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Sustainable Fire-Resistant Materials: Recycled Polyethylene Composites with Non-Halogenated Intumescent Flame Retardants for Construction Applications

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Abstract: This study explores the development of sustainable fire-resistant composites using a blend of recycled linear low-density polyethylene (rLLDPE) and low-density polyethylene (rLDPE) for construction applications. The incorporation of non-halogenated intumescent flame retardants (IFRs), specifically ammonium polyphosphate (APP) and melamine polyphosphate (MPP), was shown to enhance the flame retardance, thermal stability, and mechanical performance of these recycled polymer blends. IFRs were introduced at 5 wt.% and 10 wt.% concentrations, and their effects were evaluated using limiting oxygen index (LOI) testing and thermogravimetric analysis (TGA). Results showed that 10 wt.% APP and a combination of 5 wt.% APP with 5 wt.% MPP increased LOI values from 18.5% (neat polymer blend) to 21.2% and 22.4%, respectively, demonstrating improved fire resistance. Enhanced char formation, facilitated by IFRs, contributes to superior thermal stability and fire protection. TGA results confirmed higher char yields, with the rLLDPE/rLDPE/MPP5/APP5 composition exhibiting the highest residue (3.00%), indicating a synergistic effect between APP and MPP. Rheological and mechanical analysis showed that APP had more impact on viscoelastic behavior, while the combination of IFRs provided balanced mechanical properties despite a slight reduction in tensile strength. This research highlights the potential of recycled polyethylene composites in promoting circular economy principles by developing sustainable, fire-resistant materials for the construction industry, reducing plastic waste, and enhancing the safety of recycled polymer-based applications.

Keywords: recycled LLDPE/LDPE; flame retardants; ammonium polyphosphate; melamine polyphosphate; circular economy

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1. Introduction

Thermoplastic polymers, ubiquitous in daily life, are used in packaging, furniture, vehicles, medical devices, and clothing. These materials are lightweight, strong, flexible, durable, and resistant to chemicals and corrosion. Moreover, thermoplastics can be easily molded into various shapes, making them ideal for diverse applications across multiple industries [1].

However, the increasing accumulation of plastic waste has become a pressing environmental issue. Rapid urbanization and industrialization have significantly contributed to plastic waste generation, posing challenges in waste management and sustainability efforts. Currently, plastic waste is primarily managed through landfilling, incineration, or recycling.

Of these methods, mechanical recycling of post-consumer plastic waste is considered the most environmentally friendly approach. However, despite its potential, only 9% of post-consumer plastic waste is effectively recycled worldwide. This low recycling rate highlights significant challenges in waste collection, sorting, and processing. Consequently, the heavy reliance on landfilling and incineration for managing post-consumer plastic waste leads to substantial societal and environmental impacts, including pollution and increased carbon emissions [1].

Recent research and industrial innovations have sought to address plastic waste issues by promoting sustainable recycling methods and circular economy principles. The circular economy emphasizes the continuous reuse of materials to minimize waste generation and resource depletion [2]. Effective recycling strategies, including chemical and mechanical recycling, have been developed to facilitate material recovery. Chemical recycling breaks down polymers into their original building blocks or purified forms, while mechanical recycling, a more cost-efficient process, in some cases, involves mixing used polymers with virgin materials. Integrating these methods, along with sustainable product designs and responsible material handling, is crucial for advancing the plastics industry toward a closed-loop system [2].

Recycled polyethylene (PE), particularly low-density polyethylene (LDPE) and linear low-density polyethylene (LLDPE), is widely used in film applications and post-consumer recycled (PCR) materials, which are commercially available in pellet form [3–5]. However, PCR materials often contain different polyolefin grades, additives, contaminants, and color variations, which can affect their processability, performance, and mechanical properties when reused in new applications [6]. Moreover, despite its many advantages, polyethylene has inherent limitations, such as low melting points and high flammability, which hinder its usability in high-risk applications like construction [7]. The demand for fire-resistant materials in construction has led to the increasing need for flame-retardant additives. Traditional halogen-based flame retardants, although effective, pose environmental and health risks due to toxic emissions during combustion [8]. Consequently, non-halogenated intumescent flame retardants (IFRs), such as ammonium polyphosphate (APP) and melamine polyphosphate (MPP), have emerged as environmentally safer alternatives. These IFRs enhance fire resistance by forming protective char layers that act as thermal barriers, reducing heat transfer and slowing down material combustion. They are widely used in sustainable material development due to their low toxicity and reduced environmental impact [9,10].

Nitrogen and phosphorus compounds, particularly polyphosphates, are widely used halogen-free flame retardants. Ammonium and melamine polyphosphates play a key role in fire-resistant additive materials that reduce the combustion of wood, fabrics, and plastics. Among these, Ammonium Polyphosphate (APP) is gaining industrial attention due to its low cost, excellent processability, and lower required loadings, making it a more efficient choice than other flame retardants. Its most significant advantage is that it is halogen-free, which means that it does not generate excessive smoke and toxic fire emissions that are harmful to health during combustion, making it more environmentally friendly compared to halogen-based alternatives [11].

Ammonium polyphosphate (APP) acts as a flame retardant in the condensed polymer phase through intumescence—this is a process where the material swells upon exposure to heat, creating a porous carbon foam barrier that blocks heat, air, and combustion products [12]. When APP-containing polymers are exposed to fire, APP decomposes into polyphosphoric acid and ammonia. The polyphosphoric acid reacts with hydroxyl groups or synergistic additives like hydroxyethylcyanuarates or urea–formaldehyde resins, forming unstable phosphate esters. These esters undergo dehydration, creating a charred carbon foam layer on the polymer surface, protecting the material from further heat exposure. This

foam layer is complemented by a viscous molten layer or surface glass, further shielding the inner part from heat and oxygen [12].

Intumescent flame retardants (IFR) typically comprise three main components: an acid source (inorganic acids or salts) to dehydrate the carbonizing agent, a carbonizing agent (carbohydrates) that chars upon dehydration, and a blowing agent that releases gas to form a swollen, multicellular layer [13]. Together, these components provide robust flame resistance by forming a protective barrier [14–16].

Melamine phosphates (MPP), despite having less popularity, are gaining importance. Condensed forms of melamine phosphates, such as meta-, pyro-, poly-, and orthophosphates, contain both phosphorus and nitrogen. These compounds pass into the gaseous state at relatively high temperatures, making them suitable for polymers processed at high temperatures [17]. Melamine polymetaphosphate is especially useful for thermoplastic applications requiring high thermal resistance, as it can withstand temperatures up to 350 °C without substantial mass loss. Melamine phosphate, consisting of 37.5% nitrogen and 13.8% phosphorus, effectively reduces material combustibility [17]. The condensation of phosphate salts into polyphosphates during heating makes MPP more thermally resistant and, thus, better suited as an additive in thermoplastics [17].

