



Investigations of Flame Retardant Properties of Zinc Borate Accompanying with Huntite and Hydromagnesite in Polymer Composites

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Abstract

Fires have been a danger to human beings for the centuries. As people lost their lives and property in fires, they tried to fight the fire and their efforts in this area continued increasingly. Unfortunately, it is still not possible to completely reset the risk of starting the fire. But it seems likely to extinguish immediately after the fire has started, and this is very important to save people's lives. Scientists have been working in this field in recent years; they are concentrated on producing fire-resistant materials using different materials. This can be provided by different ways; either fire-resistant material can be produced new, or the fire resistivity can be provided by incorporating the additive material into a flammable material. In our previous studies, we used huntite and hydromagnesite minerals to give fire resistance property to polymer materials, very successful results were obtained. In this study, huntite and hydromagnesite minerals were used for accompanying with zinc borate in polypropylene composites in order to increase the flame retardant property of a polymeric materials. Different content of minerals were blended with polypropylene, and composites were produced by twin-screw extruder for observing synergistic effect. Scanning electron microscopy (SEM) analyses were conducted to determine the structural and morphological properties of the composites. Thermal properties were determined according to thermogravimetric analysis (TGA). Tensile and three point bending tests were carried out to obtain mechanical properties. Flame retardant performance was evaluated according to UL 94 vertically flammability test. It was concluded that very good synergistic effects were obtained that zinc borate was significantly influential with huntite/hydromagnesite in the flammability characteristics of composites because higher char formation is observed with zinc borate addition. Moreover, the zinc borate reduced the smoke generated during combustion.

Keywords: flame retardancy, huntite hydromagnesite, zinc borate, polypropylene, mechanical properties, thermal degradation, synergistic effect

1. Introduction

Polypropylene (PP) is the most widely used commodity polymer in different commercial applications due to its good impact strength, good chemical resistance, good electrical insulation, low cost and easy processing. However, despite these wonderful features, PP is also known with its flammable property. Aliphatic hydrocarbon structure of this polymer gives rise to its severe flammability [1]. As pointedly, it should be investigated in order to enhance the usage of the polymer against fire to reduce losses of life and property [2-5]. With this regard, tendency of the addition of the flame retardant and the smoke suppressant additives has increased rapidly. Their effect is to drop off the chances of a fire starting with providing resistance to ignition. Even if ignition does occur, flame retardants can act to delay the spread of flame, provide extra time in the early stages so the fire can be extinguished or an escape can be made [6, 7].

Flame retardant additives are generally classified into two groups; halogenated and halogen free materials. Some halogenated ones are tris(2-chloroethyl) phosphate, tris(1,3-dichloroisopropyl) phosphate, pentabromobenzyl acrylate and tris(1-chloro-2-propyl) phosphate. Indeed, they are not very preferred in the recent times because they release toxic gas to the environment and they can affect the endocrine, immune,

reproductive and nervous systems. Some animal studies have shown that long-term exposure to those flame retardants can lead to cancer [8]. Halogen free flame retardants, which involve phosphorus, nitrogen, silicone, boron, zinc, iron, and aluminum-containing materials are considered as non toxic and environmentally harmless. In order to increase flame resistivity of polymers, environmental friendly systems have been also favored lately [9-11].

Huntite $[\text{Mg}_3\text{Ca}(\text{CO}_3)_4]$ and hydromagnesite $[\text{Mg}_2(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}]$ minerals are defined as a natural mixture and classified as salt type carbonate minerals. This naturally formed mineral is commercially mined and processed as an alternative to the commonly used flame retardants because of its attractive properties, such as low smoke generation, environmentally safe, halogen free, recyclable, noncorrosive, and low combustion. At present, economically important reserves of huntite and hydromagnesite are found in Turkey and Greece. Production facilities in Turkey have lowered the cost, improved quality control, and should further the use of these minerals. [12-16]. The thermal degradation of huntite and hydromagnesite gives off both water and carbon dioxide when heated, with a minor loss at about 230°C, and major losses at about 420 and 550°C [17]. On the other hand, Zinc borate (ZnB) that is an effective inorganic

