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# Developing the Blends of Low-Density Polyethylene with Al-Zn Borate for Industrial Purposes

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**Abstract:** In this study, the blends of low-density polyethylene with aluminum-zinc borate have been prepared for further use of those blends in the packaging, wire, and cable industries. The aluminum-zinc borate was used as an additive in the low-density polyethylene with different molecular weights and the mechanical and physical properties. The melt flow rate of the samples decreased depending on the amount of aluminum-zinc borate. An increase in the tensile strength values when aluminum-zinc borate ratios were 0-1 wt.% and a decrease when the aluminum-zinc borate ratios were 5-10 wt.% were observed. Tensile strength values of the samples were measured as 12.28MPa in the sample with 0 wt.% aluminum-zinc borate, and 5.91MPa in sample with 10 wt.% aluminum-zinc borates. The elongation at break of the film with 0 wt.% aluminum-zinc borate was 507.3 percent and while the elongation value of the film with 10 wt.% aluminum-zinc borates was 285.5 percent. According to the results of thermogravimetric analysis, it was observed that the thermal stability of the polymers changed by adding aluminum-zinc borate. Considering the mass losses in terms of the temperature values, the F2-21T sample reached the highest thermal stamina. While the thermal stability of F5-21T increased significantly, the thermal stability of I22-19T also increased. The results showed that all composites are suitable for further use in packaging, wire, and cable applications.

**Keywords:** Aluminum-zinc borate, low density polyethylene, mechanical and thermal properties, polymer blends, industrial applications.

# Endüstriyel Amaçlı Düşük Yoğunluklu Polietilen ile Al-Zn Borat Karışımlarının Geliştirilmesi

**Özet:** Bu çalışmada, ambalaj, tel ve kablo endüstrileri için düşük yoğunluklu polietilen ile alüminyum-çinko borat karışımlarının geliştirilmesi incelenmiştir. Alüminyum-çinko borat, farklı moleküler ağırlık ve mekanik ve fiziksel özelliklere sahip düşük yoğunluklu polietilende katkı maddesi olarak kullanılmıştır. Örneklerin eriyik akış hızları alüminyum-çinko borat miktarına bağlı olarak azalmıştır. Alüminyum-çinko borat oranları ağırlıkça %0-1 olduğunda çekme dayanımı değerlerinde artış, alüminyum-çinko borat oranları ağırlıkça %5-10 olduğunda ise azalma gözlenmiştir. Numunelerin çekme dayanımı değerleri ağırlıkça %0 alüminyum-çinko boratlı numunede 12,28MPa, ağırlıkça %10 alüminyum-çinko boratlı numunede 5,91MPa olarak ölçülmüştür. Filmin kopma uzaması ağırlıkça %0 alüminyum-çinko borat ile yüzde 507,3 iken, filmin ağırlıkça %10 alüminyum-çinko borat ile uzama değeri yüzde 285,5 oldu. Termogravimetrik analiz sonuçlarına göre, alüminyum-çinko borat ilavesiyle polimerlerin ısıl kararlılığının değiştiği gözlenmiştir. Sıcaklık değerleri açısından kütle kayıplarına bakıldığında F2-21T numunesi en yüksek ısıl dayanıklılığa ulaşmıştır. F5-21T'nin termal kararlılığı önemli ölçüde artarken, I22-19T'nin termal kararlılığı da arttı. Sonuçlar, tüm kompozitlerin paketleme, tel ve kablo uygulamalarında daha fazla kullanım için uygun olduğunu gösterdi.

**Anahtar Kelimeler:** Alüminyum-Çinko borat, düşük yoğunluklu polietilen, mekanik ve termal özellikler, polimer karışımlar, endüstriyel uygulamalar.

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# 1. INTRODUCTION

Manufacturing plastic materials of the devices like various containers, bottles, plastic bags, and molded laboratory equipment are some of the applications of the polymers in daily life [1,2]. To reach these qualities, cross-linking polymers with organic peroxides are the main preferred process [3-5]. Especially, low-density polyethylene (LDPE) is widely used in many fields owing to its good electrical insulation, low cost, and easy processability [6]. On the other hand, their easy combustibility causes easy burnings [7] and limits their advantages [5-10]. Enhancing this weakness plays major role in finding new fields of usage. For this purpose, research efforts have been focused on trying new additives with polymers to improve the properties of polymers [8, 11-13]. Among those additives, aluminum, zinc, and several other ions can be counted [13].