Previous studies show that much work has been performed and is still ongoing to study the effects of flame retardants on thermoplastics. Lim et al. [12] reviewed the application of ammonium polyphosphate as an intumescent flame retardant in thermoplastic composites [11], and Ghomi et al. [18] reviewed other flame retardants for polyethylene. Despite extensive research on IFRs in virgin thermoplastics, limited studies have examined their effects on recycled thermoplastics, particularly rLLDPE/rLDPE blends. Given the increasing emphasis on sustainability and fire safety in the construction industry, it is essential to explore the potential of IFR-modified recycled polyethylene composites.

Therefore, in this research, we studied the impact of non-halogenated intumescent flame retardants, specifically APP and MPP, on the thermal, rheological, morphological, flame retardance, and mechanical properties of recycled LLDPE/LDPE polymer blends. By developing flame-resistant rLLDPE/rLDPE composites with sustainable additives, this research contributes to reducing plastic waste, promoting circular economy principles, and advancing the use of environmentally friendly materials in the construction sector. The findings will provide valuable insights into optimizing recycled polymer formulations for fire-safe applications across various industries.

2. Results and Discussion

2.1. Morphology of rLLDPE/rLDPE-Based FR Composites

The morphology of the rLLDPE/rLDPE blend and its composites with flame retardants (FR) were examined by SEM at low and high magnifications, as shown in Figure 1. At higher magnification, the polymer blend exhibited a homogeneous morphology with some indications of plastic deformation. This plastic deformation may be attributed to the low glass transition temperatures of both polyethylenes (below −100 °C) [19] compared to the cryofracture process temperature of −80 °C. The time the sample spent in liquid nitrogen (10 min) or the delay during extraction and fracture may allow for slight warming, leading to plastic deformation at the microscopic level, which only became visible at higher magnification.

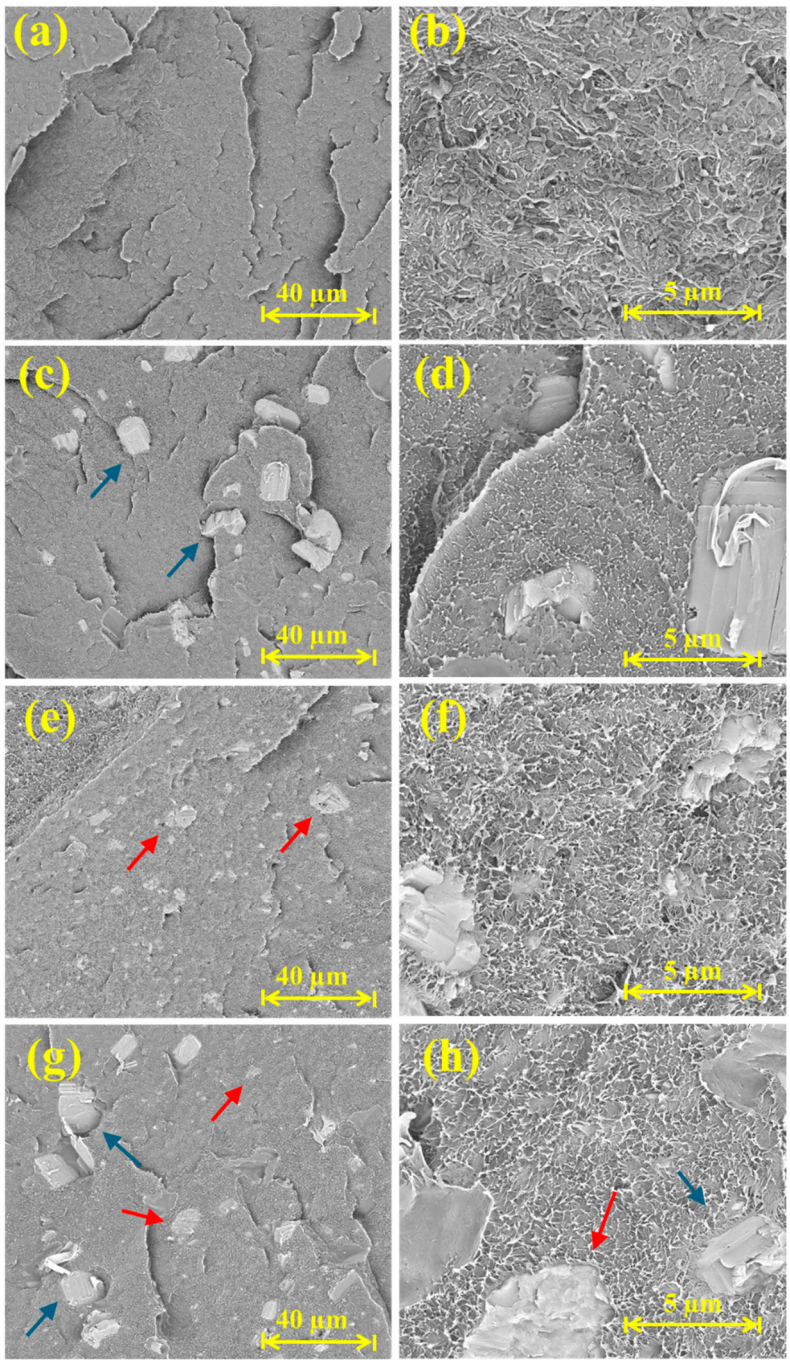


Figure 1. SEM images for different compositions with 2 magnifications: (**a**,**b**) Neat rLLDPE/rLDPE, (**c**,**d**) rLLDPE/rLDPE/APP10, (**e**,**f**) rLLDPE/rLDPE/MPP10, and (**g**,**h**) rLLDPE/rLDPE/MPP5/APP5.

For the composites containing 10 wt.% of flame retardants (FR) (Figure 1c–h), the APP particles are indicated by the blue arrows with smaller arrowheads and were observed as sharp-edged, smooth-surfaced, micro-sized particles, similar to what was observed by Shao et al. [20], whereas MPP, indicated by the red arrows with larger arrowheads, appeared as slightly smaller particles with more irregular surfaces, showing a broader distribution throughout the polymer blend matrix. These irregular surfaces observed for MPP may be agglomerates of smaller particles, which is typical for commercial MPP, as seen by Zhou et al. [21].

Neither APP nor MPP melted or decomposed during processing due to the strong ionic interactions of their respective inorganic polyphosphates, namely, ammonium for

APP and melamine for MPP [22]. Additionally, the encapsulation within the polyethylene blend matrix further prevented their decomposition. As a result, both flame retardants behaved as solid particles, exhibiting a morphology akin to that of inorganic fillers like calcium carbonate ($CaCO_3$) in polymer blends. For the rLLDPE/rLDPE/MPP5/APP5 composition, which contained 5 wt.% of each FR, both flame retardants were present and clearly seen. APP stood out due to its larger particle size and flat surface morphology at higher magnifications. In certain regions, APP and MPP were observed in close proximity, indicating a good dispersion of the flame retardants within this composition (Figure 1g,h). It can be concluded that there is a good compatibility between APP/PE and MPP/PE, as seen in the SEM images, where less agglomeration of the particles was observed.

