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Acoustic emission analysis of failure processes in polyethylene and recycled tire rubber blends

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ABSTRACT

In this study, we developed a test method using acoustic emission (AE) to investigate the failure process and degree of compatibility of low-density polyethylene (LDPE) blends filled with ground tire rubber (GTR). The blends contained 40 wt% of GTR, which is incompatible with the matrix material and altered their fracture behavior significantly. We could define three stages of the tensile curves based on the amplitude of the emitted signals. Furthermore, we demonstrated the effect of compatibilization using ethylene-vinyl-acetate: compatibilization facilitated a stronger interface between the matrix and filler, resulting in signals with higher amplitude. We analyzed the main signal properties and found a shift indicating compatibilization in all of them. We also studied the crack propagation of the specimens using tear tests and found that the incorporation of EVA in LDPE-GTR blends facilitated a more stable crack propagation, as indicated by fewer acoustic events.

1. Introduction

The recycling of rubber waste is a pressing issue nowadays: several million tons of rubber waste are generated yearly, mainly from tire rubber [1–3]. A significant obstacle to recycling is the inability of vulcanized rubber to be remelted, due to the presence of stable crosslinks formed during vulcanization. Therefore, other methods are necessary for the large-scale recycling of tire rubber. One promising approach involves incorporating rubber waste into various materials, including asphalt, elastomers, and thermoplastic polymers [4]. For this application, tires are typically processed into ground tire rubber (GTR) through shredding [5,6]. Among thermoplastic polymers, the most suitable ones for this application are polyolefins because they are relatively cheap and can be modified easily to fit a wide range of requirements [7–9].

The main advantage of filling GTR in thermoplastics lies in the cost-effectiveness of both components. Additionally, their blends can often be classified as thermoplastic elastomers (TPEs), materials that combine rubber-like elasticity with the processability and recyclability of thermoplastics [10–13]. This classification is only possible if certain requirements are met: at least 100 % elongation at break with rubber-like tensile characteristics [14–16]. This method of upcycling rubber waste is attractive to the industry because of its profitable nature. However, to achieve the desired properties for TPE, effective phase compatibilization is necessary, as GTR and polyolefins are incompatible with each other [17,18].

Compatibilization can be achieved in several ways: physical and chemical processes are widely researched in the literature [19–23]. It is important to note, however, that while the quality of the interphase can be significantly improved, full compatibility between components cannot be achieved. Physical compatibilization has the advantage of easier upscaling compared to chemical methods, as it mainly involves mixing a third type of polymer that can improve the interphase [24,25]. One of the most promising compatibilizers for polyethylene/GTR blends is ethylene vinyl acetate (EVA) copolymer: several studies show that the presence of EVA enhances interfacial bonding between PE and GTR, leading to improved mechanical performance [26,27]. The compatibilization effect of EVA can be attributed to its molecular structure: the presence of vinyl acetate in EVA contributes to its compatibility with polar and nonpolar materials, allowing for EVA to interact effectively with both PE and GTR. In addition, the modulus of EVA is between that of the rubber and LDPE, and it can provide better load dispersion [28].

Although rubber-filled thermoplastic polymers have been

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extensively studied, their failure behavior under tensile loads remains relatively unexplored. The acoustic emission (AE) test is a potential method for investigating the failure behavior. Acoustic emission means the transient elastic waves within a material generated by rapid energy release, mainly from damage-related sources [29]. The acoustic emission method is widely used in structural materials for detecting and monitoring the initiation and propagation of different types of damage [30]. In terms of polymeric materials, fiber-reinforced composites with thermoset [31–33] or thermoplastic matrix [34–37] are the main areas where the AE method is used for damage analysis and structural health monitoring purposes. Acoustic emission testing has demonstrated great potential in the field of self-reinforced polymer composites to investigate their failure mechanisms as well [29,38,39]. Furthermore, the acoustic emission method has been successfully used for the investigation of particle-filled polymers. Relevant studies show that the quality of adhesion between the polymer matrix and the particles can be associated with the intensity of acoustic activity and several properties of the noted acoustic events, e.g. amplitude, energy, counts [40–43].

The incorporation of waste tires into thermoplastic polymers is an economically viable and sustainable solution for the integration of end-of-life tires into the circular economy. However, it is not sufficient to know only the basic properties of these materials, it is also important to have a deeper understanding of their properties. Our study introduces a novel approach to evaluating the damage mechanisms in polyethylene filled with ground tire rubber using acoustic emission analysis and the active damage monitoring of these blends. By applying this method, we provide deeper insights into the microstructural damage and interfacial interactions, offering a new perspective on their performance and durability. Our findings contribute to the advancement of GTR recycling by allowing for more predictable failure.

2. Materials and methods

2.1. Materials

We used Tipolen FD 243–51 (MOL Petrochemicals Ltd. (Tiszaújváros, Hungary)) type low-density polyethylene (LDPE) as the matrix material of the blends.

We used ground tire rubber from truck tires provided by GreenTyre Ltd. (Marcali, Hungary). The GTR was characterized by a grain size below 400 μm.

We used Escorene Ultra FL 00218 ethylene vinyl acetate (EVA) copolymer as a physical compatibilizer. The material was characterized by 18 % vinyl acetate content and is manufactured by Exxon Chemicals (Texas, USA).

2.2. Preparation of the TPEs

We prepared the blends with a corotating twin-screw extruder (Labtech Engineering Co., Ltd., Samutprakarn, Thailand) with a revolution speed of 120 rpm at 180 °C. The compositions of the compounds are listed in Table 1.

We prepared dumbbell and flat specimens of the blends by injection molding using an Arburg Allrounder Advance 270 S 400–170 type (Arburg GmbH, Lossburg, Germany) injection molding machine with parameters shown in Table 2.

