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The Flame Retardancy Performance of Aluminum Hypophosphite and its Synergistic Effect with Expandable Graphite in Carbon Fiber Reinforced Poly (Lactic Acid) Composites

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Abstract: The aim of this study is to investigate the effects of both aluminum hypophosphite (AHP) and its synergistic effect with expandable graphite (EG) on the thermal and flame retardant properties of carbon fiber reinforced polylactic acid (PLA) composites. The composites were produced by melt blending method by keeping the carbon fiber (CF) ratio (30 wt.%) constant. The flame retardancy effect of AHP was studied at three different concentrations (10, 15, 20 wt.%). The synergic effect studies were also carried out using mixture formulations of 7 wt.% AHP and 3 wt.% EG, 5 wt.% AHP and 5 wt.% EG. The characterization of the composites was performed using thermogravimetric analysis (TGA), mass loss cone (MLC), limiting oxygen index (LOI) and vertical burning tests (UL-94 V). The TGA results showed that only the addition of AHP decreased the thermal stability, while the addition of AHP and EG combination did not affect the thermal properties much. From the LOI and UL-94 V test results, it was determined that only the addition of AHP significantly increased the LOI values and the composites passed to UL-94 V0 rating. It was found that the LOI values increased with the addition of AHP and EG combination, but this increase was less compared to the composite containing only AHP (20 wt.%). However, these composites failed the UL-94 V test. The MLC test results showed that only the addition of AHP significantly reduced the pHRR values, while the combination of AHP and EG was close to these values. As a result, only the addition of AHP improved the thermal and flame retardant properties of carbon fiber reinforced PLA more than the combination of AHP and EG.

Keywords: Polylactic acid, Aluminum hypophosphite, Expandable graphite, Flame retardancy.

Introduction

Large quantities of synthetic polymers are produced worldwide and a significant amount of them introduced the ecosystem as industrial waste products (Nampoothiri et al, 2010). As well as polluting the ecosystem, these polymers consume petrochemical resources and produce large amounts of greenhouse gases (Wang et al, 2019). Therefore, it is important to develop biodegradable materials that can replace synthetic polymers to eliminate

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environmental problems and support sustainable development (Nampoothiri et al, 2010; Wang et al, 2019). Among the numerous biodegradable polymers, poly(lactic acide) (PLA) is the most researched and used material with the brightest development prospects (Nampoothiri et al, 2010; Rasal et al, 2010; Faraf et al, 2016). PLA is a linear aliphatic thermoplastic polyester produced from renewable resources such as starch and sugar, corn, potato, cane molasses, sugar-beet etc (Wang et al, 2019; Rasal et al, 2010; Zhu et al, 2016; Murariu & Dubois, 2016). It has a high level of biocompatibility, high stiffness and UV-stability (Wang et al, 2019). Due to its inherent properties, PLA occupies a key position in the bio-polymers market, which has led to an increased commercial interest in PLA and a large number of scientific studies on it (Rasal et al, 2010; Murariu & Dubois, 2016; Lee & Wang, 2006; Hsieh et al, 2016).

Although PLA has many advantages, it also has drawbacks such as brittleness and low temperature resistance, limiting its use in applications where high toughness and temperature resistance are required (Wang et al, 2019). These drawbacks of PLA can be manipulated by adding various additives such as some tough polymers and fibers. However, fibers are more effective in improving the mechanical properties of PLA (Wang et al, 2019; Avérous, 2008; Lin et al, 2014). Both natural and synthetic fibers can be used to improve the mechanical properties of PLA. In addition, synthetic fibers have higher strength than natural fibers and therefore improve the mechanical properties of PLA more effectively (Wang et al, 2019). Due to its high strength, modulus and outstanding thermal properties, carbon fiber is a very popular fiber in industry (Cheng et al, 2014).

As PLA and its composites are flammable, flame retardant treatments are required to expand their range of applications (Yang et al, 2015). The most effective substances used to improve the flame retardant performance of PLA are metal hydroxides, halogenated compounds, phosphorus and silicon-containing compounds (Gu et al, 2019). The eco-friendly and low-cost aluminum hypophosphite (AHP) is one of the phosphorus-containing flame retardants and is highly effective in PLA, PET, PBT and PA (Savas et al, 2020; Tang et al, 2012). Tang, G. et al. reported that a flame retardant PLA containing 20 wt.% AHP achieved the highest UL 94 rating of V-0 (Tang et al, 2012). Another study by Gu et al. showed that a high LOI value of 28.8 can be achieved with the addition of 20% AHP to PLA (Gu et al, 2019).