The SAOS rheological behavior provides further insight into the interactions between the FR additives and the polyethylene blend matrix [23,24] (Figure 2). The rLLDPE/rLDPE blend exhibited a highly elastic, solid-like behavior ($G' > G''$ and with a shouldering in G'') in the terminal zone, which is uncommon for polymer blends without fillers. This behavior can be explained by the specific grade of the material used, which might contain other processing additives and has a low melt flow index (MFI of 1.0 g/10 min (190 °C/2.16 kg)), implying high viscosity and was designed for extrusion applications. Polymers for extrusion purposes generally have high molecular weights so that they maintain their inherent properties despite chain breakage during the extrusion process performed at high temperatures and high shear rates [25].

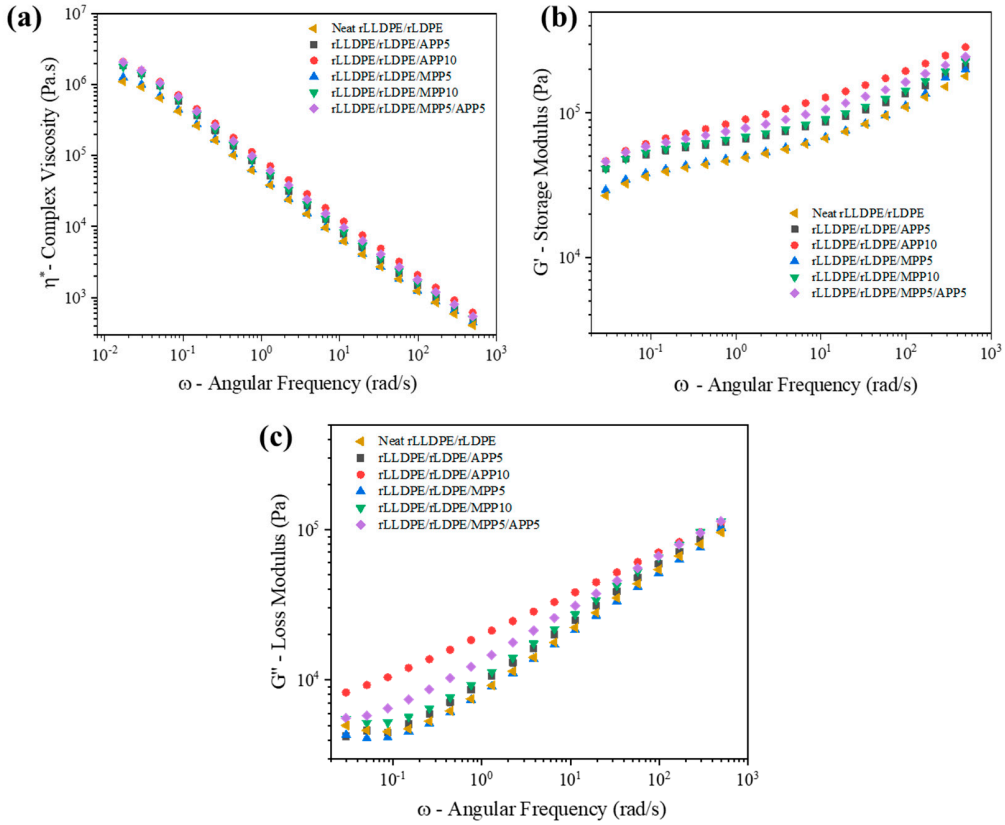


Figure 2. SAOS rheological behavior: (**a**) η*—complex viscosity, (**b**) G′—storage modulus, and (**c**) G″—loss modulus.

For the composites, the SAOS rheological behavior closely resembled that of the rLLDPE/rLDPE blend (Figure 2), reinforcing our conclusion that adding low surface area, micro-sized particles (such as these FRs) does not significantly alter the SAOS rheological behavior at low filler contents [24] unlike what happens with the addition of

nanoparticles [26]. However, the slight changes observed indicate that APP had a more pronounced effect on rheological behavior, increasing G′ more than MPP and substantially increasing G″ in the terminal zone. That change in behavior led to higher complex viscosity and shear modulus for the rLLDPE/rLDPE/APP10 composite (Figure 2). As expected, the rLLDPE/rLDPE/MPP5/APP5 composition exhibited intermediate behavior between rLLDPE/rLDPE/APP10 and rLLDPE/rLDPE/MPP10, reflecting a balance in the rheological effects of the different flame retardants.

The steady-state rheological behavior obtained via capillary rheometry provides valuable insight into the effect of adding flame retardant (FR) to these composites. The increase in viscosity due to the addition of 10 wt.% APP was evident at lower shear rates, as was the intermediate behavior of the rLLDPE/rLDPE/MPP5/APP5 composition (Figure 3). However, all these thermoplastic-based materials exhibited pronounced shear-thinning behavior at higher shear rates (10^2 to 10^4 s^{-1}), as measured by capillary rheometry. In this shear rate range, the viscosity values of the composites converged and matched the behavior of the rLLDPE/rLDPE polymer blend.

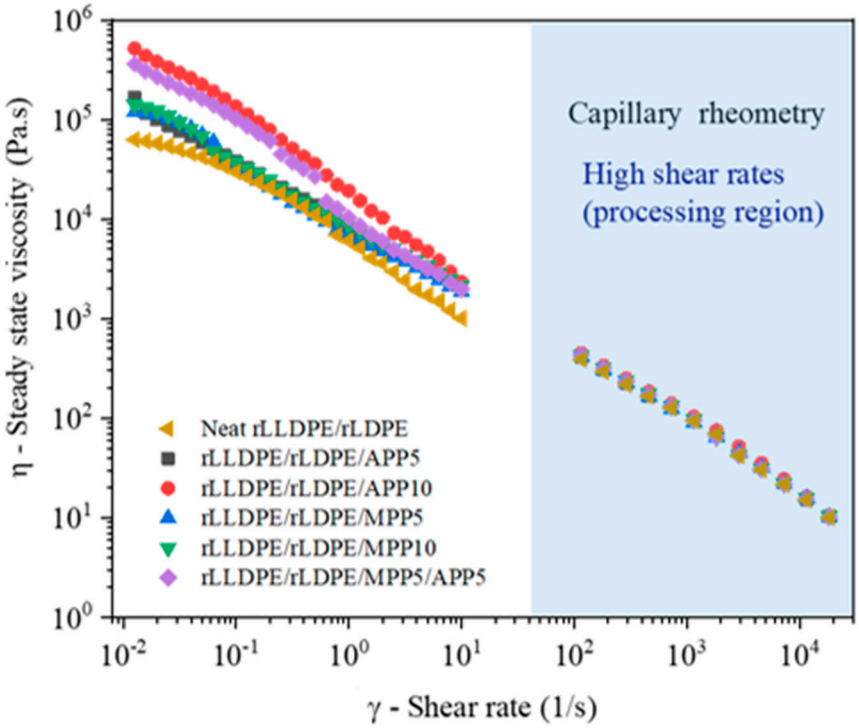


Figure 3. Steady shear viscosity for the r-LLDPE/r-LDPE polymer blend and composites.