Tab. 1. Designation and composition of PP and PP composites
 Tab. 1. Oznaczenie i skład PP i kompozytów PP

Sample Code	PP (weight per cent)	Huntite/Hydromagnesite (weight per cent)	ZnB (weight per cent)
PP	100	0	0
PP80H10ZnB10	80	10	10
PP70H20ZnB10	70	20	10
PP60H30ZnB10	60	30	10
PP50H40ZnB10	50	40	10
PP40H50ZnB10	40	50	10
PP70H30	70	30	0
PP70ZnB30	70	0	30

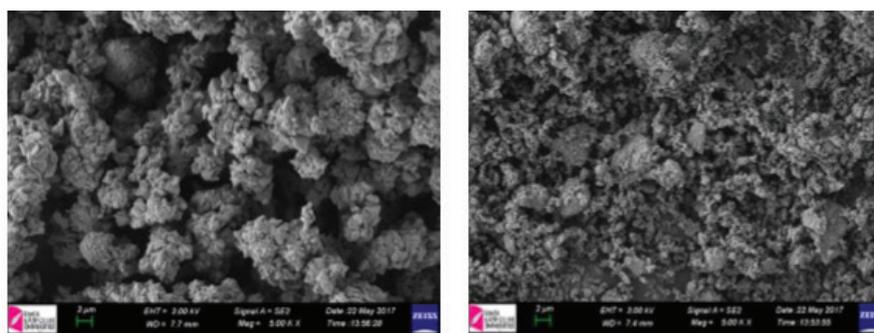


Fig. 1. SEM micrographs of the powders of (left) Zinc Borate and (right) Huntite/Hydromagnesite
 Rys. 1. Mikrografie SEM proszków (po lewej) boranu cynku i (po prawej) Huntite/Hydromagnesite

ic flame retardant additive is widely used both solely and in combination with other materials. Its influence as corrosion inhibitor, smoke suppressant, afterglow suppressant, synergistic agent improves elongation properties of polyolefins reported and emphasized by many authors [18–20].

To observe the synergistic effect with huntite and hydromagnesite and to increase the flame retardant property of the polymer composite, ZnB is used as a collateral additive in this study. Obtained samples were characterized by scanning electron microscopy (SEM) analysis to determine the structural and morphological properties of the composites. Tensile and three point bending tests were carried out to obtain mechanical properties. Thermal properties were determined according to thermogravimetric analysis (TGA). Finally, flame retardant performance of samples were observed according to UL 94 vertically flammability tests.

2. Experimental Section

2.1. Materials

Polypropylene (PP) pellets were used as a main polymer matrix, were supplied by Petkim Petrochemical Co. (Izmir, Turkey) with the melt flow index of 4.7 g/10 min (MFI; 230°C/2.16 kg, ASTM D1238), melting point of 163°C and density of 0.905 g/cm³. All the samples of natural huntite/hydromagnesite, which were used to improve flame retardancy properties of PP, were supplied from Tirtar region of Isparta. Commercial zinc borate was supplied by MELOS A.S. to increase flame retardancy properties of PP with huntite and hydromagnesite and its particle size range from 5 to 45 µm.

2.2. Sample Preparation

Polypropylene based composites containing huntite/ hydromagnesite and zinc borate additives were produced by us-

ing twin-screw extruder (co-rotate) (Labtech) with L/D ratio of the 44:1 at rpm. Screw speed was adjusted as 190 rpm and zone temperatures of extruder were selected between the temperatures 170°C and 185°C. 0, 10, 20, 30, 40, 50 wt% huntite/hydromagnesite and 10, 30 wt% zinc oxide reinforced PP composites were manufactured to observe synergistic effect (Table 1).

The samples for thermal, mechanical and flammability tests were produced by using laboratory scale press (Hydraulic Laboratory Press, Labtech) which was heating and cooling platens at 200°C for 9 min. according to ISO R 527 and ISO 178, respectively.