In many fire-resistant applications, zinc borate ($2ZnO.3B_2O_3.3.5H_2O$) has proven to be more flame retardant than borates used alone after the research in halogen-free flame retardant has become a popular issue [6, 14]. Previous research efforts have already completed the production, characterization, and kinetics of zinc borate with the formula $2ZnO\ 3B_2O_3\ 3.5H_2O$ [5, 13-16]. Thus, it can be added to the hot polymer charge. Zinc Borate has an index like the refractive index of many polymer systems. Therefore, it allows both low pigment load and preserves translucency. It can be added to other solid polymer additives by a similar method, namely extruders, or injection molding [15-17].

The advantages of zinc borate are counted as flame retardants in a wide variety of plastics [14, 16, 17]. However, its hard oxidization property [18, 19] has not been studied enough for its usage as a packaging material and as a cable insulator. No special tools are needed during the process, it does not absorb moisture and is insoluble in water [20, 21]. For all these reasons, this study investigated to use of aluminum-zinc borate as an additive to the samples of LDPE at different ratios (1, 2.5, 5, and 10 wt.%) to prepare samples for enhancing materials for industrial purposes [22].

# 2 MATERIALS AND METHOD

## 2.1 Materials

Low density polyethylene (LDPE F5-21T, LDPE F2-21T, LDPE I22-19T) was supplied as granular by Petkim Petrochemical Company (Aliaga, Izmir, Turkey). LDPE types were produced by tubular method with the help of peroxide catalysts between 2550kg / $cm^2$ and 170-295℃ in PETKİM's Low Density Polyethylene factory. In coding the samples, “F” means film extrusion method and “I” means injection molding method, “5, 2 and 22” means the melt flow rate (g/10min), “21, 21, and 19” means a special code given by the producer, and “T” means tubular method. Molecular weights of the polymers change as LDPE F5-21T > LDPE F2-21T > LDPE I22-19T.

Boric acid was purchased from EtiMaden Operations General Directorate (Kecioren, Ankara, Turkey). NaOH, $Al(NO_3)_3$, and $Zn(NO_3)_2$ were purchased in technical grade.

## 2.2 Preparation of the Samples

In this study, aluminum-zinc borate was synthesized from aluminum (Al), zinc (Zn) and boric acid ($H_3BO_3$) according to previous studies [23, 24]. First, aluminum-zinc complex (Al-Zn) was prepared. Al-Zn complex were prepared at certain temperature (50 ±2°C) by dropwise addition of 0.02M $Zn^{2+}$ and 0.01M $Al^{3+}$ solutions [25]. $Zn^{2+}$ and $Al^{3+}$ solutions were prepared from their nitrate salts. The Zn:Al ratio chosen for the synthesis was 2:1 to obtain stable layered compounds. During this reaction, the pH was kept around (9.0±0.2) by simultaneous addition of 0.1M NaOH. The resultant slurry was filtered and dried at 105 °C in an oven.

**Table 1.** Melt Flow Rates (MFR) values of the polymer samples.

| Polymer name | | Al-Zn borate (weight %) | MFR (g/10 min) |
|---|---|---|---|
| F2-21T | A1 | 0 | 2.55 |
| | A2 | 1 | 2.45 |
| | A3 | 2.5 | 1.35 |
| | A4 | 5 | 0.95 |
| | A5 | 10 | 0.65 |
| F5-21T | B1 | 0 | 4.47 |
| | B2 | 1 | 4.43 |
| | B3 | 2.5 | 3.50 |
| | B4 | 5 | 2.60 |
| | B5 | 10 | 0.80 |
| I22-19T | C1 | 0 | 19.15 |
| | C2 | 1 | 15.73 |
| | C3 | 2.5 | 12.50 |
| | C4 | 5 | 10.50 |
| | C5 | 10 | 6.89 |

Then, boric acid and Al-Zn complex was mixed in the ratio of 4:6 (Table1). LDPE samples were enriched with 0 wt.%, 1 wt.%, 2.5 wt.%, 5 wt.% and 10 wt.% Al-Zn borate additives. Both chemicals were mixed by a single screw cast-type Collin E30 extruder was used to prepare polymer-peroxide samples at a specified temperature, under the weight of a certain load for each component. The samples with 0.25 mm thickness and 10 cm width were laminated by the extruder at 200°C for 2 min [21, 26].