According to Table 1, small but insignificant changes were observed in the mathematical fit of the data to the power-law model described in Equation (1). This lack of variation suggests that the addition of the FRs did not change the processability of the composites compared to the rLLDPE/rLDPE polymer blend. This is advantageous for most industrial processes like injection molding and extrusion, which typically operate at elevated shear rates (10^2 to 10^5 s^{-1}), and thus, these materials will maintain their processability under standard operating conditions.

Table 1. Power law fit of capillary rheometry results of r-LLDPE/r-LDPE polymer blend and composites.

Samples	Fit Intercept	Fit Slope	Consistency Index (m) 10^4 (Pa·s^n)	Pseudoplasticity Index (*n*)	R^2
Neat rLLDPE/rLDPE	4.13 ± 0.05	−0.72 ± 0.02	13.6 ± 0.7	0.28 ± 0.02	0.994
rLLDPE/rLDPE/APP5	4.13 ± 0.05	−0.72 ± 0.01	13.6 ± 0.6	0.28 ± 0.01	0.996
rLLDPE/rLDPE/APP10	4.21 ± 0.06	−0.73 ± 0.02	16.4 ± 0.9	0.27 ± 0.02	0.994
rLLDPE/rLDPE/MPP5	4.15 ± 0.03	−0.72 ± 0.01	14.1 ± 0.5	0.28 ± 0.01	0.998
rLLDPE/rLDPE/MPP10	4.20 ± 0.04	−0.74 ± 0.01	15.7 ± 0.6	0.26 ± 0.01	0.997
rLLDPE/rLDPE/MPP5/APP5	4.20 ± 0.04	−0.74 ± 0.01	15.8 ± 0.6	0.26 ± 0.01	0.997

Differential Scanning Calorimetry (DSC) was used to characterize the thermal behavior of the materials, and the results are summarized in Figure 4 and Table 2. Polyethylenes have well-documented thermal properties [27,28], and the expected melting temperature (T_m) values were confirmed in our blends at around 120–130 °C, with a high degree of crystallinity (~60%). Comparing the first and second heating cycles, both the crystallinity and melting temperatures remained consistent, reflecting the rapid crystallization behavior typical of both polyethylenes.

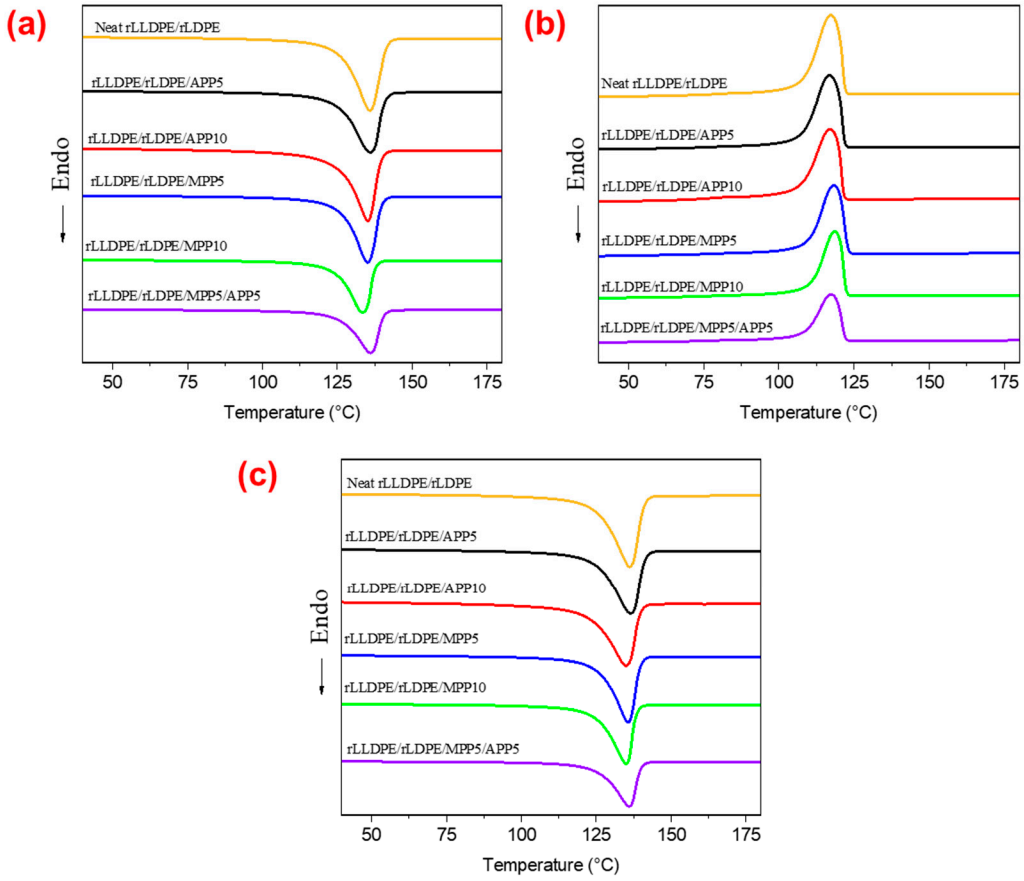


Figure 4. DSC thermograms: (**a**) first heating, (**b**) cooling, and (**c**) second heating of neat rLLDPE/rLDPE blend and rLLDPE/rLDPE containing APP and MPP.

Table 2. Thermal properties obtained by DSC for the neat rLLDPE/rLDPE blend and rLLDPE/rLDPE containing APP and MPP.