The expanded graphite (EG) is a material obtained by adding sulfuric acid or nitric acid between graphite layers. The addition of EG improves the mechanical, thermal, electrical and barrier properties of various polymers (Tang et al, 2013). It is also a flame retardant with intumescent and smoke suppressing properties. When EG is exposed to heat, it could expand to form large volumes of "worm-like" insulative char (Tang et al, 2013; Zhu et al, 2018). The EG is used in combination with phosphorous based flame retardants to increase the flame retardant efficiency as a synergistic agent (Tang et al, 2013; Zhu et al, 2018; Yang et al, 2016; Ji et al, 2020; Shih et al, 2004; Feng & Qian, 2014). Zhu et al. (2018) used EG and APP to prepare flame-retardant PLA composites. Results showed that PLA composites with 15 wt.% APP/EG (1:3) combination achieved an LOI of 36.5 and a V0 class in UL-94 testing. They emphasized that this composite has better properties compared to composites containing only EG and AHP (Zhu et al, 2011). A series of flame retardant PLA composites containing aluminum hypophosphite (AHP) and EG were investigated by Tang et al. They reported that an optimum result was achieved when 10 wt% each of AHP and EG were added to PLA. This combination resulted in a UL 94 V-0 rating with an LOI value of 34% (Tang et al, 2013).

Except for the studies mentioned above, there is no study that examines the effect of AHP and EG combination on the flame retardancy properties of carbon fiber reinforced PLA composites. This study aimed to examine the synergistic effect of AHP and EG on the flame retardant properties of carbon fiber reinforced PLA composites. Their flame retardancy and combustion properties were investigated by (limiting oxygen index) LOI, vertical burning test (UL-94) and mass loss cone (MLC) and their thermal stability was analyzed by thermogravimetric analysis (TGA).

Materials and Methods

Poly(lactic acid) (PLA) Ecolen HZ40P was provided by Hellenic Petroleum (Greece). It has a density and melt flow rate of 0.90 g/cm^3 (ASTM D792) and 12 g/10 min (2.16 kg, 230 °C, ASTM D1238), respectively. Carbon fiber (CF), trade name AC0101, was obtained from DOWAKSA (Yalova, Turkey). Aluminum hypophosphite (AHP) was supplied from Beijing Purkinje General Instrument Co. Ltd. (China). Expandable graphite (EG), trade name TEG 315, was purchased from Minelco Ltd. (Italy). The coefficient of expansion, bulk density and pH of EG are >220 mL/gm at 1000°C, 0.45-0.5 g/cm^3 and 5-7, respectively.

Production of PLA Composites

Prior to extrusion, PLA and all additives were dried at 80 °C for 12 hours. Extrusion was performed using a rotating twin screw extruder (Gulnar, Turkey) with a barrel temperature profile of 30-200-205-210-205-200 °C and a screw speed of 150 rpm. Mixing was done using the proportions in Table 1. After mixing, the composites were chopped into pellets. The pellets were used to shape standard size specimens for flammability tests using an injection molding machine (Xplore IM12, Netherlands) with a barrel temperature of 210 °C and mold temperature of 20 °C. The injection pressure was set at 8 bar. The flame retardancy of AHP and its synergistic effect with EG was investigated under 30 wt. % constant CF addition.

Characterization Method

The thermal properties of PLA and its composites were carried out by thermogravimetric analysis (Hitachi-High Tech STA-7300) at a heating rate of 10 °C/min from room temperature to 700 °C under nitrogen atmosphere. The LOI values were determined according to ASTM D2863 using a Limiting Oxygen Index Analyzer (FTT) on 130 × 6.5 × 3.2 mm^3 test bars. UL 94 V tests were performed according to ASTM D3801 on specimens with dimensions 130 × 13 × 3.2 mm^3. MLC testing was carried out on 100 × 100 × 3 mm3 specimens according to ISO 13927 standard using a Mass Loss Cone (FTT, UK). The specimens were characterized under a heat flux of 35 kW/m^2.

Results and Discussions

Thermal Decomposition Behavior

The decomposition behavior of PLA and its composites investigated with TGA analysis under nitrogen atmosphere. Plots of TGA and DTG are given in Figure 1. The related data are tabulated in Table 1. Pure PLA degrades in single step with maximum degradation rate at 366 °C and leaves 5.5 % residue at 700 °C.