2.3. Characterization of Composites

The surface morphology and microstructure of ZnB, huntite/hydromagnesite and composite samples were characterized by a scanning electron microscope (SEM, Zeiss Sigma 300 VP-FESEM). Thermal properties of the all composites were investigated by TGA analyzer (Perkin Elmer, STA 8000) by heating from room temperature to 600°C at a heating rate of 10°C/min under nitrogen atmosphere. Tensile testing samples were obtained by using a mold which cut the plate with blades according to ASTM D638 standard. Tensile properties were tested by using tensile test machine (SHIMADZU, AGS-X 5kN) with a crosshead speed of 50 mm/min. The flexural modulus and flexural strength of samples were tested by test machine (SHIMADZU AGS-X 5kN) which involves a three-point bending tests at a crosshead speed of 1 mm/min according to DIN EN ISO 178. Vertical burning test (UL 94) were conducted to investigate the flammability properties of composites on the test bars of 125 x 13 x 3.2 mm³ angular radius 1.3 mm, according to American National Standard UL-94 standard.

3. Results and Discussion

Tab. 2. TGA results of the samples

Tab. 2. Wyniki TGA próbek

Sample Code	$T_{1\%}$ (°C)	T_{max} (°C)	Char Residue at 600 °C (%)
PP	416.80	451.06	1.32
PP80H10ZnB10	439.94	457.89	14.89
PP70H20ZnB10	425.50	459.94	21.26
PP60H30ZnB10	438.46	461.25	25.05
PP50H40ZnB10	434.70	456.07	28.72
PP40H50ZnB10	431.35	452.44	35.56
PP70H30	435.14	456.57	15.23
PP70ZnB30	422.69	446.71	25.32

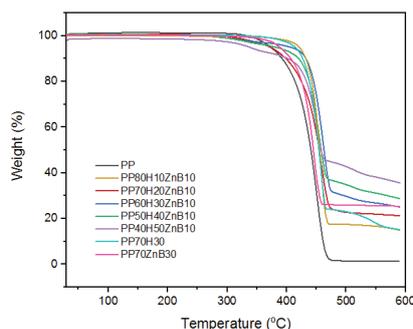


Fig. 2. TGA curves of the samples

Rys. 2. Krzywe TGA próbek

Fig.1 shows the SEM images of ZnB and huntite/hydromagnesite powder. As can be seen from the Fig.1-a that the particle size of ZnB is in between 5–10 micron; however, there seems also some agglomeration. Huntite/hydromagnesite particles change from 1 to 10 micron. To prevent agglomeration, some deflocculating materials can be used.

Fig. 2 shows the detected TGA curves of the samples. The initial degradation temperature ($T_{1\%}$), the maximum degradation temperature (T_{max}) and the char residue values of PP and PP composites at 600°C are shown in Table 2. Degradation temperature for polypropylene matrix starts at 416.80°C and ends at 451.06°C. Initial degradation temperature of PP matrix was retarded approximately 15°C by adding maximum amount of huntite/hydromagnesite (50%) and 10% Zinc Borate. Moreover, it is seen in Fig. 2 and Table 2, increasing percentage of additives increases char residue. Maximum degradation temperature of the composites alters at the higher temperatures compared with pure PP, because of char layer formed by flame retardants. Therefore, PP matrix is protected from heat and combustion [21]. PP70H30 and PP60H30ZnB10 have 435.14°C and 438.46°C initial temperature of the sample's decomposition process. ($T_{1\%}$), respectively. The difference of the initial degradation temperature between PP70H30 and PP60H30ZnB10 shows that synergistic effect of zinc borate on huntite/hydromagnesite reinforced PP.