2.3. Characterization methods

We performed tensile (according to ISO 37, Type 2 dumbbell specimens) and tear tests (according to ISO 34, Method B, angle test pieces) on standardized specimens. A Zwick Z005 (Zwick GmbH (Ulm, Germany)) universal testing machine equipped with a 5 kN load cell was used with a 100 mm/min crosshead speed for both tests.

Acoustic emission signals were recorded during tensile and tear tests in a measurement setup shown in Fig. 1. We recorded the signals with a Mistras PCI-2 (MISTRAS Group, Princeton Junction, USA) AE system. We used an IL40S preamplifier (Physical Acoustic Corporation, Princeton Junction, USA) with a gain of 40 dB and a Micros30s (Physical Acoustic Corporation, Princeton Junction, USA) microphone (operating frequency range: 150–400 kHz).

The proper connection between the sensor and the specimen was ensured by the application of an Oxett silicon grease (T-Silox Ltd., Budapest, Hungary) coupling agent. We set a 30 dB threshold for the measurements to filter out ambient noises according to previous experience [29]. The evaluation of the acoustic emission testing results was carried out with Noesis 9.0 and MATLAB R2024b software. In the case of analyzing AE signals, besides the number of detected AE events, several parameters of the acoustic wave can be investigated (Fig. 2). These parameters help identify different damage mechanisms and provide insights into the material's structural integrity. Amplitude is one of the key parameters, which means the peak voltage of an AE signal, typically measured in decibels (dB). More significant damage mechanisms result in signals with a higher amplitude, thereby, amplitude might indicate the quality of interfacial connections. Signal strength is a measure of the total power of the AE signal, often calculated as the integrated energy over time. It provides additional insight into the severity of damage, complementing amplitude and energy analysis. Counts mean the

Table 1
Formulation of the compounds.

Compound	Components
LDPE	100 wt% LDPE
LDPE_EVA	85 wt% LDPE +15 wt% EVA
LDPE_GTR	60 wt% LDPE +40 wt% GTR
LDPE_GTR_EVA	45 wt% LDPE +40 wt% GTR +15 wt% EVA

Table 2
Injection molding parameters.

Temperature profile [°C]	190/190/185/180/175/45
Dosage [cm^3]	45
Residual cooling time [s]	20
Injection rate [cm^3/s]	25
Holding pressure [bar]	350
Back pressure [bar]	40
Mold temperature [°C]	30

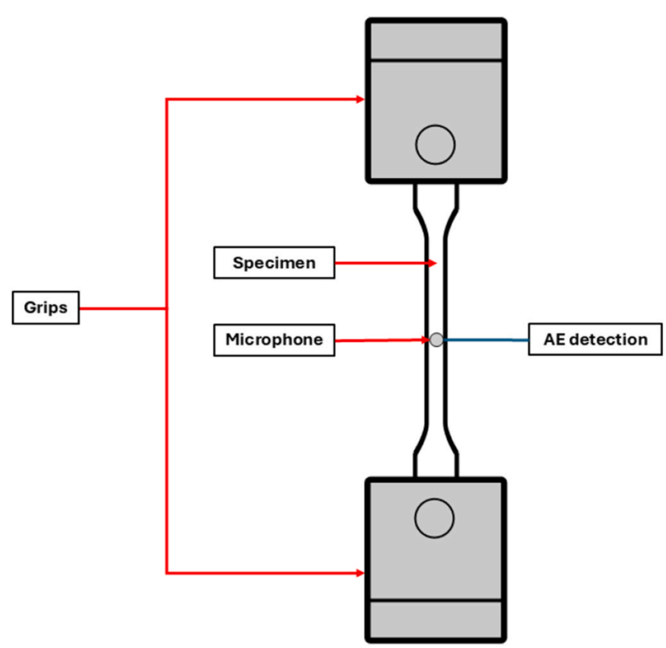

Fig. 1. Measurement setup for detecting acoustic emission signals.

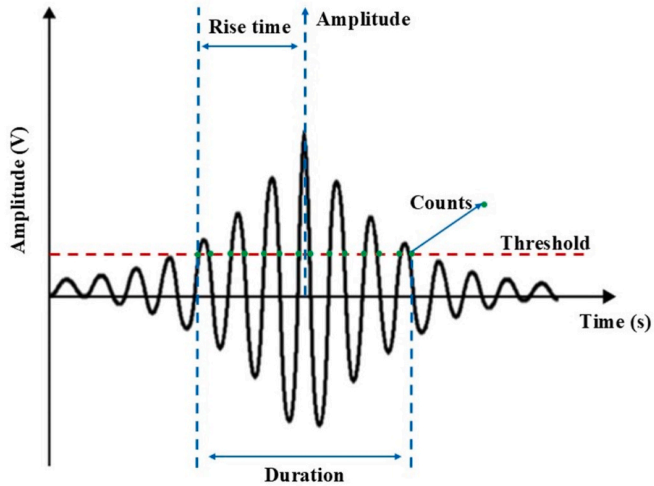

Fig. 2. Interpretation of acoustic emission signal properties (based on [46]).

number of times the waveform signal crosses the threshold, indicating the complexity of the event. Duration means the time interval between the first and last threshold crossing. Usually, longer durations may suggest progressive damage and stronger connections. Rise time, which means the time between the first threshold crossing and the peak amplitude, can be an important parameter as well. A shorter rise time may indicate more sudden damage events, meanwhile, a longer rise time suggests a more stable damage mechanism. Besides, analysis of the signal's frequency can provide additional information about the manner of the damage and might even help with differentiating between damage types [29,44,45].