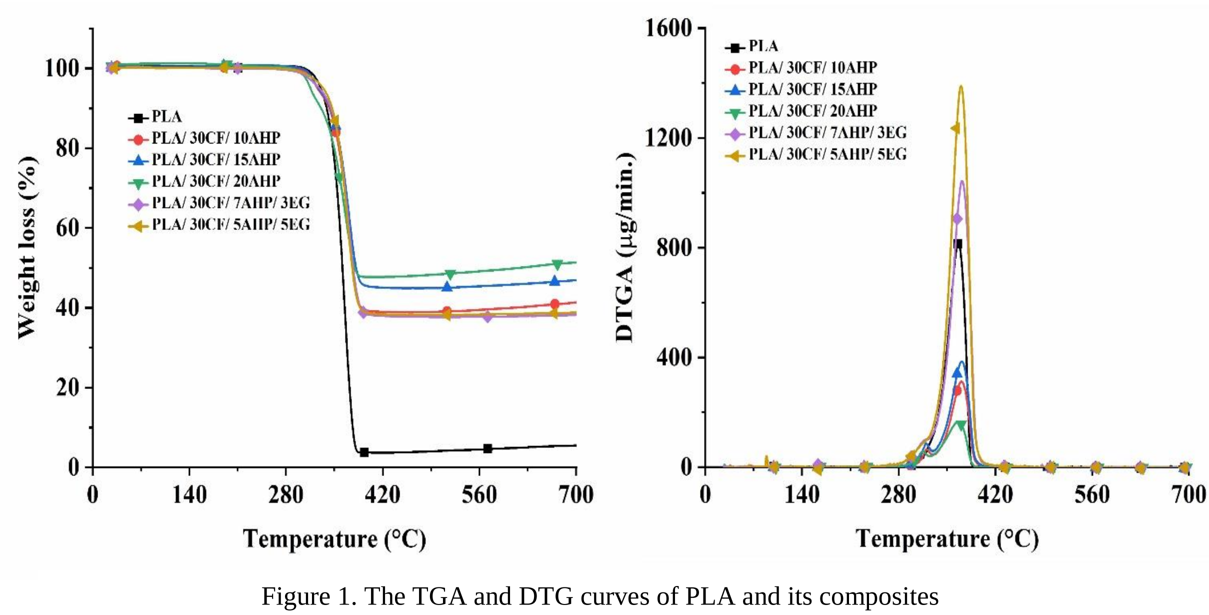

Figure 1. The TGA and DTG curves of PLA and its composites

The composites containing only AHP as a flame retardant degraded in single step. The initial degradation temperature ($T_{5\%}$) of these composites is lower compared to PLA. Similar results were observed in the study conducted by Doğan et al (Savas et al, 2020). The maximum degradation temperature (T_{max}) of the composites slightly increased with the addition of AHP. This increase is due to the fact that phosphate and pyrophosphate formed by the degradation of AHP acts as a promoting formation of residue in PLA (Wang et al, 2022). This is also supported by the increase in residue amounts shown in Table 1. The increase indicates that AHP react with PLA and more residues can be obtained. A similar trend has been observed in previous studies (Savas et al, 2020).

Table 1. TGA tests results of PLA and its composites

Sample	$T_{5\%}$ (°C)	T_{max} (°C)	Residue (%)
PLA	329	366	5.50
PLA/ 30CF/ 10AHP	322	370	41.3
PLA/ 30CF/ 15AHP	323	371	46.9
PLA/ 30CF/ 20AHP	320	370	51.2
PLA/ 30CF/ 3EG/ 7AHP	327	371	38.2
PLA/ 30CF/ 5EG/ 5AHP	330	369	38.8

The addition of EG in combination with AHP did not significantly change the $T_{5\%}$ value and slightly increased the T_{max} values of the composites, compared to PLA. At last, the char yield of the composites containing a combination of AHP and EG is high compared to pure PLA and low compared to the composites containing only AHP. That proves that there is an antagonistic effect between EG and AHP. Similar trend of results was observed in a study in which APP was used together with EG. The swelling of the EG to form a worm-like structure causes the interface between the polyphosphoric acid and its environment to widen, resulting in accelerated degradation and reduced residue yield (Meng et al, 2009).

Flammability Properties

Combustion resistance of PLA and its composites was determined by LOI and UL94 V tests. The results of LOI and UL-94 V tests are presented in Table 2. The pure PLA has LOI value of 21.3% and achieved V2 rating in UL-94 V test. The LOI values of the composites containing only AHP increased with increasing AHP concentration and reached V0 rating in UL-94 V test. Here, AHP increased the combustion resistance of the composites by forming an effective char layer. This char layer protected the composites against further combustion by preventing mass and heat transfer (Tang et al, 2012).