Tensile strength, Young's modulus, Flexural strength and modulus values of the samples are listed in Fig. 3 and Table 3. It can be seen that the tensile strength and strain properties were deteriorated by increasing huntite/hydromagnesite mineral quantity. Tensile strength of the pure PP was 35.28 MPa. The values were estimated as 24.81 MPa, 28.48 MPa, 28.00 MPa, 25.22 MPa and 21.22 MPa with regard to 10%, 20%, 30%, 40% and 50% of huntite/hydromagnesite and zinc

borate addition, respectively. This decreasing is related to the poor compatibility between filler and polymer matrix which can be improved by the use of filler surface treatment [26]. In the tensile elongation results, it was obtained that increasing amount of mineral powder decreased the tensile elongation of PP by 91%; strain value of pure PP is 20.76% and 1.10% for PP40H50ZnB10 [26, 27]. The plastic deformation of the polymer matrix and isolated filler particles are related to poor adhesion, which is directly related with the decrease of elongation for the samples with higher filler content [22]. In addition, agglomeration of the additives is another reason for deterioration of the mechanical properties due to the weakened interaction between the agglomerated filler and polymer matrix [23]. This interaction is hindered at higher amount additive contents, resulting in lower strength of the composites [24]. Elongation is inversely proportional to the modulus of a material [28, 29]. Elastic modulus of pure PP is 0.996 GPa, while PP40H50ZnB10 is 2.257, thus it is increased by 126%. It is markedly improved by adding fillers that have higher stiffness than the matrix and are able to improve the modulus of composite to a polymer matrix [30].

If the comparison is made between huntite/hydromagnesite and zinc borate reinforced samples, it can be seen that there is no big difference in tensile strength and elastic modulus values, as PP70H30 has 24.52 MPa while PP70ZnB30 has 23.43 MPa tensile strength, and PP70H30 has 1.480 GPa while PP70ZnB30 has 1.389 GPa elastic modulus. However, there is a big difference in strain values because P70H30 has 2.62% while PP70ZnB30 has 13.67%. Therefore zinc borate containing samples have higher ductility than huntite/hydromagnesite reinforced samples.

Flexural strength of pure PP is 44.53 MPa. It decreased with increasing huntite/hydromagnesite content as following;

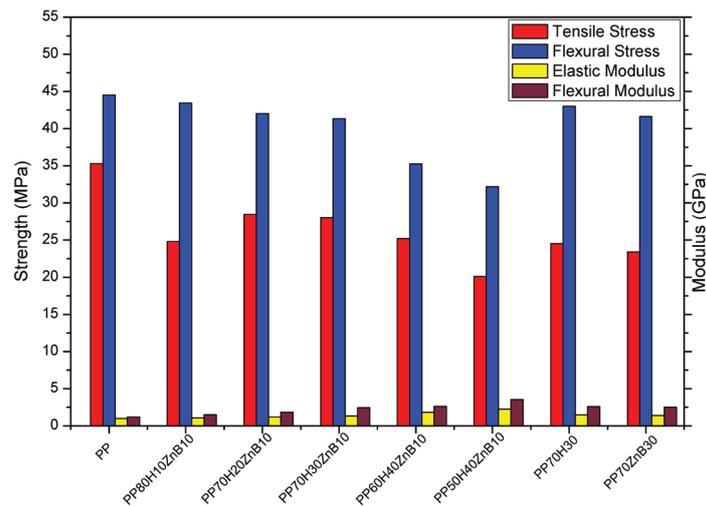


Fig. 3. Tensile&Flexural Strength and Elastic&Flexural Modulus of neat PP and composites

Rys. 3. Wytrzymałość na rozciąganie/wytrzymałość na zginanie i moduł sprężystości/na zginanie czystego PP i kompozytów

Tab. 3. Mechanical test results of the samples

Tab. 3. Wyniki badań mechanicznych próbek

Samples	Elastic Modulus (GPa)	Tensile Strength (MPa)	Tensile Strain (%)	Flexural Modulus (GPa)	Flexural Strength (MPa)	Flexural Strain (%)
PP	0.996	35.28	20.76	1.197	44.53	7.46
PP80H10ZnB10	1.084	24.81	5.02	1.521	43.44	4.97
PP70H20ZnB10	1.196	28.48	4.36	1.815	42.03	4.94
PP60H30ZnB10	1.351	28.00	3.82	2.447	41.36	4.25
PP50H40ZnB10	1.828	25.22	2.04	2.615	35.26	3.66
PP40H50ZnB10	2.257	21.22	1.10	3.536	32.17	1.31
PP70H30	1.480	24.52	2.62	2.601	43.01	4.72
PP70ZnB30	1.389	23.43	13.67	2.520	41.62	4.45