Melting flow rate, tensile strength at break, elongation at break, and thermogravimetric analysis were administered on the prepared pellets of the film samples.

### 2.3 Melt Flow Rate

Melt flow rate was determined by using MFI Tester-Sangyo TP401B apparatus at 190°C and under 2.16-kg weights in accordance with ASTM D1238 test method. Average weight was found with sequentially cut five samples. The preheated sample was shifted to flow freely under loading.
The weight of the flowing sample in 10 minutes was calculated by cutting the flowing material at certain time intervals.

### 2.4 Tensile Strength

Instron tensile strength device (model 4411) was used for tensile testing. To prepare the samples for the tensile strength test, 2 mm thick plates at 190 °C were prepared in accordance with the ASTM D4703 test method. The samples were cut from the plates prepared in the Shinto Metal Industrie SFA-37 press for tensile strength testing in accordance with ASTM D638 with the help of the molding apparatus. The samples were removed from the plates for tensile testing and the values of the samples' elongation at break, tensile strength at yield and tensile strength at break were measured and plotted.

### 2.5 Thermogravimetric (TG) Analysis

To examine weight changes in the sample with temperature in a controlled atmosphere environment, Perkin Elmer Diamond brand TGA analyzer was used. The device allows simultaneous application of TGA and DTA techniques on a single sample. The temperature range of the device is 25-1500 ℃ and the heating rate varies between 0.01-100 ℃ / min. DSC tests of the prepared mixtures were carried out in accordance with ASTM D3418. Small pieces (5-10 mg) were cut from the samples and placed in a differential scanning calorimeter device. In order to change the thermal memory of the sample, the sample was heated up to 600 ℃ at a rate of 10 ℃ / min. After keeping it at 600 ℃ for 1 minute, the sample was cooled again at 10 ℃ / min. Then the same process was repeated and the melting and crystallization points (℃) from the peaks in the thermograms were taken in the second time and the melting and crystallization energies (J/g) from the areas under the peaks were calculated.

### 2.6 Differential Scanning Calorimetry (DSC)

Perkin Elmer Jade DSC device was used for DSC analyses under nitrogen atmosphere. First, the samples were heated from 50 ℃ to 180 ℃ at a rate of 10 ℃ $\text{min}^{-1}$. Then, they were cooled to 50 ℃ at the same rate. This process repeated again. Melting temperatures ($T_m$), crystallization ($T_c$) temperatures and enthalpies of the samples were obtained from this second scan. The maximum point of the endothermic peak during heating was taken as $T_m$ and the highest point of the exothermic peak during cooling as $T_c$. Melting point and crystallization point were calculated from the areas under the respective peaks.

The crystallinity ratio of the mixtures was measured by the total enthalpy method using the following equation.

$$X_c = \frac{\Delta Hf}{\Delta Hcrys} \text{ x } 100 \qquad (1)$$

In this equation (1), $\Delta H_f$ is melting enthalpy (J/g), $\Delta H_{crys}$is 100% Crystallization enthalpy of the crystal polymer (J/g), and $X_c$ is % Crystallinity. In all calculations, the melting temperatures of the LDPE crystals at the equilibrium melting points were taken as 293 $Jg^{-1}$ [27, 28].

## 3 RESULTS AND DISCUSSION

### 3.1 FT-IR

Figure 1 displays the FT-IR results of Al-Zn borate. There are several characteristic peaks determined in this graph. The band at 3373 $cm^{-1}$ represents the stretching of O–H which in the broad band of 3000-3500$cm^{-1}$ [29, 30]. The band at 1638 $cm^{-1}$ is also a sign for the crystal water appearing with bending mode of the H–O–H vibration mode. The band at 1336 $cm^{-1}$ may be the asymmetric stretching of B (3)–O anions. The band at 1063 $cm^{-1}$ is named the asymmetric and symmetric stretching of B (4)– O. The band 666 $cm^{-1}$ points out the symmetric pulse vibration of stretching between triborate anion and zinc [25]. The frequencies around 450 and 550 $cm^{-1}$, in this case at 420 $cm^{-1}$, are assigned to AlO stretching correspond to the vibration of metal-oxygen in pure zinc borate [29, 30].