The morphology of the samples was examined using a JEOL JSM 6380 L A (Jeol Ltd., Tokyo, Japan) scanning electron microscope (SEM) with an accelerating voltage of 10 kV and a spot size of 40 nm, and a magnification of 100 and 1000. We examined the cross-section of severed tensile test specimens in order to assess the contact between the phases.

3. Results and discussion

3.1. Morphology

The SEM pictures of the cross-sections (Fig. 3) show the morphology and the connection between the thermoplastic phase and the GTR. The two phases are clearly identifiable in the images: the thermoplastic phase is characterized by a structured surface due to the tough failure, while the GTR is recognizable by its polygonal shape. It can be observed that although LDPE surrounds the GTR well, there is no connection between the phases, with cavities and separations observed at the interphase (Fig. 3/a and c). In contrast, in the case of blends compatibilized with EVA, the thermoplastic phase not only surrounds the GTR well (Fig. 3/b), but the compatibilizing effect of EVA results in small bridges between the phases, indicating better connection ((Fig. 3/d). However, EVA does not provide full compatibility, cavities can also be seen at some interfaces.

3.2. Identifying the stages of failure

The analysis of the blends (Fig. 4) shows that GTR-filled LDPE blends can be divided into three distinct stages based on the signal amplitudes and the number of acoustic events (as marked in the figures with red lines): elastic deformation, plastic deformation and failure stage. In the elastic deformation stage few signals with amplitudes between 30 and 60 dB can be observed. This can be related to the yielding of the matrix ligaments, as well as some phase separation at the weakest interphases. The plastic deformation stage is characterized by many signals, typically below 50 dB of amplitude. These signals can be attributed to the separation of the LDPE and GTR phases. Due to the incompatibility of the two

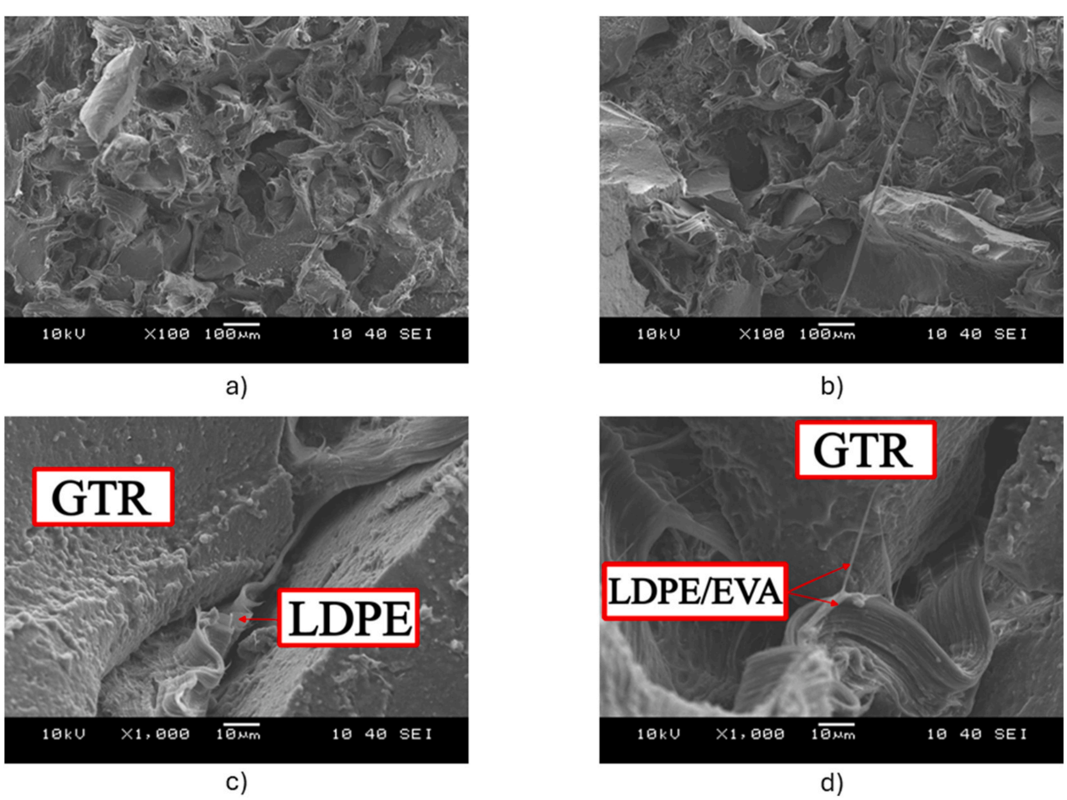

Fig. 3. SEM images of the blends a) and c): LDPE_GTR, b) and d): LDPE_GTR_EVA.

