (Klyosov 2007). APP is an alternative to commonly used FRs in WPCs such as magnesium hydroxide and aluminum trihydrate. In addition, APP reportedly has an excellent FR effect in paper and wood products containing cellulose (Clariant 2008a). APP has been used in wood, steel, and polyurethane foam as a FR (Li et al. 2001; Li and He 2004; Wang et al. 2007).

Boron compounds (BCs) have been used as FRs and as wood preservation chemicals against fungi, insects, and mold for wood-based composites (Laks and Manning 1995; Yalınkılıç et al. 1998; Örs et al. 1999; Çolakoğlu et al. 2003; Baysal et al. 2003; Kartal 2006; Kurt and Mengeloğlu 2008). There is growing interest in their low mammalian toxicity and environmental acceptability (Laks et al. 1994). Boric acid (BA) suppresses glowing with little effect on flame spread. In contrast, borax (BX) tends to reduce flame spread, but promotes smoldering or glowing (Wang et al. 2003). BA and BX were used together in different ratios to provide reduced flame

spread and smoldering/promote glowing (Baysal et al. 2007; Dhamodaran and Gnahanan 2007; Kurt and Mengeloglu 2008; Özçiftçi 2008).

APP can be used alone or in synergy with FR additives. Synergists enhance the effectiveness of the FRs. Some of examples of inorganic compounds reported as synergists are antimony trioxide (for halogenated FRs) and boron-based compounds such as zinc borate (Klyosov 2007). APP was used with boric acid in the electrical cable and steel industries successfully (Jimenez et al. 2006a, 2006b; Mosnacek et al. 2008).

The lack of information available in the literature related to boron compounds usage as synergists with APP prompted the initiation of this research. Therefore, this research was designed to investigate the use of boron compounds (borax, boric acid, and zinc borate) as synergists with APP in the manufacture of WPCs. Special focus is concentrated on the effect of boron compounds' addition on selected WPCs' physical, mechanical, and FR properties. The study was aimed to contribute information on the suitability of boron compounds as synergists in FR WPC production. It will provide scientific data for boron compounds' use in WPC production for flame retardancy. The findings are thought to promote the use of boron compounds in the production of WPCs and wood-based composites in higher amounts.

## Materials and methods

### Materials

Polypropylene (PP) (Petoplen MH 418) (Petkim 2008) was used as a polymer matrix while maleic anhydride-grafted polypropylene (MAPP) (Licomont AR 504) (Clariant 2008b) was utilized as a coupling agent. As FRs, BA, BX, zinc borate (ZB), and APP (Exolit AP 422) (Clariant 2008a) were used. First 3 FRs were purchased from Aromas, Turkey, while the last one was provided by Clariant, Turkey. As a filler, nominal 40 mesh (420  $\mu\text{m}$ ) pine (*Pinus sylvestris*) wood flour was used.

### Manufacture of wood-polymer composites

Manufacturing of the composites was accomplished based on the description in Table 1. A control sample (Cont.) was made of 67 wt% PP, 30

wt% wood flour, and 3 wt% MAPP. For groups A to E, additional 25% FR was added to the formulation of the control group mixture. Added to 22% APP were 3 different BCs (BA, BX, ZB) and 2 different BA/BX mixtures (ratio of 71:29 and 53:47) to be used as FRs as well as 25% APP alone. The components of WPCs were added to high speed mixer in the following order: wood flour, FR (if any), MAPP, and PP. They were mixed for at least 10 min to produce a homogeneous blend. WPC pellets were manufactured at Kahramanmaraş Sütçü İmam University's Wood Technology Laboratory, using a single-screw extruder. The temperature setting from the hopper to die was 180 / 190 / 195 / 200 °C. The screw speed was set at 100 rpm. The pelletized composites were dried at 100 °C for 6 h before pressing on a hydraulic hot press. WPCs were pressed at 180 °C to approximately 5(t)  $\times$  150(w)  $\times$  150(l) mm boards. Fifteen boards were manufactured for each group.