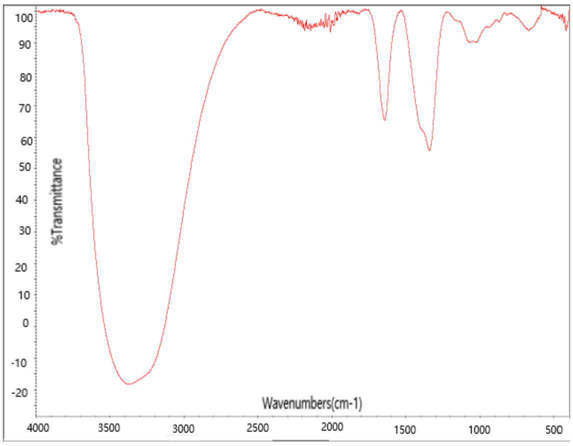

**Figure 1.** FT-IR spectrum of Al-Zn borate

### 3.2 Fluorescence

Fluorescence spectrum was taken in the range between 200-900nm with Thermo Scientific Lumina instrument. 290, 315, 570 nm are the three peaks appeared in the spectra (Figure 2) [31, 32]. 290 nm is corresponded to the s-p transitions of $Al^{3+}$ in the host lattice. The weak peak around 315nm is a sign of $Zn^{2+}$ [33]. The sharp band at 570 nm is located due to the transitions from the excitation state $^5D_4$ to the ground states $^7F_J$ ($J$ = 6, 5, 4, 3) of $Al^{3+}$ in the borate lattice [34].

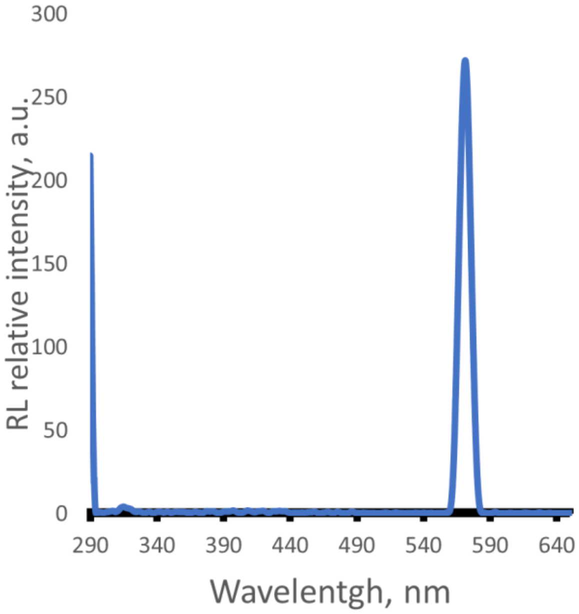

**Figure 2.** Photoluminescence spectrumof Al-Zn borate

### 3.3 Mechanical Analysis

The MFI test results for all the three polymer types are presented in Table 1. According to the table, MFI values dropped with increasing percentage of Al-Zn borate in the samples. For all three types of LDPE showed similar results in MFI measurements. The most dramatic drop was determined in #I22-19T. Adding 1 wt.% Al-Zn borate to the LDPE sample caused the highest difference between MFI values (from 19.15 to 15.73). The same ratio of Al-Zn borate in LDPE#F5-21T produced a decrease from 4.47 to 4.43, and in LDPE#F2-21T reasoned a drop from 2.55 to 2.45.

Considering tensile strength values at yield for all types of the LDPE samples, there was an increase with addition of Al-Zn borate in only for the LDPE#F2-21T polymer samples (Table 2). The value has moved from 7.62 MPa (no additive) to 9.65 MPa (with 10% of Al-Zn borate). For others, there was a drop noticed as seen in Table 2.

Tensile strength values at break showed a drop for all the three blends (A, B, C). The level of drop was low for the A and C film samples (from 11.85 MPa to 9.78 MPa, from 6.60 MPa to 4.68 MPa, respectively) while the B film samples have experienced considerably high drop nearly over 2 times (from 12.28 MPa to 5.91 MPa).