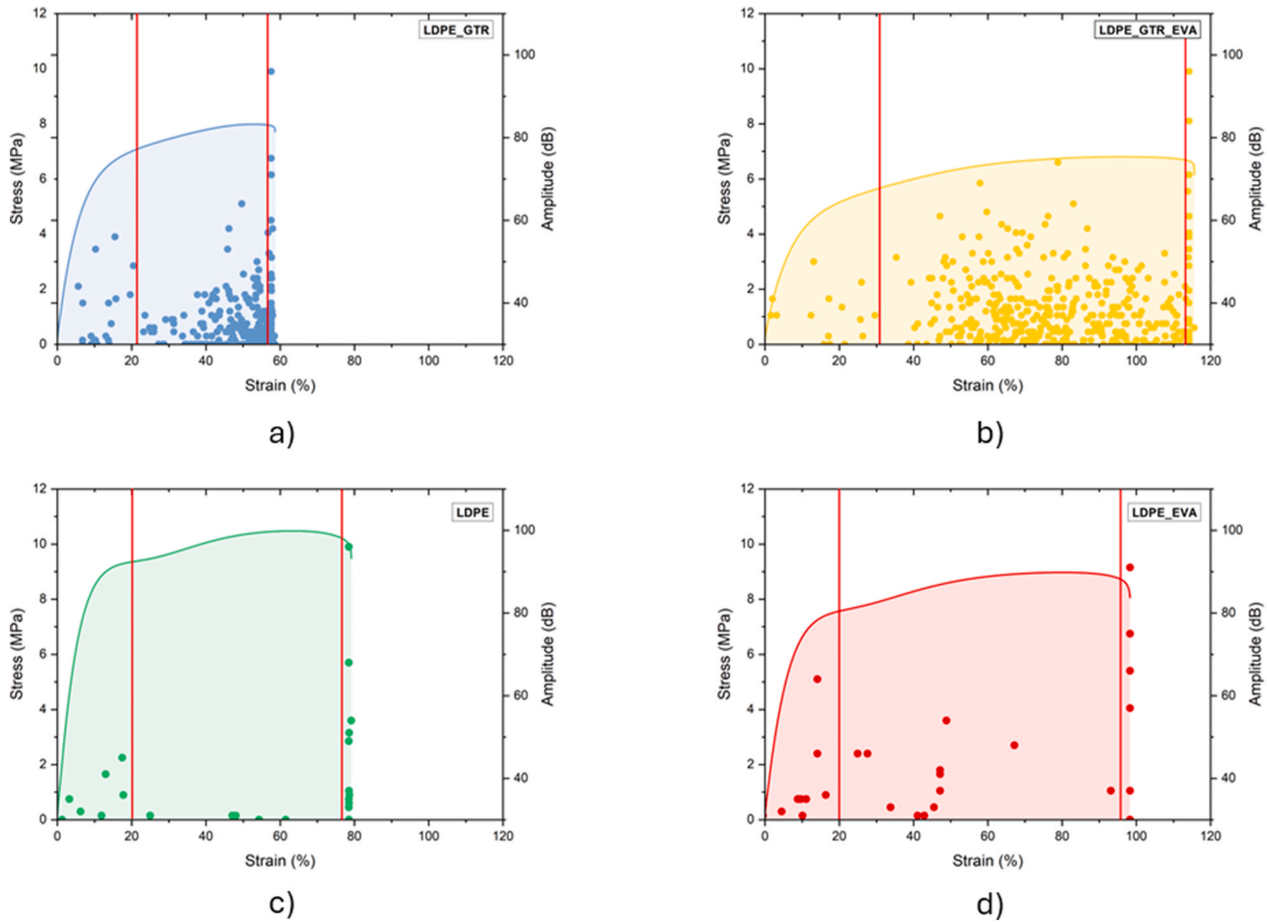

Fig. 4. Tensile curves of the blends with the corresponding amplitudes: a) LDPE_GTR, b) LDPE_GTR_EVA, c) LDPE, d: LDPE_EVA.

phases, their separation does not require high energy; therefore, it is accompanied by low amplitude signals. The increased number of signals in this stage comes from the numerous interphases that can fail at higher strain. The last stage marks the macroscopic break: it is accompanied by a signal with an amplitude above 90 dB and presumably its echoes.

The effect of EVA (Fig. 4/b) is most evident in the plastic deformation stage of the curve; both the number of acoustic events and the signal amplitudes increase significantly. This can be in connection with improving the interphase between LDPE and GTR, which results in more gradual damage processes. Improved interphase quality increases the energy required for failure, which correlates with higher amplitude values.

Based on FEM models, a micromechanical model for the failure process of rubber-filled thermoplastic polymers has been previously established in the literature [47,48]. According to this model, the failure happens in the thermoplastic ligaments, the stepwise nature of the failure ensures elastic recovery. A ligament is formed parallel to the load, which can spring back the other segments, guaranteeing elastic recovery at high strains. If the connection between the matrix and the GTR is strengthened, this will postpone the failure of the ligaments and increase the elastic recovery and, consequently, the rubbery behavior. Failure starts at the boundary of the rubber domains and propagates from there to the matrix ligaments, with AE signals resulting from ligament rupture. We can observe that if the boundary phase improves, the amplitude of the signals increases as more energy is required to tear the ligaments. We can also observe the improvement of these "spring ligaments" in the SEM pictures (Fig. 3): in the compatibilized blends, the ligaments are observed to be more elongated and deformed. This suggests that they were able to undergo greater deformation, thus providing greater elastic recovery and rubber-like behavior to the blends.

We also examined the failure process of the matrix materials as well (Fig. 4/c and d) to confirm that the enrichment of signals in the plastic deformation stage is truly in connection with the matrix-GTR separations. It can be seen in Fig. 4/c that the failure process of neat LDPE can also be divided into the same three stages: elastic, plastic and failure. The elastic deformation stage is marked by signals of 40–50 dB, and the failure stage is indicated by a strong signal of 70 dB and its echoes. The plastic section contains fewer signals compared to the LDPE_GTR blend due to the lack of fillers and interphases. Signals in this stage can be attributed to microscopical failures caused by the crazing of the material [49].

The curve belonging to LDPE_EVA (Fig. 4/d) also exhibits the three stages of failure, except more signals were detected with higher amplitude (up to 55 dB). This can be attributed to the dual-phase morphology of this blend: since EVA and LDPE are immiscible [50], the EVA particles can separate from the LDPE matrix, generating AE signals. These signals can be observed in the plastic deformation stage of TPEs as well, along with the signals generated by GTR debonding.