### Testing

#### Physical properties

The moisture content (MC), specific gravity (SG), and dimensional stability characteristics (water absorption (WA) and swelling in thickness (ST)) of the produced WPCs were determined according to ASTM D4442 (2003), ASTM D792 (Method A) (2007) and ASTM D1037 (1998) respectively. To determine MC and SG, specimens with 5  $\times$  20  $\times$  20 mm dimensions were used. Specimens to determine WA and ST properties were 5  $\times$  30  $\times$  30 mm in size. They were conditioned at a relative humidity of 65  $\pm$  5% and a temperature of 23  $\pm$  2 °C and submerged in distilled water for 24 h. Weight and dimensions measurements were taken after tests to find out WA and ST properties. Tests were run with 5 replicates for each group.

#### Mechanical properties

Mechanical tests were conducted to determine the effect of FRs on mechanical properties. Modulus of rupture (MOR) and modulus of elasticity (MOE), tensile strength (TS), and impact strength (IS) tests were performed. All specimens were conditioned at a relative humidity of 65  $\pm$  5% and a temperature of 23  $\pm$  2 °C. Ten replicates were tested for each group. Specimens (5 (t)  $\times$  13 (w)  $\times$  150 (l) mm) for MOR and MOE were tested in 3 point loading mode with

a crosshead speed of 2 mm/min in accordance with ASTM D6109 (2005a) using a Zwick Roel Z010 Universal Testing machine. Tensile specimens (dogbone shape (type III)) were tested with a crosshead speed of 5 mm/min in accordance with ASTM D638 (2001a) on the aforementioned testing machine. Notched impact specimens (5 (t) × 12.7 (w) × 64 (l) mm) were tested in accordance with ASTM D256 (2005b) on a Zwick HIT5.5P Impact Testing machine. The notches were made using a Polytest notching cutter by RayRAn.

### Scanning electron microscope (SEM) study

WPC bars were dipped into liquid nitrogen and then snapped into 2 to get fractured surfaces for SEM analysis. The surface was plated with a thin layer of gold before the observation. The SEM observations were performed using a JEOL scanning electron microscope (Model JSM 5500LV) at 10 kV voltage with 100 and 1000 magnification.

### Burning rate tests

The flame retardancy of WPCs was determined by horizontal burning tests according to ASTM D635 (1997). Specimens (5 (t) × 13 (w) × 125 (l) mm) were conditioned in an environmentally controlled room at relative humidity of  $50 \pm 5\%$  and temperature of  $23 \pm 2\text{ }^\circ\text{C}$  for 48 h. For each variable, 10 specimens were tested.

### Statistical analysis

Analysis of variance (ANOVA) was used to determine the effect of FRs on selected mechanical

and FR properties of WPCs using SAS statistical package program (SAS 2001). The resulting F value was compared to the tabular F value at the 95% probability level. When F tests resulted in significant differences, comparisons between means were made by Bonferroni (Dunn) t-test.

## Results

### Physical properties

The physical properties of the manufactured WPCs are given in Table 2. The average SG and MC of WPCs were 0.93%-1.07% and 0.29%-0.89%, respectively. Water absorption (WA) and swelling in thickness (ST) values were evaluated after a 24 h submersion of the WPC samples under distilled water. The mean WA and ST values of WPCs were 0.41%-2.36% and 0.59%-1.76%, respectively.

### Mechanical properties

MOR, MOE, TS, and IS of manufactured WPCs were evaluated. The results are summarized in Table 3. The mean MOR values ranged from 31.30 to 38.60 MPa. Control specimens showed the highest MOR values, with group A providing the lowest value. Addition of FRs into matrix reduced the MOR values by 2.85%-18.91% compared to those of the control specimens. Statistical analysis showed the mean MOR values changed with FRs loading. There was a significant difference between MOR values at the 0.05 level of probability among the samples. Comparisons

Table 1. Experimental design of the study.