Samples	T_m (°C)	ΔH_m (J/g)	X_c (%)	Tc (°C)	ΔH_c (J/g)	T_m (°C)	ΔH_m (J/g)	X_c (%)
Neat rLLDPE/rLDPE	135.83 ± 0.05	184.2 ± 0.2	63	117.24 ± 0.04	188.0 ± 0.2	136.14 ± 0.03	186.7 ± 0.3	64
rLLDPE/rLDPE/APP5	135.73 ± 0.58	179.4 ± 0.2	64	116.82 ± 0.07	195.2 ± 0.2	136.57 ± 0.09	186.8 ± 0.2	67
rLLDPE/rLDPE/APP10	135.26 ± 0.15	175.4 ± 0.2	67	117.06 ± 0.02	177.3 ± 0.2	134.85 ± 0.01	167.3 ± 0.2	63
rLLDPE/rLDPE/MPP5	133.39 ± 0.33	169.2 ± 0.2	61	118.55 ± 0.08	186.3 ± 0.2	134.81 ± 0.15	180.5 ± 0.2	65
rLLDPE/rLDPE/MPP10	135.29 ± 0.21	182.3 ± 0.2	69	118.41 ± 0.13	182.5 ± 0.2	135.62 ± 0.11	177.7 ± 0.2	67
rLLDPE/rLDPE/ MPP5/APP5	136.27 ± 0.18	157.3 ± 0.2	60	117.30 ± 0.28	153.4 ± 0.2	136.04 ± 0.04	154.7 ± 0.3	59

It is important to note that miscibility in polymer blends is complex and influenced by various factors, including the specific properties of the polymer constituents and the blend ratio. As discussed by Delgadillo-Velázquez et al. [28], who conducted extensive thermal and rheological studies on LDPE/LLDPE blends, they observed single melting and crystallization peaks for blends with lower LDPE content. However, these peaks alone are insufficient to definitively prove miscibility. Their findings indicated that immiscibility became evident when the LDPE content exceeded 20%, as shown by the appearance of a third melting peak.

For the composites, the addition of FRs (both APP and MPP) at 5 and 10 wt.% did not significantly alter the thermal behavior, following the trend observed in the rheological analysis. These findings indicate that the flame retardants did not negatively affect the thermal properties of the polymer blend. This is essential since their role is to enhance flame retardancy without compromising other key material properties.

2.2. FTIR Analysis

The IR spectra of neat r-LLDPE/r-LDPE polymer blends and the composites were studied in the wavenumber ranges of 4000–400 cm^{-1}. As shown in Figure 5, the characteristic absorption peaks for stretching and bending vibrations of the C–H bonds present in neat rLLDPE/rLDPE polymer blends at wavenumber 2912 cm^{-1} (C–H bond, antisymmetric stretching) and 2852 cm^{-1} (C–H bond, symmetric stretching) and 1464 cm^{-1} (C–H bond, deformation), indicate the presence of alkyl groups as seen in the results of Czarnecka-Komorowska et al. [29]. The additional peaks at 1251, 1055, 908, 979, 480, 440 cm^{-1} for samples rLLDPE/rLDPE/APP5 and rLLDPE/rLDPE/APP10 show the presence of ammonium polyphosphate group, while the additional peaks at 1678, 1270, 1018, 881, 781, 591, 502, 472, 425 cm^{-1} for samples rLLDPE/rLDPE/MPP5 and rLLDPE/rLDPE/MPP10 show the presence of melamine polyphosphate. Similar peaks were observed in the APP and MPP by Amariei et al. [30], and these wavenumbers were assigned to LDPE: CH_2 stretch (ν) bands at 2915 and 2850 cm^{-1}, –CH_2 bending (δ) bands at 1470, 1465, 730, and 720 cm^{-1} [30]. Wavenumbers assigned to APP and MPP include 1245 cm^{-1} (νP = O of PO_4^{3-}), 1060 cm^{-1} (νP–O symmetric), 1020 cm^{-1} (symmetric νPO_2 and νPO_3), 885 cm^{-1} (νP–O asymmetric), and 800 cm^{-1} (νP–O–P) [31,32]. The IR spectra show that the samples are representations of the blending of r-LLDPE/r-LDPE polymer blends and the non-halogenated flame retardants of ammonium polyphosphate and melamine polyphosphate.

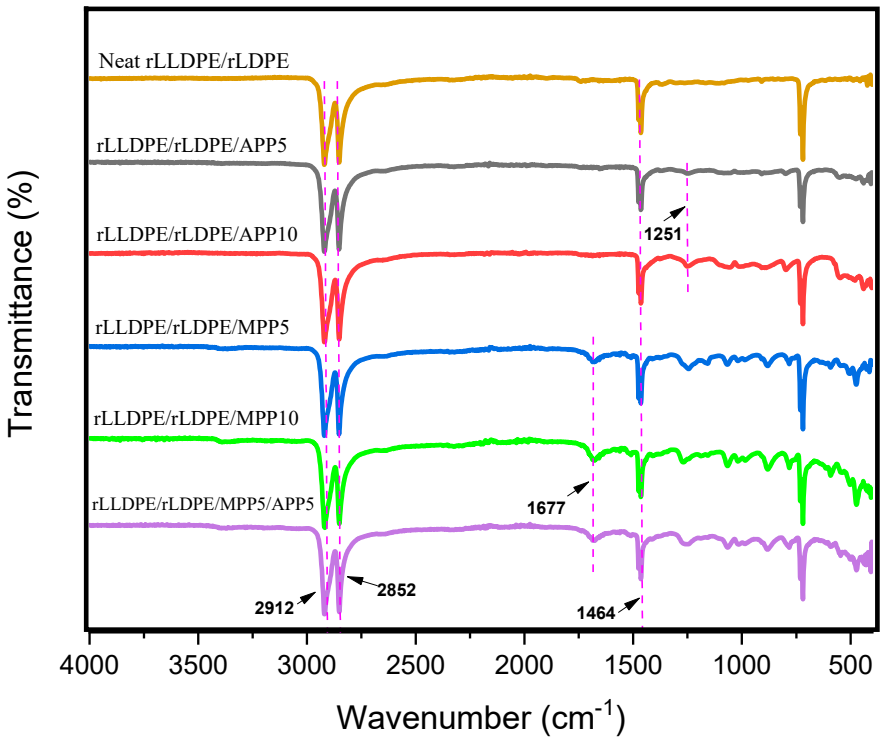


Figure 5. FTIR spectra of neat r-LLDPE/r-LDPE polymer blends and the composites.

2.3. TGA of rLLDPE/rLDPE Composites

TGA was used to investigate the thermal stability and char residue of the neat r-LLDPE/r-LDPE blend and its composites. The char residue and decomposition temperature of polymeric materials are important parameters in determining flame retardance properties because they provide information on how the material behaves when exposed to high temperatures (up to 900 °C). Figure 6 shows the TGA and DTG curves of the neat r-LLDPE/r-LDPE blend and its composites, and the TGA results are listed in Table 3.