3.3. Analysis of the signal properties emitted during the tensile tests

In the next step, we analyzed the effect of EVA compatibilization on the properties of the acoustic signals collected during the tensile tests. The failure stage was not included in the analysis of the signal characteristics as it refers to the global failure instead of the formation and propagation of damage inside the material. Furthermore, the properties such as the amplitude of the signals found there would have distorted the analysis. In Fig. 5/a, it can be observed that the amplitude of the signals increases as a result of the compatibilization. This shift can be attributed to the fact that more energy is released during phase

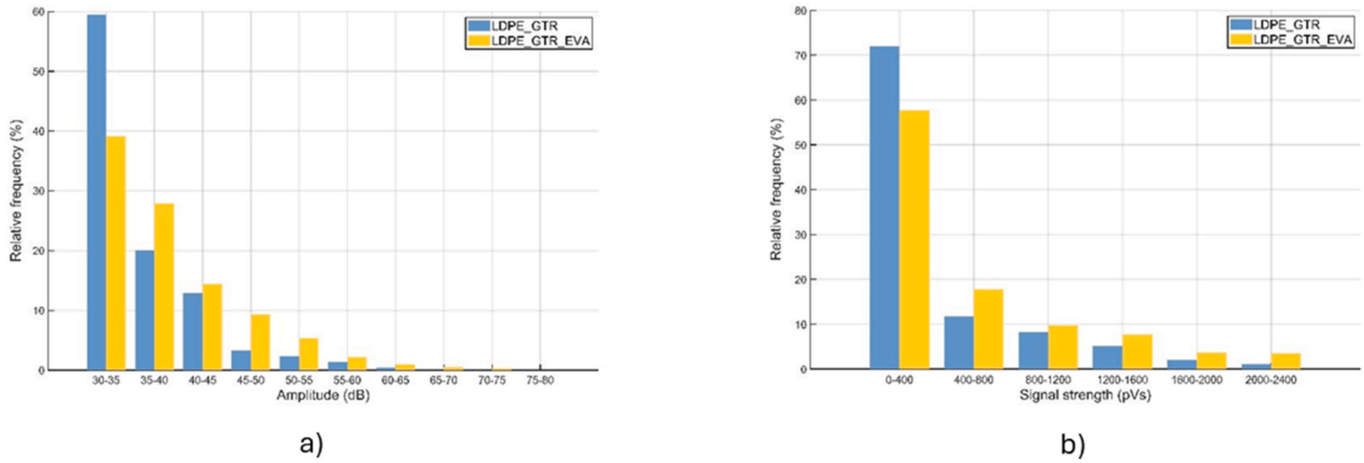

Fig. 5. Amplitude (a) and signal strength (b) distribution of the signals emitted during the tensile tests.

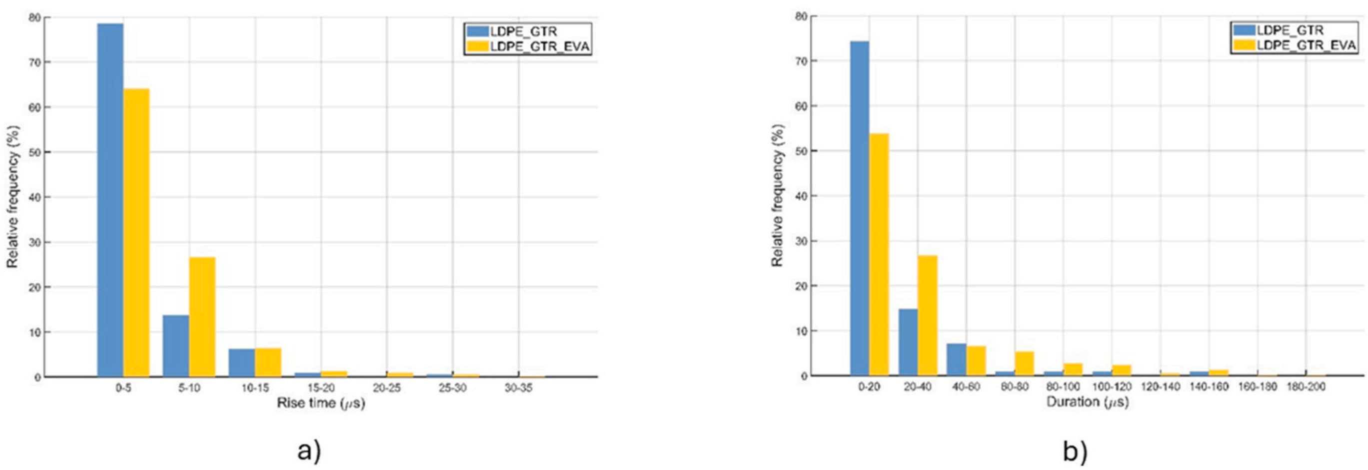

Fig. 6. Rise time (a) and duration (b) distribution of the signals emitted during the tensile tests.