Table 2. LOI and UL-94 V tests results of PLA and its composites

Sample	LOI	UL-94 V
PLA	21.3	V2
PLA/ 30CF/ 10AHP	29.2	V0
PLA/ 30CF/ 15AHP	32.0	V0
PLA/ 30CF/ 20AHP	32.7	V0
PLA/ 30CF/ 3EG/ 7AHP	29.2	BC
PLA/ 30CF/ 5EG/ 5AHP	28.6	BC

The LOI values of the composites containing a combination of AHP and EG increased compared to pure PLA, but could not exceed the LOI values of the composites containing only AHP. It also failed the UL-94 V test and did not qualify for any test rating. This result indicates the existence of an antagonistic effect between EG and AHP.

Mass Loss Calorimeter Studies

Combustion behavior of PLA and its composites was evaluated by mass loss cone. Figure 2 shows the HRR curves of PLA and its composites and the relevant data are listed in Table 3, including time to ignition (TTI), peak heat release rate (pHRR), average heat release rate (avHRR), total heat evolution (THE) and residue amount. The pHRR of pure PLA reached 385 kW/m^2 after 95 s ignition. After adding only AHP, the pHRR values of the composites decrease significantly. The PLA/30CF/20AHP decreased to 123 kW/m^2, which was 68% lower than that of pure PLA. This reduction indicates that AHP is effective in the gas and condense phase by forming protective char layer. The pHRR values of the composites containing the combination of AHP and EG were close to the composites containing only AHP and changed slightly. This shows that both AHP and the combination of AHP and EG reduce the fire risk to a similar extent. The THE values of the composites containing only AHP decreased compared to pure PLA. This reduction is due to the decrease in the amount of combustible material (PLA) with increasing char content and the inability to complete pyrolysis due to the barrier effect of char (Has et al, 2022). The value of THE of the composites containing the combination of AHP and EG increased compared to the composites containing only AHP. From the photographs given in Figure 3, it is thought that this is due to the char containing more pores compared to the composites containing only AHP.

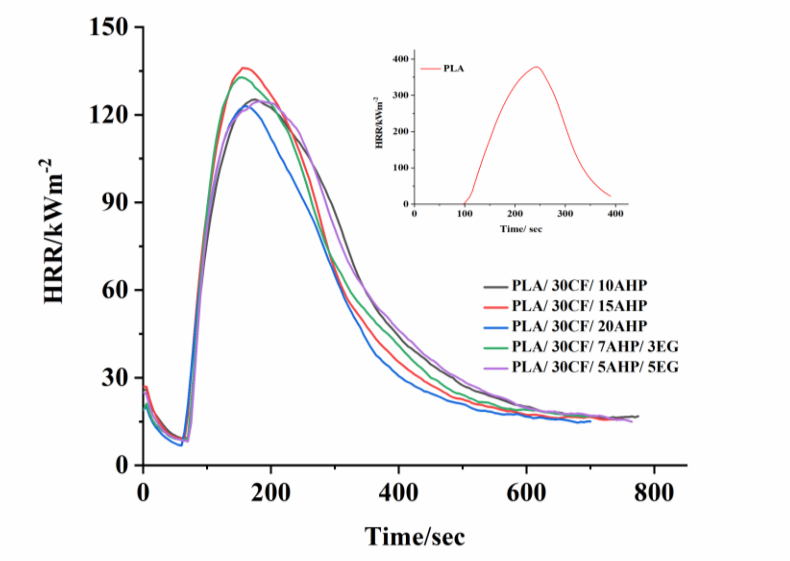

Figure 2. The HRR curves of PLA and its composites

The addition of both AHP and the combination of AHP and EG causes decreases in TTI value. It is thought that this decrease is due to the decrease in the thermal stability of the composites as described in the TGA section. Flammable gas formation starts early in composites with reduced thermal stability. This causes the composites to ignite earlier. In addition, black colored carbon fibers absorb too much heat, causing early degradation of the polymer (Aydogan et al, 2023).

Table 3. MLC results of PLA and its composites

Sample	**TTI (sec.)**	**pHRR (kW/m^2)**	**THE (MJ/m^2)**	**Char Residue (%)**
PLA	95	385	68	2.8
PLA/ 30CF/ 10AHP	70	125	26	38.0
PLA/ 30CF /15AHP	65	136	32	39.5
PLA/ 30CF/ 20AHP	59	123	30	48.8
PLA/ 30CF/ 7AHP/ 3EG	68	132	34	38.1
PLA/ 30CF/ 5AHP/ 5EG	71	124	36	38.9

The addition of flame retardants significantly increases the amount of char residue compared to PLA. The highest char residue content was obtained in PLA/30CF/20AHP. The combination of AHP and EG did not reach this value and produced less char residue. This is a result of the antagonistic effect.