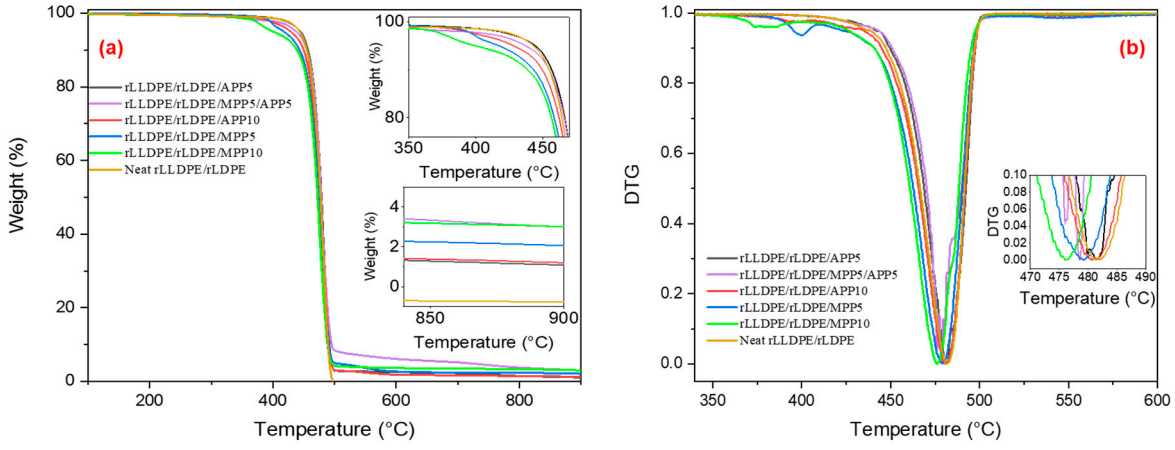


Figure 6. TGA profiles of (**a**) neat rLLDPE/rLDPE blend and rLLDPE/rLDPE containing APP and MPP; (**b**) DTG curves of (**a**) neat rLLDPE/rLDPE blend and rLLDPE/rLDPE containing APP and MPP. The concentration of APP and MPP varies between 5 and 10 wt.%, respectively.

Table 3. TGA and LOI results of neat rLLDPE/rLDPE blend and rLLDPE/rLDPE containing APP and MPP.

Samples	T_{onset} (°C)	T_{max} (°C)	Char Residue at 900 °C (%)	LOI (%)
Neat rLLDPE/rLDPE	451.06 ± 0.32	505.35 ± 0.11	0.75 ± 0.02	18.5 ± 0.5
rLLDPE/rLDPE/APP5	457.20 ± 0.21	449.18 ± 0.32	1.08 ± 0.01	19.3 ± 0.3
rLLDPE/rLDPE/APP10	456.44 ± 0.14	500.42 ± 0.21	2.99 ± 0.01	21.2 ± 0.7
rLLDPE/rLDPE/MPP5	453.23 ± 0.42	503.46 ± 0.14	1.20 ± 0.03	19.4 ± 0.2
rLLDPE/rLDPE/MPP10	458.59 ± 0.17	502.11 ± 0.02	2.05 ± 0.02	20.3 ± 0.6
rLLDPE/rLDPE/MPP5/APP5	459.73 ± 0.13	506.49 ± 0.16	3.00 ± 0.05	22.4 ± 0.4

The intumescent flame retardants did not significantly increase the thermal stability of the PE, which can be explained by the lower thermal stability of APP and MPP. Furthermore, it can be observed that the neat polymer starts to decompose at 451.06 °C to the maximum temperature of decomposition of 505.35 °C with no formation of char residue (Table 3). On the other hand, the rLLDPE/rLDPE with intumescent flame retardants demonstrated a lower initial temperature of decomposition (Figure 6b), which is associated with the decomposition of intumescent flame retardants (APP and MPP) and cross-linking reactions, producing char that can protect the rPE matrix from degradation and heat [33]. These small peaks associated with the decomposition of APP and MPP are easily observed in DTG.

When the temperature is increased, the rLLDPE/rLDPE blends containing APP and MPP degrade further and are more cross-linked, and char is produced. The char yield of the samples containing intumescent flame retardants increased with a high amount of IFR, meaning that more IFR in the polymer can meaningfully enhance the charring capability of the composites and, thus, improve flame retardancy. The highest char residue (3.00%) was observed for rLLDPE/rLDPE/MPP5/APP5 composite at 900 °C (Table 3), demonstrating the synergistic effect of combining APP and MPP. This result shows that during the thermal decomposition of the rLLDPE/rLDPE/MPP5/APP5 composite, the compatibility between APP and MPP facilitated the production of a rigid and compact char capable of decreasing the level of degradation of the polymer matrix.

2.4. Flammability Study of rLLDPE/rLDPE Composites

The flame retardancy of these polymeric materials was evaluated using the limiting oxygen index (LOI). LOI is a simple technique that gives rapid evidence of the minimum amount of oxygen needed to sustain the combustion of materials at ambient temperature. This means that a high LOI correlates to lower flammability and, consequently, better flame retardancy of polymeric materials [34]. As reported in the literature, the intumescent flame retardants added to the polymer play a crucial role in flame retardance, as these IFRs swell and expand the polymer, promoting char formation, which prevents heat transmission and, thereby, reduces the spread of the flame [34]. Table 3 lists the LOI values of the neat rLLDPE/rLDPE blend and its composites. From Table 3, we observe that intumescent flame retardants (APP and MPP) increase the LOI values of the composites. For example, the neat polymer presented a LOI value of 18.5%, which means that the neat polymer is extremely flammable in ambient air. During the LOI test of neat polymer, it was observed that it was easily ignited, and a high amount of melt and flame was observed when compared to the samples with APP and MPP.

A low amount of individual additive, e.g., 5 wt.% of MPP or 5 wt.% APP, in the polymer, had limited influence on the flammability of the rLLDPE/rLDPE blend, as the rLLDPE/rLDPE/APP5 and rLLDPE/rLDPE/MPP5 samples had LOI values of 19.3% and 19.4%, respectively. However, at a high loading of APP and MPP (10 wt.%), the LOI values

increased to 21.2% for rLLDPE/rLDPE/APP10 and 20.3% for rLLDPE/rLDPE/MPP10 system. When MPP and APP were added at a loading of 5 wt.% each, a synergistic effect was observed, and the LOI value of polymer composites increased to 22.4%.

The high LOI values of the composite are explained by the high char residue of these composites, as seen in TGA. Another reason may be related to the good dispersion of APP and MPP in PE, as seen in the SEM images, where less agglomeration of the particles was observed. The LOI values of the samples obtained in this study are satisfactory since the amount of IFR used was lower (5 to 10 wt.%) compared with LOI values (18 to 22%) reported in the literature by Zaghloul et al. [17], who evaluated the effect of MPP at high concentrations (10 to 30 wt.%) in LLDPE composites. In their study, the highest LOI value (22%) was obtained for LLDPE composite only at the highest concentration of 30 wt.% MPP.