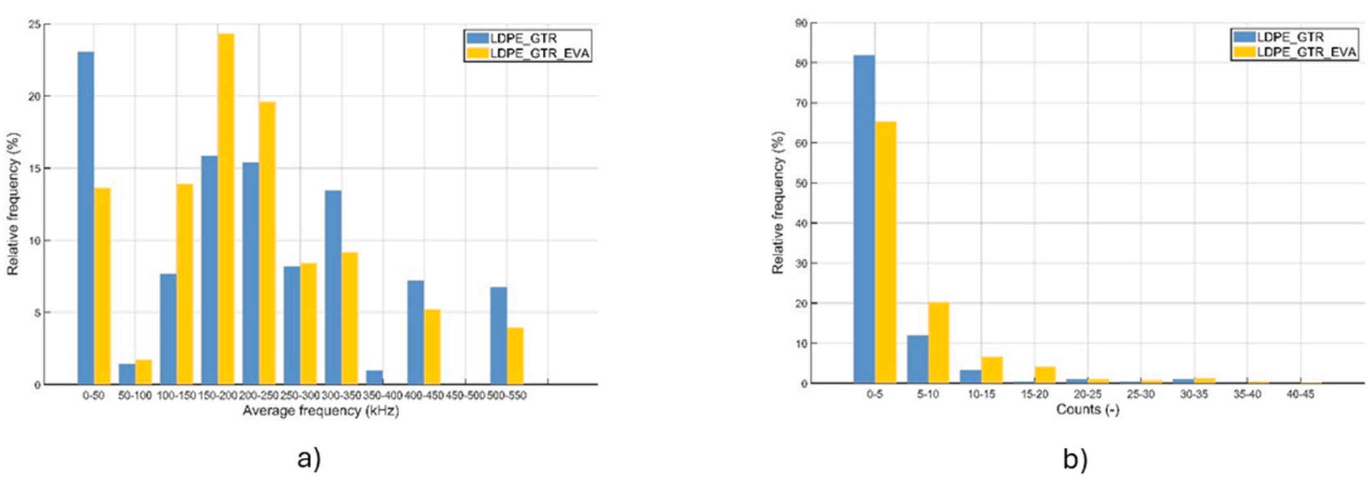

Fig. 7. Average frequency (a) and counts (b) distribution of the signals emitted during the tensile tests.

separation due to the improvement of the interphases. Low amplitude signals indicate phase separations where the connection between the phases is poor. This is because less energy is released when such a connection is broken; therefore, an increase in amplitude and signal strength indicates an improved connection. This effect can also be observed in the signal strength histogram (Fig. 5/b): the distribution shifts towards higher signal strengths as the interphases improve due to compatibilization.

We also analyzed the change in the rise time and the duration of the signals (Fig. 6/a and b). The upward shift of these properties indicates a more prolonged signal resulting from the improvement of the interphase. The incorporation of EVA results in a more gradual failure: the damage spreads more slowly at the improved interface and phase separation is less abrupt.

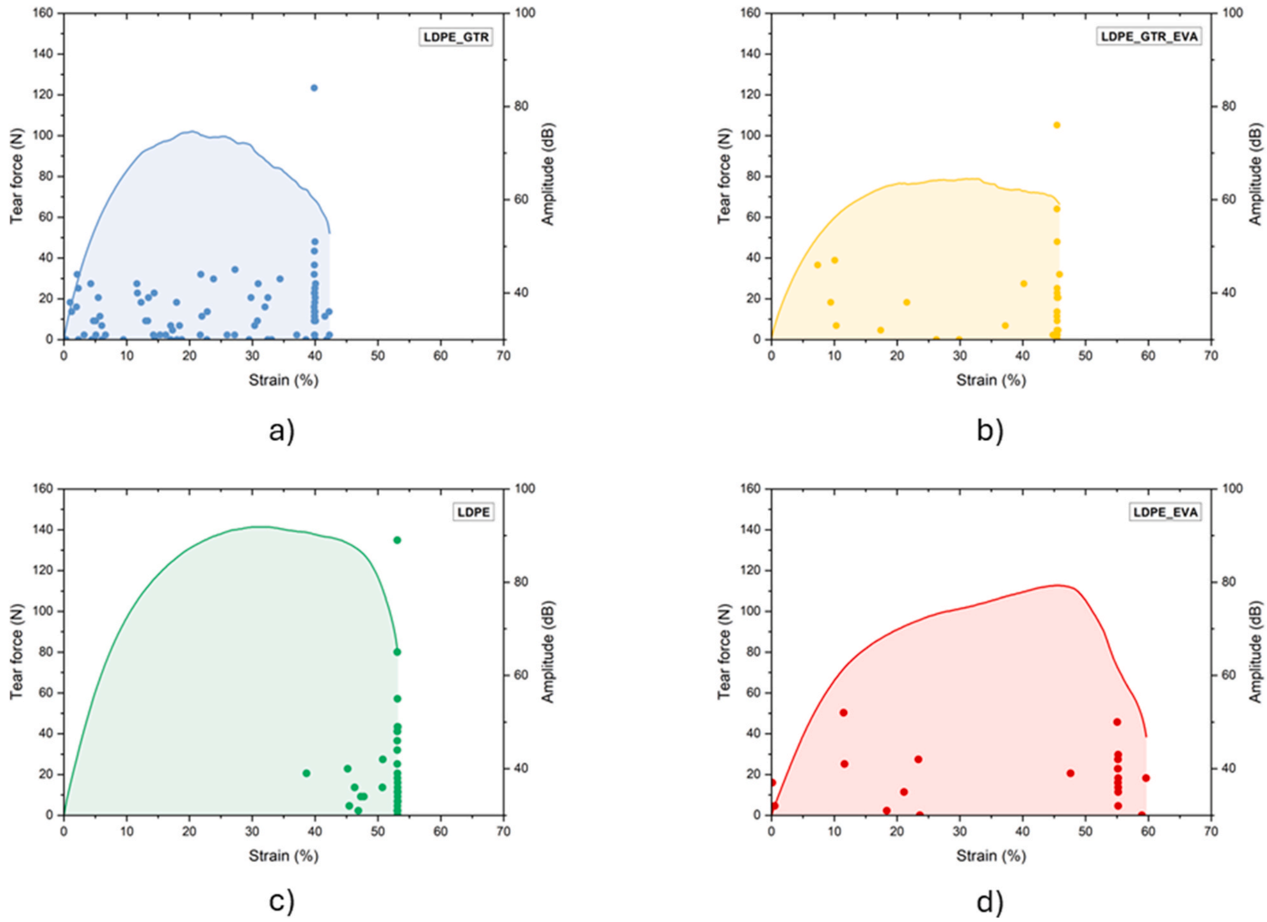

Fig. 8. Tear curves with the corresponding amplitudes: a) LDPE_GTR, b) LDPE_GTR_EVA, c) LDPE, d) LDPE_EVA.