ID	Polypropylene (PP) (%)	Wood flour (%)	MAPP (%)	Flame retardants (%)			
				BA	BX	ZB	APP
Control	67.00	30.00	3.00	-	-	-	-
A	67.00	30.00	3.00	-	-	-	25.00
B	67.00	30.00	3.00	2.10	0.90	-	22.00
C	67.00	30.00	3.00	1.59	1.41	-	22.00
D	67.00	30.00	3.00	3.00	-	-	22.00
E	67.00	30.00	3.00	-	3.00	-	22.00
F	67.00	30.00	3.00	-	-	3.00	22.00

Table 2. Selected physical properties of WPCs.

ID/Properties	Specific gravity	Moisture content (%)	Swelling in thickness (%)	Water absorption (%)
Control	0.93 (0.01)*	0.89 (0.00)	0.66 (0.07)	0.41 (0.07)
A	1.02 (0.00)	0.48 (0.01)	1.76 (0.53)	2.36 (0.21)
B	1.03 (0.01)	0.42 (0.02)	0.59 (0.00)	0.83 (0.02)
C	1.04 (0.01)	0.29 (0.02)	1.22 (0.20)	1.03 (0.09)
D	1.07 (0.03)	0.31 (0.01)	0.96 (0.22)	1.04 (0.20)
E	1.04 (0.00)	0.32 (0.01)	1.13 (0.26)	1.46 (0.23)
F	1.03 (0.00)	0.30 (0.01)	1.17 (0.24)	1.07 (0.12)

\* Values between parenthesis show standard deviation

Table 3. Selected mechanical properties of the manufactured WPCs.

ID/ Properties	Modulus of rupture (MPa)	Modulus of elasticity (GPa)	Tensile strength (MPa)	Impact strength (J m <sup>-1</sup> )	Burning rate (mm min <sup>-1</sup> )
Control	38.60 a (1.61)*	1.61 b (0.13)	19.96 ab (0.43)	15.20 abc (0.56)	23.27 a (1.41)
A	31.30 c (1.99)	1.65 ab (0.15)	15.68 d (0.51)	16.24 a (1.54)	14.32 b (1.49)
B	37.50 a (1.32)	1.92 a (0.15)	20.28 a (0.37)	13.97 c (0.83)	13.58 b (1.79)
C	36.74 ab (0.92)	1.89 ab (0.16)	20.00 ab (0.32)	14.00 c (0.83)	14.50 b (0.75)
D	36.46 ab (1.28)	1.81 ab (0.20)	18.28 c (0.53)	14.74 bc (0.85)	12.49 b (1.09)
E	34.76 b (2.97)	1.70 ab (0.21)	19.46 b (0.78)	15.27 ab (0.70)	14.87 b (0.55)
F	36.01 ab (2.37)	1.72 ab (0.31)	18.30 c (0.61)	15.27 ab (0.83)	14.46 b (1.49)

\* Values between parenthesis show standard deviation

Different letters in a column are statistically significant at  $P < 0.05$ .

n = 10

of mean MOR values were made by Bonferroni (Dunn) t-test. According to the results, there was no significant difference between MORs' of control and groups B, C, D, and F. In the case of modulus of elasticity (MOE) of WPCs, values ranged from

1.61 to 1.92 GPa. The results showed MOE increased between 2.10% and 19.25% compared to that of the control group. There was no significant difference between the controls and all specimens based on the statistical analysis, except for group B.

The mean TS values ranged from 15.68 to 20.28 MPa (Table 3). Similar to the MOR values, the lowest value was recorded for group A. Statistical analysis showed a significant difference between control group and groups A, D, and F.

The mean IS values ranged from 13.97 to 16.24 J m<sup>-1</sup> (Table 3). In contrast to other mechanical properties, group A provided the highest impact strength value. Statistical analysis showed a significant difference between group A and groups B, C, and D.