43.44 MPa, 42.03 MPa, 41.36 MPa, 35.26 MPa and 32.17 MPa for 10%, 20%, 30%, 40% and 50% huntite/hydromagnesite content, respectively. 30% huntite/hydromagnesite and zinc borate added composite values are 43.01 MPa and 41.62 MPa, respectively. It can be said that zinc borate has less effect on decreasing flexural strength value of PP than huntite/hydromagnesite filler. The highest huntite/hydromagnesite content is added into PP, the strain is decreased by 82.4%. Flexural modulus is increased with the filler content from 1.197 GPa for pure PP up to 3.536 GPa for PP60H50ZnB10.

Fig. 4 represents the SEM micrographs of tensile fractured surfaces of PP based on the composites which are PP70H20ZnB10, PP60H30ZnB10, PP50H40ZnB10 and PP60H50ZnB10 composites. Morphology of samples was investigated by 1000 times magnification. Pure PP shows ductile behaviour. It is seen that the fractured morphologies of huntite/hydromagnesite and ZnB reinforced composites show resembling brittle failure by increasing amount of huntite. If SEM images of the samples of PP70H20ZnB10 and PP40H50ZnB10 are compared, it can be found out that 20% reinforced composite shows the ductile fracture surface which is separated as fibers.

Flame retardant properties of specimens have been investigated by UL 94 vertical test method. Flame starting time, flame growing time, UL 94 rating and cotton ignition results of the samples are indicated in Table 4. Fig. 5 shows that photographs of the samples after UL 94 testing. Flaming time

of all composite samples are higher than neat PP. The flame starting and growing times were delayed by 5 seconds with the addition of 50% huntite/hydromagnesite in composites. It can be seen that UL 94 rating is changed from V2 to V1 by increasing the amount of huntite/hydromagnesite from 20% to 30% in the composite. This result is consistent with the results found in the Ref [31] by 50% huntite/hydromagnesite addition to thermoplastic polyurethane, thus UL 94 rating was achieved as V1. The work principle of the additives is related with the dilution effect in gas phase by the help of water and CO₂ release, endothermic decomposition and protective char layer [12, 21, 31, 32]. On the other hand, the assistive effect of ZnB was observed, and this effect is considered that it is based on increasing barrier effect of char layer in the condensed phase and formation of incombustible gasses due to the acid base interactions. As mentioned Table 2, the char layer was 25.32% for the sample of PP70ZnB30 while it is 15.23% for PP70H30. Dike et al. [20] obtained the similar result of contribution of ZnB as synergistic additive for flame retardant in polymer.

4. Conclusion

Synergistic effect of ZnB on the flame retardant, mechanical and thermal properties of huntite/hydromagnesite reinforced PP composites were investigated. Composites were produced by using twin-screw extruder. SEM observations showed that there is no interaction between the flame retardant additives and polypropylene, except to mechan-