Outstanding char layer formation on the surface of polymer material for rLLDPE/rLDPE/APP10 and rLLDPE/rLDPE/APP5/MPP5 inhibits the production of droplets and smoke, thereby blocking the propagation of flame during the burning process. Based on Schinazi et al. [34], the LOI of 21% is a reasonable LOI value since the amount of oxygen in the atmospheric air is right below 21 vol%. Consequently, the materials with higher LOI values ($\geq$21%) are considered self-extinguishing; i.e., they do not burn under ambient temperature conditions without external energy. On the other hand, materials with low LOI levels (<21%) are considered flammable. In this study, the rLLDPE/rLDPE/APP10 and rLLDPE/rLDPE/APP5/MPP5 systems meet the requirements to be considered self-extinguishing [35]. It is important to note that although these materials demonstrate fire resistance at ambient scenarios, this is not enough to prevent flame in realistic fire conditions since, in a fire, the materials are often exposed to higher temperatures than ambient temperature.

Furthermore, in realistic fire conditions, for the material to be considered self-extinguishing, the minimum LOI value recommended at ambient temperature is 28% [36]. Recognizing that achieving the recommended LOI values of the materials is crucial to meet exigency in practical fire scenarios, additional studies are required. Thus, in our future work, we will explore various processing conditions of the composites, methods of processing (e.g., single or twin-screw extrusion process), and adding various compatibilizers and lubricating agents to the recycled PE. The combination of intumescent flame retardants (APP or MPP) with inorganic flame retardants and other new FR materials based on carbon nanomaterials will also be investigated.

2.5. Evaluation of the Mechanical Properties

The mechanical properties of the neat r-LLDPE/r-LDPE polymer blend and its composites were assessed through tensile and Izod impact tests, and the results are summarized in Table 4. The tensile test provides three critical properties—elastic modulus, tensile strength, and tensile strain at break, and these are essential for evaluating the materials' potential applications. Tensile properties of rLLDPEr/LDPE are typically affected by the addition of flame-retardants, but the specific effect depends on the types and contents of the flame-retardant [37].

In Table 4, on the one hand, it is observed that the elastic modulus increases with an increase in the weight fraction of APP, while the tensile strength decreases with an increase in APP loading. On the other hand, a decrease is observed for elastic modulus, tensile strength, tensile strain at break, and IZOD impact strength with an increase in the weight fraction of melamine polyphosphate. It is important to note that the addition of IFR into the polymer can create more defects, such as holes and cracks, during the preparation of composites, and therefore, mechanical properties (i.e., tensile strength) are reduced [38].

Table 4. Summary of mechanical properties of neat r-LLDPE/r-LDPE polymer blend and its composites.

Samples	Elastic Modulus (MPa)	Tensile Strength (MPa)	Strain at Break (%)	Notched Izod Impact Strength (kJ/m^2)
Neat rLLDPE/rLDPE	451.9 ± 2.2	29.1 ± 0.3	60.6 ± 2.0	28.7 ± 0.9
rLLDPE/rLDPE/APP5	461.4 ± 1.6	26.9 ± 1.6	54.2 ± 1.7	7.8 ± 0.4
rLLDPE/rLDPE/APP10	478.4 ± 1.4	26.2 ± 1.2	59.8 ± 2.0	7.6 ± 0.7
rLLDPE/rLDPE/MPP5	520.4 ± 1.9	28.5 ± 0.8	39.1 ± 1.5	7.2 ± 0.7
rLLDPE/rLDPE/MPP10	515.2 ± 1.7	28.0 ± 0.5	28.9 ± 2.0	5.3 ± 0.4
rLLDPE/rLDPE/MPP5/APP5	503.1 ± 1.9	26.7 ± 1.0	31.0 ± 1.6	6.6 ± 0.9

When APP and MPP flame retardants were combined, a synergistic effect was observed for the mechanical properties. MPP was more effective in improving the modulus due to its smaller particle size and better distribution, as shown in the SEM images. Remarkably, the elastic modulus of the rLLDPE/rLDPE/MPP5 composition was the highest amongst the composites, and this composition had similar tensile strength to the neat, recycled blend, while the rLLDPE/rLDPE/APP10 showed the highest strain at break among all the composites. Our results concur with the findings of Lim et al. [12] that increasing APP had a negative effect on the tensile strength of polyethylene composites. Moreover, an increase in APP or MPP causes a decrease in the tensile strain at break and notched Izod impact strength of the composite [17]. We postulate that the tensile and impact strength of composites is reduced compared with neat rPE due to the presence of defects at the interfaces between IFR and rPE.

3. Materials and Methods

3.1. Materials

LLDPE/LDPE Recycled Resin (Ex-PCR-NC4 with a density of 0.925 g/cm^3 and melt flow index (MFI) of 1.0 g/10 min—2.16 kg at 190 °C) was supplied by NOVA Chemicals (Calgary, AB, Canada). Non-halogenated intumescent flame retardants, Ammonium Polyphosphate (APP) $(NH_4PO_3)_n$, and Melamine Polyphosphate (MPP) $(C_3H_8N_6)_m(HPO_3)_n$ were supplied by Xingxing Flame Retardant Co., Ltd. (Zhenjiang, China).

3.2. Preparation of r-LLDPE/r-LDPE Containing Intumescent Flame Retardants

The r-LLDPE/r-LDPE polymer blend and 5 wt.% and 10 wt.% of non-halogenated intumescent flame retardants (APP and MPP) were prepared using a Haake Rheomix batch mixer (Thermo Fischer Scientific, Waltham, MA, USA). The flame retardants were added in raw form to the polymer blend (rLLDPE/rLDPE), and mixing was performed for 12 min at a rotor speed of 100 rpm and temperature of 210 °C. During processing, melt temperature and torque were continuously monitored. The sample preparation for all characterizations was performed using a compression molding machine. Carver benchtop molding press (Wabash, IN, USA) and mold dimension of each sample are as stated in the different standards used. The compound formulations are listed in Table 5.

Table 5. Preparation of r-LLDPE/r-LDPE-based intumescent flame retardant compound.

Samples	rLLDPE/rLDPE (wt.%)	APP (wt.%)	MPP (wt.%)
Neat rLLDPE/rLDPE	100	0	0
rLLDPE/rLDPE/APP5	95	5	-
rLLDPE/rLDPE/APP10	90	10	-
rLLDPE/rLDPE/MPP5	95	-	5
rLLDPE/rLDPE/MPP10	90	-	10
rLLDPE/rLDPE/MPP5/APP5	90	5	5

3.3. Characterization Methods

The viscosities of the neat rLLDPE/rLDPE polymer blend and their composites were measured using a Dynisco Galaxy V capillary rheometer in the steady-state regime according to ASTM D3835-16 [39]. For this test, a silicon carbide die with a diameter of 1.25 mm and a length-to-diameter (L/D) ratio of 20 was used. Shear rates ranging from 10 to 1000 s^{-1} were analyzed at a temperature of 230 °C, and the data were corrected using the Weissenberg–Rabinovitch method. Mathematical fitting of the data was performed using the power law model (Equation (1)).

$$\eta = \mathrm{m}\gamma^{n-1} \quad (1)$$

where the steady viscosity (η) is described as a function of the applied shear rate (γ), the consistency index (m), and the pseudoplasticity index (n).