### Burning rate tests

The effect of FRs on the burning rate of WPCs is presented in Table 3. The mean burning rate values ranged from 12.49 to 23.27 mm min<sup>-1</sup>. Group D showed the lowest burning rate, with control specimens displaying the highest burning rate. Loading of FRs decreased the burning rate between 36.10% and 46.33% compared to control specimens. There was no significant difference between burning rate values of the formulations containing FRs.

## Discussion

### Physical properties

The average specific gravity and moisture content of the manufactured WPCs fell within a narrow range of 0.93%-1.07% and 0.29%-0.89%, respectively. The control specimens exhibited the lowest density values. The specific gravities of groups having FRs were close to each other. Specific gravities of PP, wood flour, MAPP, APP, BA, BX, and ZB were 0.90, 1.30, 0.91, 1.9, 1.44, 1.82, and 2.77, respectively, in the references (Kylosov 2007; Clariant 2008a, 2008b; Toker 2007; Eti Holding 2003). Specific gravities of wood flour, PP, and FRs (mostly APP) define the specific gravities of WPCs produced. Groups having FRs in their formulations had additional 22% to 25% APP. The main reason for higher specific gravities of groups was having FRs in their formulations.

WA and ST values are quite important for the final use of the WPCs. The mean WA and ST (24 h) values of WPCs were 0.41%-2.36% and 0.59%-1.76%, respectively. The WA values were lower than reported WA values for WPCs, i.e. 3% (Kylosov 2007). The water sorption of WPCs was lower than that of wood. Lower WA and ST values indicated wood flour

were more likely coated with plastic and blocked the pathway for moisture penetration (Wang and Morrell 2004). The control specimens had the lowest WA value and groups having FRs in their formulations had higher WA values due to higher hygroscopicity resulting from use of FRs (LeVan 1984).

### Mechanical properties

MOR of the WPC formulation having 25% APP (group A) was significantly lower than that of the WPC control group. Since there was no reaction between APP and either wood or polymer matrix, this reduction was expected. It should also be noted that the control group contained more polymer compared to other groups. Although the polymer matrix was fixed as 67% in the formulations, groups with FRs had extra 25% materials in the formulation, which reduced the actual amount of polymer in the formulation from 67% to 54%. This could be another cause of lower MOR values.

Interestingly, formulations having APP with BA and BX together did not show this adverse affect. The combination of BA and BX with APP seems to overcome the negative effect of the APP. Jimenez et al. (2006a) used BA and APP derivatives in the coating formulation of thermoset epoxy-amine resin system and reported an improved mechanical resistance of the char and adhesion due to a synergetic agent present in the formulation. In another study, Jimenez et al. (2006b) detected the boron phosphate peak through solid state NMR at around 250 °C, but this peak was not present at 150 °C. They reported that the degradation product of BA and APP reacted together, resulting in the formation of boron phosphate, which was thought to be responsible for the improved mechanical resistance of the char. In our study, both extrusion and compression molding were used during manufacturing, exposing samples to a prolonged processing time and high temperature. The process had the potential to provide a good environment for the formation of the boron phosphate, which might have been responsible for the improved properties. ASTM D 6662 (2001b) standard requires the minimum MOR values of 6.9 MPa (1000 psi) for polyolefin-based polymer lumber decking boards. WPCs produced in this study provided flexural strength values (31-38 MPa) well over the requirement by the standard.

In contrast to the MOR values, in modulus of elasticity (MOE) values of WPCs, there was no significant difference between the control group and group A. Results showed formulations with the BA/BX mixture have slightly higher MOE values compared to formulations without the BA/BX mixture (control and group A). This could be due to the crystalline formation on these samples since increase crystallinity might increase the MOE results. SEM images of the group C at 100 and 1000 magnification shows the formation of crystalline on the samples, which might be responsible for the higher MOE values (Figure 1). The mean MOE values ranged from 1.61 to 1.92 GPa. The ASTM D 6662 (2001b) standard MOE is 0.34 GPa (50,000 psi) for polyolefin-based polymer lumber decking boards. WPCs produced in this study provided MOE (1.61-1.92 GPa) well over the requirement by the standard.