Significant changes in the distribution of average frequency are also observed due to improved compatibility (Fig. 7/a). The frequency distribution for the compatibilized blend is more similar to normal distribution, with values within a narrow frequency range dominating. In addition, the proportion of signals with very low average frequencies is reduced: this is due to the more controlled failure process caused by compatibilization. The shift in the distribution of counts (Fig. 7/b) indicates a reduced number of weak signals due to compatibilization. The incorporation of EVA can reduce the impact of sudden debonding.

3.4. Tear tests

We analyzed the fracture mechanics of the blends with tear tests (Fig. 4). The neat LDPE (Fig. 8/c) is characterized by "soft" crack propagation: signals are observed only before fracture, and all signs of failure might emit signals with amplitudes below 30 dB. Fracture is indicated by a large amplitude signal similar to the tensile curves. The tear properties of the LDPE_EVA blend (Fig. 8/d) show signs of phase separation indicated by the appearance of signals in the beginning and middle of the curve, similar to the tensile results. We also observed a significant decrease in the amplitude of the failure and the increase of

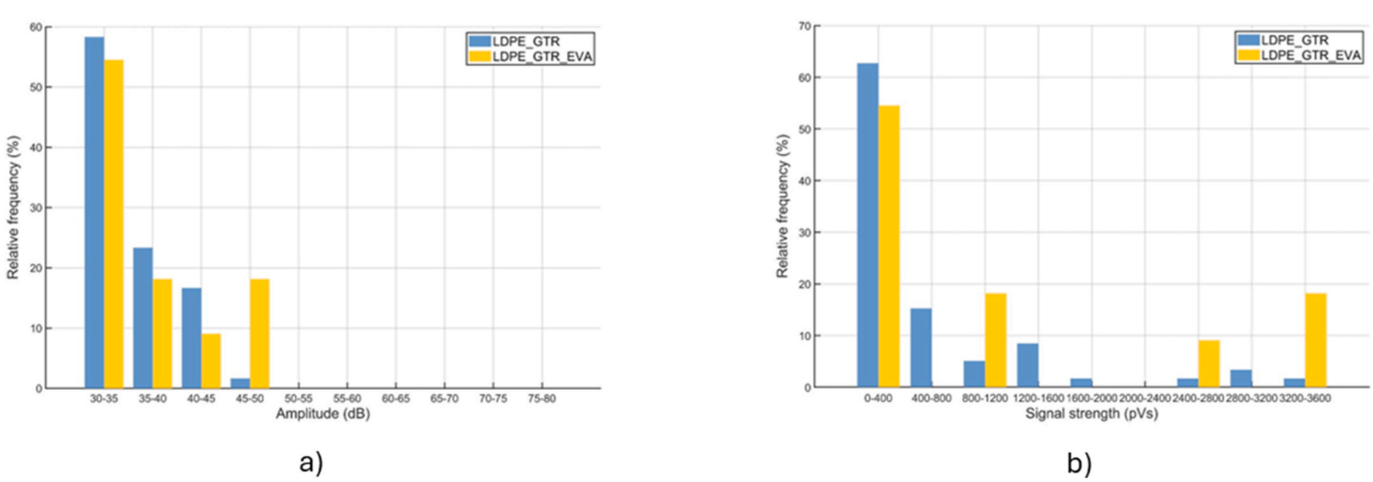

Fig. 9. Amplitude (a) and signal strength (b) distribution of the signals emitted during the tear tests.

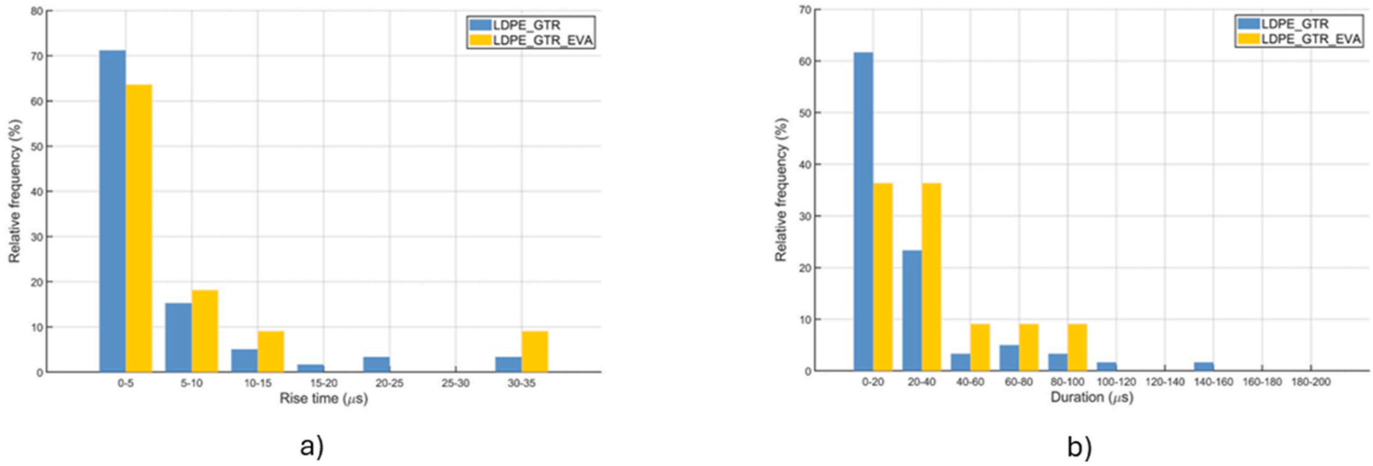

Fig. 10. Rise time (a) and duration (b) distribution of the signals emitted during the tear tests.

tear strain caused by the incorporation of EVA, which can be explained by the crack propagation through the interphases as well. The signals are distributed more evenly throughout the failure process, which might suggest a more even crack propagation.