**Table 2.** Tensile strength values of the polymer samples.

| Sample code | Tensile strength at | Tensile strength at | Elongation at break |
|---|---|---|---|
| **A1** | 7.62 | 11,85 | 490,7 |
| **A2** | 7.42 | 11.50 | 467.6 |
| **A3** | 8.52 | 11.29 | 448.3 |
| **A4** | 9.25 | 11.24 | 420.2 |
| **A5** | 9.65 | 9.78 | 395.4 |
| **B1** | 9.60 | 12.28 | 507.3 |
| **B2** | 10.40 | 13.21 | 496.6 |
| **B3** | 9.40 | 12.20 | 459.9 |
| **B4** | 9.22 | 8.64 | 375.8 |
| **B5** | 9.22 | 5.91 | 285.5 |
| **C1** | 9.15 | 6.60 | 225.3 |
| **C2** | 9.06 | 6.15 | 201.1 |
| **C3** | 9.05 | 5.67 | 198.4 |
| **C4** | 8,93 | 5,23 | 190,8 |
| **C5** | 8,73 | 4,68 | 187,3 |

The values of elongation at break pointed out a decrease for all the blends. The highest drop occurred in the B film samples (from 507.3% to 285.5%). Then, the next highest drop was determined in the A film samples (from 490.7% to 395.4%). The lowest drop was in the C film samples (from 225.3% to 187.3%).

Overall, the lowest changes at tensile strength values were obtained in the blends of the C film sample. This means that addition of Al-Zn borate has created the lowest effect on the C blends.

### 3.4 Thermal analysis

In thermal analysis, thermal stability is used for finding how the sample resists against the temperature and how temperature acts on degradation process. According to the Figure 3-5, when the thermal stability of the F2-12T, F5-21T and I22-19T samples were examined, the highest mass losses temperatures of the samples occurred between 300-500 °C, and a stability was observed after 500°C. In Figure 3 and 4, the LDPE blends with 10 wt.% of Al-Zn borates showed a little bit higher degradation temperature than other samples, but the same ratio in Figure 5, there was no difference between the blend samples.