The mean TS values of the produced WPCs ranged from 15.68 to 20.28 MPa (Table 3). Similar to the MOR results, addition of the APP reduced the tensile strength of the samples. Once again, presence of the BA/BX mixture and APP together in the formulation overcame this adverse affect on the TS of the WPCs produced. It is thought that the formation of boron phosphate from the degradation product of BA and APP could be responsible for this improvement.

In the case of impact strength (IS), the mean IS values ranged from 13.97 to 16.24 J m<sup>-1</sup> (Table 3). WPCs having BA/BX in their formulation showed lower impact strength in general compared to both

the control group and group A. Usually tested brittle composite samples provides lower IS values (Mengeloğlu and Karakuş, 2008).

### Burning rate tests

The mean burning rate values ranged from 12.49 to 23.27 mm min<sup>-1</sup>. Group D showed the lowest burning rate and control specimens showed the highest. Even so, all formulations, except the control group, provided statistically similar burning rates. Boron compounds and APP have similar flame retarding effect and they can promote carbonification during the process of combustion (Camino et al. 1984; Montaudo and Scamporrino 1985). Formation of char and no polymer dripping were observed in WPCs that had 25% FRCs loadings. This observation was similar with previous studies done by Le Bras et al. (2005). The char observed during burning of the FR WPC acts first as an insulator to keep condensed phase at lower temperatures, thus reducing volatile fuel; and second as a barrier to prevent the volatile fuel from reaching the flame front (Savides et al. 1979).

In conclusion, during the formulation of WPCs with good mechanical and flame retardancy properties, BA/BX and APP should be used together. Addition of small amounts (3%) of various BCs acted as synergists to the WPC formulation having APP as a FR. Reduced mechanical properties due to the use of APP would be recovered without losing the fire performance of the composites.

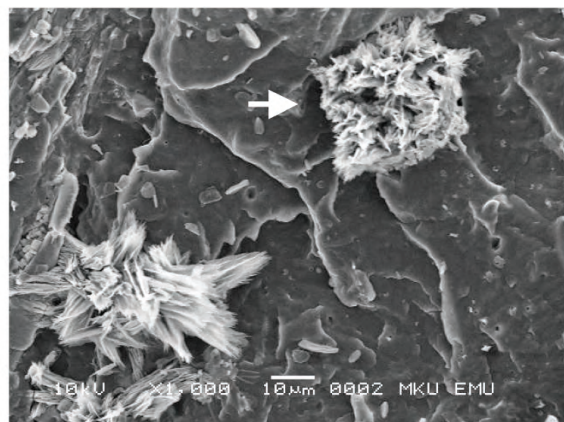
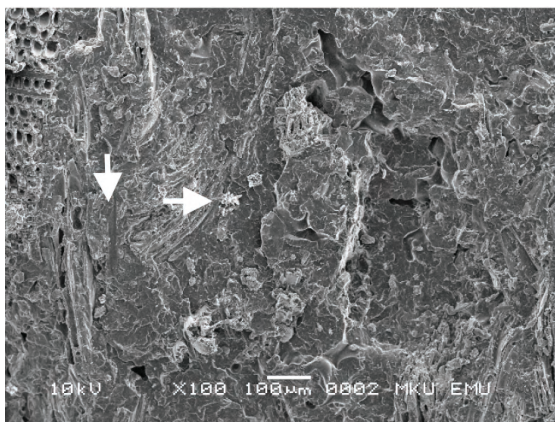


Figure 1. SEM images of WPCs a) group C at  $\times 100$  magnification, and b) group C at  $\times 1000$  magnification.



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