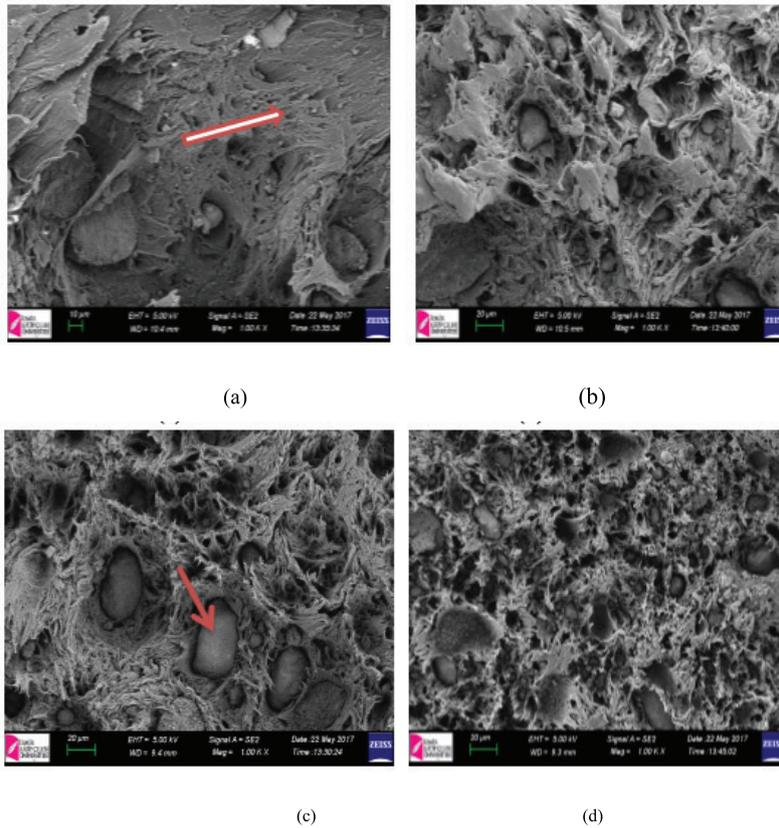


Fig. 4. SEM micrographs of (a) PP70H20ZnB10 (b) PP60H30ZnB10 (c) PP50H40ZnB10 and (d) PP60H50ZnB10 composites
 Rys. 4. Mikrografie SEM (a) PP70H20ZnB10 (b) PP60H30ZnB10 (c) PP50H40ZnB10 i (d) PP60H50ZnB10

Tab. 4. UL 94 Vertical test results of the samples
 Tab. 4. UL 94 Pionowe, wyniki badań próbek

Samples	Flame starting time (s)	Flame growing time (s)	UL 94 (Vertical) rating	Cotton ignition
PP	5	8	No rate	Yes
PP80H10ZnB10	7	9	V2	Yes
PP70H20ZnB10	8	10	V2	Yes
PP60H30ZnB10	10	14	V1	No
PP50H40ZnB10	12	14	V1	No
PP40H50ZnB10	12	14	V1	No
PP70H30	10	13	V1	No
PP70ZnB30	10	12	V1	No

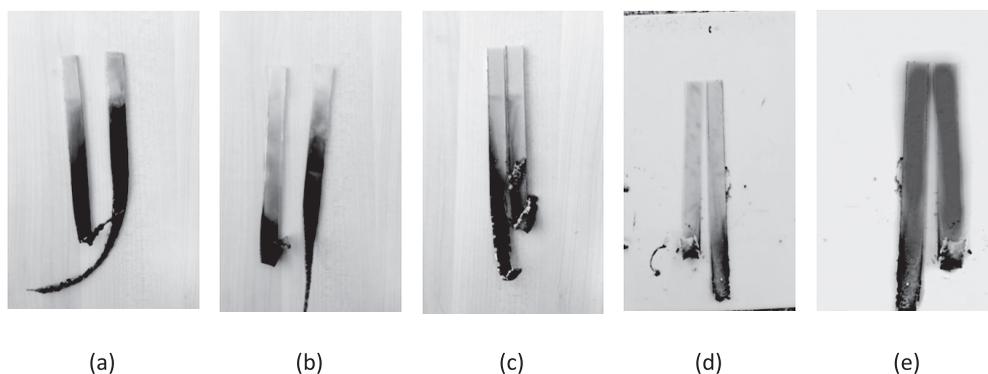


Fig. 5. Photographs of selected after UL 94 test sample of (a) PP (b) PP70H20ZnB10 (c) PP60H30ZnB10 (d) PP50H40ZnB10 and (e) PP40H50ZnB10