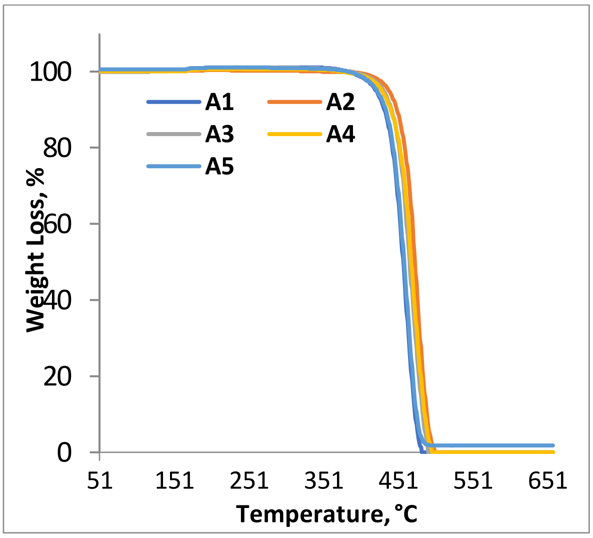

**Figure 3.** TGA diagram of F2-21T with aluminum-zinc borate at different ratio

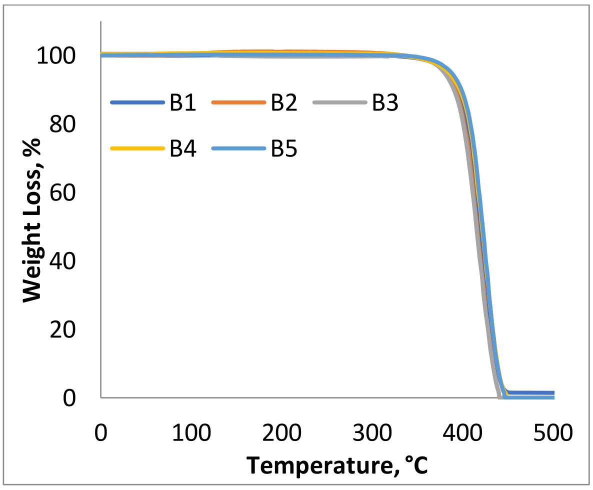

**Figure 4.** TGA diagram of F5-21T with aluminum-zinc borate at different ratio

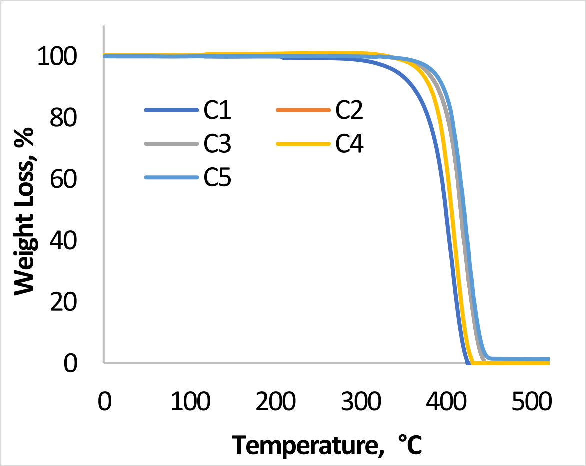

**Figure 5.** TGA diagram of I22-19T with aluminum-zinc borate at different ratio.

Table 3 shows that Al-Zn borate addition to LDPE samples did not change much $T_i$ temperatures except for C4 and C5 samples in which Al-Zn borate ratio was 5 wt% and 10 wt%, respectively. A2, B4, B5, C2, C4, and C5 samples were observed to have the highest heat resistance. Even though A2 had the highest thermal stability with 1 wt% of Al-Zn borate, in general 5 and 10 wt% ratios became projecting the best thermal stability of the samples. There are similar results found in mechanical and DSC analyses.

**Table 3.** TGA analysis of the LDPE blend samples.

| Mass loss | | | | | | Mass loss percentage | | |
|---|---|---|---|---|---|---|---|---|
| Sample code | Conversion (%) | $T_i^a$, °C | $T_{max}^b$, °C | $T_f^c$, °C | 400°C | 450°C | 500°C | Char yield (%) |
| A1 | 0.03-99.97 | 400 | 453 | 481 | 0.90 | 40.20 | 99.70 | 0.03 |
| A2 | 0.03-99.97 | 401 | 468 | 540 | 0.83 | 18.10 | 95.65 | 0.40 |
| A3 | 0.01-99.99 | 406 | 470 | 499 | 0.70 | 15.87 | 99.24 | 0.08 |
| A4 | 0.01-99.99 | 396 | 471 | 505 | 0.60 | 23.05 | 96.46 | 0.35 |
| A5 | 0.02-99.98 | 402 | 470 | 508 | 2.34 | 24.90 | 95.16 | 0.48 |
| B1 | 0.02-99.98 | 380 | 456 | 479 | 0.50 | 37.60 | 99.90 | 0.01 |
| B2 | 0.02-99.98 | 385 | 469 | 507 | 1.48 | 17.31 | 98.62 | 0.14 |
| B3 | 0.04-99.96 | 361 | 460 | 495 | 4.33 | 31.71 | 99.52 | 0.05 |
| B4 | 0.04-99.96 | 384 | 474 | 506 | 1.49 | 15.93 | 95.37 | 0.46 |
| B5 | 0.03-99.97 | 388 | 473 | 504 | 2.54 | 21.13 | 92.80 | 0.72 |
| C1 | 0.04-99.96 | 389 | 454 | 483 | 3.60 | 39.50 | 99.80 | 0.02 |
| C2 | 0.04-99.96 | 362 | 463 | 494 | 3.93 | 29.27 | 99.50 | 0.05 |
| C3 | 0.04-99.96 | 363 | 460 | 488 | 4.22 | 32.20 | 99.90 | 0.01 |
| C4 | 0.04-99.96 | 402 | 469 | 497 | 0.80 | 16.02 | 99.70 | 0.03 |
| C5 | 0.04-99.96 | 401 | 477 | 505 | 2.20 | 26.03 | 99.80 | 0.02 |

[a]:initial decomposition temperature based upon 1% weight loss, [b]:decomposition temperature based upon 50% weight loss, [c]:decomposition temperature based upon 99% weight loss, LDPE: low-density polyethylene, TGA:thermogravimetric analysis

Table 4 points out the DSC analysis of the samples. By adding Al-Zn borate into the LDPE F2-21T samples, the $T_m$, $T_c$ values of the samples were observed to be close to the homopolymer LDPE F2-21T. When the melting, crystallization enthalpies and crystallization rates ($X_c$) of the same samples were examined, an increase was observed compared to Homopolymer LDPE F2-21T. The same is true for the F5-21T. After Al-Zn borate was added to the LDPE I22-19T sample, $T_m$, $T_c$ values did not change much, while the melting, crystallization enthalpies and crystallization rates ($X_c$) of the same samples were first decreased and then increased. This means that a rapid decrease in the number of terminal vinyl groups in crosslinking in polyethylene also confirms this. Increasing long chain branching at a certain molecular weight causes the polymer molecule to shrink. Theseshrinking increase with intramolecular interactions rather than intermolecular interactions. As a result, low density increases intramolecular bonding in polyethylene.