The dynamic rheological behavior of the neat rLLDPE/rLDPE polymer blend and the composites were analyzed using small-amplitude oscillatory shear (SAOS) tests with Rheometer MCR 302 (Anton Paar GmbH, Graz, Austria) under ambient air using a parallel-plate geometry (25 mm diameter and 1 mm gap). The samples were compression-molded into 25 mm discs using a Carver benchtop molding press (Wabash, IN, USA), and the analyses were conducted at a temperature of 230 °C. Before each test, strain sweep tests were performed to confirm that the applied strain amplitude of 0.5% was within the linear viscoelastic range.

By using scanning electron microscopy (SEM), the morphological properties of the neat polymer and its composites were determined using the cryo-fractured surfaces of the samples prepared at −80 °C. All surfaces were initially coated with platinum using a sputter-coater and imaged with an FEI Quanta 250 FESEM microscope (FEI Company, Pantelimon, Romania)

The functional groups present in the neat rLLDPE/rLDPE polymer blends and rLLDPE/rLDPE polymer blend composites were determined using a Cary 630 FTIR spectrometer (Agilent Technologies, Santa Clara, CA, USA) spanning wavenumber ranging from 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1}. The samples were analyzed using a universal attenuated total reflection (ATR) accessory. IR spectroscopic measurements were performed on the rLLDPE/rLDPE polymer blend with ammonium polyphosphate and melamine polyphosphate composites.

TA model Q2000 dynamic scanning calorimetry (TA Instruments, New Castle, DE, USA) analyses of the polymer blend and its composites were carried out under a nitrogen atmosphere, over a temperature range from 25 °C to 250 °C, with a heating rate of 5 °C·min^{-1}. The DSC procedure included a heating cycle, followed by cooling, and then a second heating cycle. From the melting enthalpy (ΔH_m^0) obtained in the second heating

cycle, the degree of crystallinity (X_c(%)) of the r-LLDPE/r-LDPE matrix was determined using Equation (2).

$$X_c(\%) = \frac{\Delta H_m}{\theta \Delta H_m^0} \times 100\% \quad (2)$$

where X_c is the degree of crystallinity; ΔH_m is the enthalpy of melting of the semi-crystalline polymer; θ is the specific polymer mass fraction in the sample, and ΔH_m^0 is the enthalpy of melting of the 100% crystalline polymer (For Polyethylene, ΔH_m^0 = 293 J/g) [19].

The thermal stability and char yield of the polymer composites was evaluated using thermogravimetric analysis equipment TGA Q500 V20.10 Build 36 (TA Instruments, New Castle, DE, USA). All samples had weights ranging from 5 to 10 mg, and the conditions used in this study were as follows: temperature range from ambient temperature to 900 °C, nitrogen gas flowrate of 50 mL min^{-1}, and heating rate of 10 °C min^{-1}. The flame retardance of rLLDPE/rLDPE blends was investigated using the limiting oxygen index (LOI) equipment Model AT-P6012A (Amade Technology Co., Ltd., Hong Kong, China). This equipment gives rapid information regarding the minimum oxygen concentration needed to stop the burning of the material at ambient temperature, following the ASTM D2863 standard [40]. Bar-shaped samples with dimensions of length of 80 mm ± 2 mm, width of 12.5 mm ± 0.02 mm, and thickness of 3 mm ± 0.2 mm that were compression-molded using a Carver benchtop molding press (Wabash, IN, USA) were used for the LOI test.

Tensile measurements for both neat rLLDPE/rLDPE polymer blends and the composites were evaluated using an INSTRON 5965 tensile testing machine (Norwood, MA, USA) with a 5 kN maximum force capacity. The dumbbell-shaped samples were compression-molded using a Carver benchtop molding press (Wabash, IN, USA), and the tests were performed following ASTM D638 [41], and the reported values represent the average of five samples for each formulation. The impact strength of the neat and flame-retardant rLLDPE/rLDPE polymer blend composites was measured using a Tinius Olsen Model 104 Notched Izod impact tester (Horsham, PA, USA) at a temperature of 23 ± 2 °C, following ISO 180 standards [42]. The sample is a rectangular-shaped bar prepared by compression molding using a Carver benchtop molding press (Wabash, IN, USA). For this test, notched samples were prepared using a Qualitest QC-640A impact specimen angle-cutting device (Lauderdale, FL, USA) to create a v-notch. The dimensions of the samples are the length of 80 mm ± 2 mm, width of 12.5 mm ± 0.02 mm, thickness of 3 mm ± 0.2 mm, notch depth of 2 mm ± 0.1 mm, notch angle of 45° ± 1° (for v-notch), and notch radius of 0.25 mm ± 0.05 mm (v-notch). The Izod impact strengths (kJ/m^2) represent the average values of five samples per formulation.

4. Conclusions

Non-halogenated intumescent flame retardants (IFRs) blended into recycled linear low-density polyethylene (rLLDPE) and low-density polyethylene (rLDPE) polymer blends increased flame retardance and improved the material's thermal stability at high temperatures. Morphological analysis confirmed that IFRs were well dispersed within the polymer matrix, giving improved fire resistance. Rheological tests revealed that APP had a greater effect on increasing viscosity and shear modulus than MPP. The addition of APP and MPP improved LOI values of recycled PE via effective char formation by the IFRs; i.e., the char acted as a protective barrier, inhibiting heat and oxygen transfer during combustion. The rLLDPE/rLDPE blends containing a combination of APP and MPP exhibited the highest char residue (3.00%) at 900 °C and the highest LOI value (22.4%). This suggests a synergistic effect between APP and MPP, facilitating enhanced char formation, and this char is essential for improving flame retardance of rPE.

Mechanical testing results revealed that while the addition of IFRs generally reduced tensile strength by 9.7%, the composites exhibited an increase in elastic modulus by 15.0%, and these trends are in agreement with the literature. The combination of APP and MPP led to a balanced performance in terms of mechanical properties, with MPP significantly influencing the modulus by an increase of 9.1%. The results provide valuable insights into the potential of recycled polyethylene composites in advancing circular economy practices, reducing plastic waste, and developing sustainable building materials. These findings open the door to further studies to optimize the formulation and explore new flame-retardant systems to meet stringent fire safety standards across various industries.

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