The digital photograph of char residues is shown in Figure 3. It can be seen from the images that; PLA has no char residue after MLC tests. The char layer for the composites containing only AHP (PLA/30CF/15AHP) is fine and rigid, but it is so brittle that several cracks and holes are formed during combustion. These holes are caused by the impact of PH_3 gas generated during the degradation of AHP (Tang et al, 2013). Composite containing a combination of AHP and EG has more holes. It is thought that these holes could weaken the barrier effect of the char layer and led to bad flame retardant properties. These results were in agreement with LOI and UL-94 V performances.

Conclusion

This study investigated the effects of aluminum hypophosphite (AHP) and its combination with expandable graphite (EG) on the thermal and flame retardant properties of carbon fiber reinforced polylactic acid (PLA) composites. Through comprehensive testing including thermogravimetric analysis (TGA), mass loss calorimeter (MLC), limiting oxygen index (LOI), and vertical burning tests (UL-94 V), several key findings emerged:

- Firstly, for the composites containing only AHP, the LOI value increased with increasing AHP concentration (10, 15, 20 wt.%), and all composites achieved a UL-94 tested V0 rating. All this represents a significant improvement in flame retardancy.

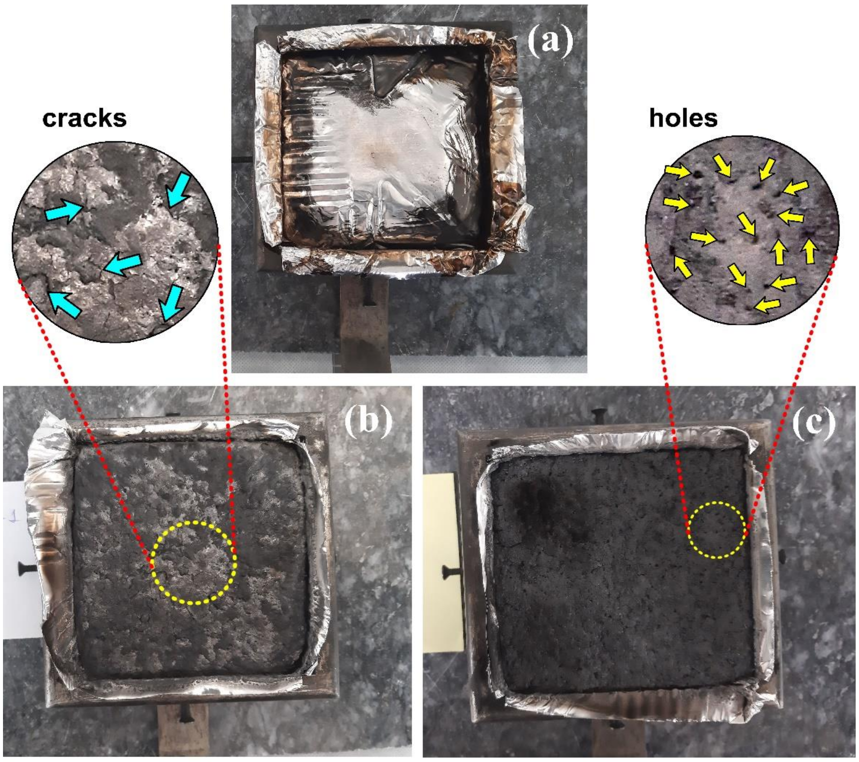

Figure 3. Digital photography of (a) PLA, (b) PLA/30CF/15 AHP, (c) PLA/30CF/7AHP/3EG

- Secondly, the synergistic effect of AHP and EG, tested at formulations of 7 wt.% AHP and 3 wt.% EG, and 5 wt.% AHP and 5 wt.% EG, demonstrated mixed results. Although the combinations of AHP and EG improved LOI compared to pure PLA, they failed the UL-94 test. However, the LOI values of the combinations OF AHP and EG could not reach the LOI values of the composites containing only AHP.
- Thirdly, as evaluated by TGA, it is indicated that AHP alone slightly decreased thermal stability, whereas the addition of EG alongside AHP did not significantly affect this property.
- Moreover, MLC results showed that AHP effectively reduced the peak heat release rate (pHRR), crucial for fire safety, while the combination with EG performed similarly to composites with AHP alone.

Overall, the findings underscore that AHP is more effective in enhancing the thermal and flame retardant properties of carbon fiber reinforced PLA compared to its combination with EG. This research provides valuable insights for optimizing the formulation of flame retardant composites, contributing to advancements in materials science aimed at enhancing fire safety in various applications of carbon fiber reinforced PLA composites.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Acknowledgements or Notes

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