**Table 4.** DSC analysis of the samples

| Melting (2. Heating) | | | Crystallization (2. Cooling) | | Crystallization percent |
|---|---|---|---|---|---|
| **Sample No** | **$T_m^a$ °C** | **$\Delta H_f^b$ (J/g)** | **$T_c^c$ °C** | **$\Delta H_c^d$ (J/g)** | **$X_c^e$ (%)** |
| A1 | 107.47 | 95.88 | 93.07 | 75.07 | 32.72 |
| A2 | 106.98 | 104.85 | 93.56 | 75.18 | 35.79 |
| A3 | 106.83 | 100.23 | 93.34 | 72.07 | 34.21 |
| A4 | 106.95 | 101.18 | 93.76 | 67.40 | 34.53 |
| A5 | 107.51 | 93.78 | 93.20 | 74.72 | 32.01 |
| B1 | 107.12 | 84.53 | 92.42 | 67.86 | 28.85 |
| B2 | 106.45 | 86.89 | 92.60 | 68.54 | 29.66 |
| B3 | 106.15 | 92.40 | 92.70 | 72.68 | 31.54 |
| B4 | 106.13 | 93.01 | 92.58 | 68.30 | 31.74 |
| B5 | 107.03 | 89.51 | 92.64 | 72.29 | 30.55 |
| C1 | 104.63 | 102.77 | 91.21 | 74.43 | 35.08 |
| C2 | 104.83 | 86.03 | 90.83 | 70.78 | 29.36 |
| C3 | 104.31 | 94.86 | 90.89 | 69.99 | 32.38 |
| C4 | 104.68 | 89.80 | 90.18 | 71.36 | 30.65 |
| C5 | 104.83 | 106.09 | 91.18 | 75.69 | 36.21 |

$^a$:melting point, $^b$:enthalpies of fusion, $^c$:crystallinity point, $^d$:enthalpies of crystallinity, $^e$:crystallinity ratio

## 3.5. Conclusion

The results showed that melting flow rates of the samples decreased with the increasing amount of Al-Zn borate. The viscosity of the samples increased due to the increasing amount of Al-Zn borate, and a decrease in the melt flow rate was observed. Considering the tensile strength of the samples, an increase was observed when the Al-Zn borate ratios were 0 wt.% -1 wt.%, and a decrease was observed when it was 2.5 wt.% -10 wt.%. The increase of long chain branching at a certain molecular weight in the structure of LDPE causes the polymer molecule to shrink. This situation leads to an increase in intramolecular interactions rather than intermolecular interactions, in which a decrease in tensile strength at yield values of more than 1 wt% by weight was observed. The values of tensile strength at break point out that with an increasing amount of the Al-Zn borate ratio the tensile strength values at break decreased. In the Al-Zn borate doped LDPE F5-21T film sample, a decrease in the elongation at break was observed with the increasing Al-Zn borate values.

The direct effect of molecular weight is that the higher the Mn, the less zinc borate is required to achieve thermal deformation in the sample because it is easier to connect polymer chains already in a certain network structure with a few crosslinking points. A rapid decrease in the number of terminal vinyl groups in crosslinking for both linear and branched polyethylene also confirm.

Mass losses against temperature were investigated in thermogravimetric TG analyzes and it was observed that the heat resistance of the samples with the highest heat stability increased as the Al-Zn borate ratio increased.

In this study, the mechanical properties of the samples, in which Al-Zn borate was added to LDPE at different rates (0-10 wt.%), changed. The results indicated that the samples could be used in packaging, cable and wire applications for industrial purposes.

**Conflicts of Interest:** All authors declare no conflicts of interest.

**Ethical Approval:** Ethics Approval is not required for this study.

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