Rys. 5. Zdjęcia wybranych próbek testowych UL 94 (a) PP (b) PP70H20ZnB10 (c) PP60H30ZnB10 (d) PP50H40ZnB10 i (e) PP40H50ZnB10

ical adhesion. Besides, fractured morphologies of huntite/hydromagnesite and ZnB reinforced PP composites showed resembling brittle failure by increasing amount of huntite/hydromagnesite in PP composite. According to TGA results, increasing additive amount of minerals decreased the degradation weight. Additive minerals increased the maximum degradation temperature compared to PP. Initial degradation temperature of polypropylene was increased approximately 15°C by the addition of 50% huntite/hydromagnesite and 10% Zinc Borate into PP matrix. Tensile strength and strain values of polypropylene composites decreased with increasing loading level. The tensile strength of PP was around 35 MPa. The highest loading of 50 wt.% decreased the tensile strength of PP to 21 MPa. However, elastic modulus was increased with the increasing huntite/hydromagnesite in polypropylene composites. Elastic modulus of PP was 1000 MPa and the highest loading of 50wt.% increased the elastic modulus to 2.3 GPa. Flexural modulus values were increased with the

addition of mixtures of huntite/hydromagnesite into the polymeric matrix. The flexural modulus value of PP was around 1.2 GPa and this was much lower than loading levels of 10–20–30–40–50% by weight of huntite/hydromagnesite filled PP. UL94 vertical flammability test results showed that PP burned with melt dripping. 50% huntite/hydromagnesite and 10% ZnB reinforced polymer composites were much more successful in burning test because fire starting time was higher. The addition of ZnB caused significant synergistic effect with huntite/hydromagnesite in the flammability characteristics of composites. Higher char formation is observed by zinc ZnB addition. In addition, it reduced smoke that generated during combustion.

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Badania właściwości ogniochronnych boranu cynku w obecności huntitu i hydromagnezytu w kompozytach polimerowych

Pożary od wieków stanowią zagrożenie dla ludzi. Gdy ludzie stracili życie i mienie w wyniku pożarów, próbowali zwalczyć pożar, a ich wysiłki w tej dziedzinie były coraz bardziej skuteczne. Niestety, nadal nie jest możliwe całkowite wyeliminowanie ryzyka rozpoczęcia pożaru. Ważne jest ugaszenie pożaru natychmiast po wybuchu aby uratować ludzkie życie. Naukowcy pracują w tej dziedzinie w ostatnich latach; koncentrują się na wytwarzaniu materiałów ognioodpornych przy użyciu różnych surowców. Można to zapewnić na różne sposoby; albo materiał ognioodporny może być wytworzony jako nowy, albo ognioodporność może być zapewniona przez włączenie materiału dodatkowego do materiału łatwopalnego. W badaniach używano minerałów huntite i hydromagnesitu, aby nadać materiałom polimerowym właściwości ognioodpornych, osiągając bardzo udane wyniki. W artykule przedstawiono wyniki wykorzystania minerałów huntitu i hydromagnezytu w obecności boranu cynku w kompozytach polipropylenowych w celu zwiększenia właściwości ognioodpornych materiałów polimerowych. Różne ilości minerałów zmieszano z polipropylenem, a kompozyty wytworzono za pomocą wylączarki dwuślismakowej w celu zaobserwowania efektu synergicznego. Przeprowadzono analizy skaningowej mikroskopii elektronowej (SEM) w celu określenia właściwości strukturalnych i morfologicznych kompozytów. Właściwości termiczne określono za pomocą analizy termogravimetryczną (TGA). Przeprowadzono próby rozciągania i zginania trzypunktowego w celu określenia właściwości mechanicznych. Działanie zmniejszające palność oceniono zgodnie z testem palności w pionie UL 94. Stwierdzono, że uzyskano bardzo dobre efekty synergiczne, boran cynku miał znaczący wpływ na huntit i hydromagnezyt i zmianę charakterystyki palności kompozytów, ponieważ obserwuje się podwyższone powstawanie węgla drzewnego przy dodawaniu boranu cynku. Ponadto boran cynku zmniejszył ilość dymu powstający podczas spalania

Słowa kluczowe: *uniepalniacz, hydromagnezyt huntitu, boran cynku, polipropylen, właściwości mechaniczne, degradacja termiczna, efekt synergiczny*