Similarly to the tensile tests, the number of AE signals increases drastically with the incorporation of GTR (Fig. 8/a). This effect can be attributed to the change in crack propagation: the initiated crack can proceed in multiple ways through the weak interphases between LDPE and GTR. This behavior can also be seen on the tear curve itself: phase separation resulting in unstable crack propagation can be observed by downward steps consistent with numerous AE signals with an amplitude between 30 and 50 dB. The tear test qualifies the propagation of a crack in a known cross-section. In this case, a different trend from the tensile test is observed: the number of signals does not increase but decreases due to compatibilization. This can be explained by the improved interphases, which prevent crack propagation in multiple directions resulting in stable crack propagation along one crack path. Therefore, the cracks can only propagate through weaker interphases as seen in the SEM pictures (Fig. 3), so the collected signals in case of the compatibilized specimen are related to these remaining – low number of – weak interphases.

This can be supported by the fact that the amplitude of signals associated with LDPE_GTR_EVA is within the same interval as the signals observed at LDPE_GTR, which indicates that the crack does not propagate along stronger interphases, compatibilized with EVA. The addition of EVA in LDPE_GTR blends also affects the tear curve: the curve lacks the downward steps in tear force due to the more stable crack propagation.

3.5. Analysis of the signal properties emitted during the tear tests

We carried out further investigation related to the acoustic properties of the signals collected during tear tests. Our focus was on the effect of EVA as a compatibilizer on crack propagation. An upward shift can be seen in the amplitude distribution (Fig. 9/a) which indicates the improvement of interphases similar to the signals collected from tensile tests (Fig. 4/a). However, it should be noted that both materials emit signals in the same range. The increasing signal strength (Fig. 9/b) also indicates stronger interphases. The values between 2600 and 3400 pVs might be associated with crack initiation on stronger interphases.

As for the rise time and duration (Fig. 10/a and b), the range of the signals is the same as crack propagation could have occurred in LDPE_GTR_EVA through some interphases with less effective compatibilization. However, the characteristic shift can also be observed in these properties, indicating a more stable crack propagation because even the weaker interphases in the blend are improved compared to the blend without compatibilization.

The average frequency distribution (Fig. 11/a) shows a similar trend as the signals from tensile tests: the concentration of signals indicates that even the weaker interphases are improved in the blend, resulting in more stable and controlled crack propagation. Meanwhile, due to the incorporation of EVA, the distribution of counts (Fig. 11/b) is in the same range as the uncompatibilized blend with a slight shift to the right. This indicates that the crack can propagate only through the weakest interphase with similar signal counts.

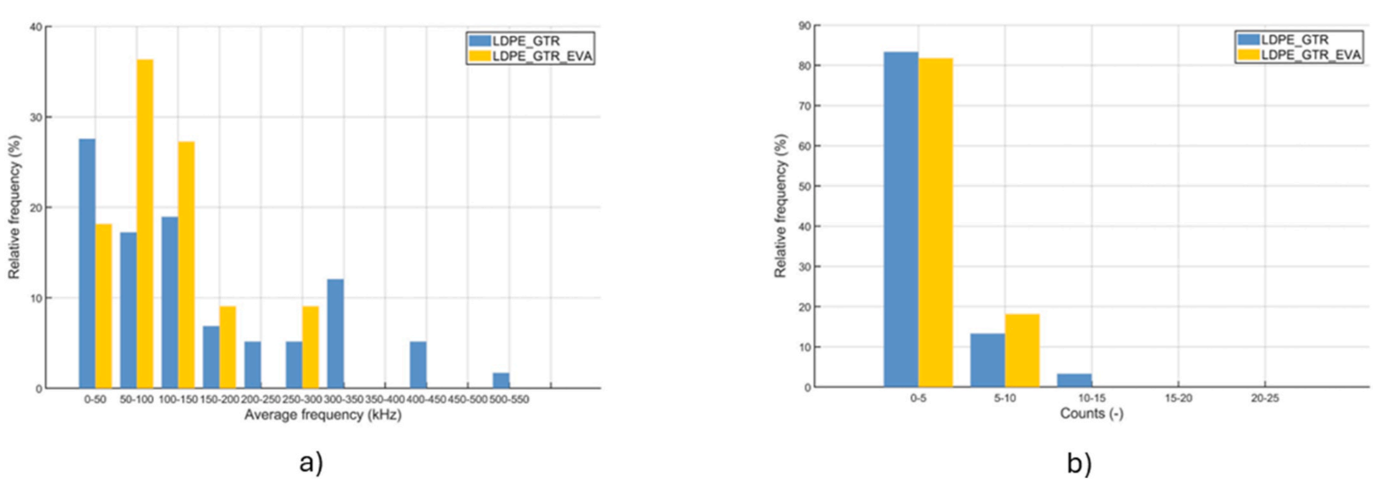

Fig. 11. Average frequency (a)) and counts (b)) distribution of the signals emitted during the tear tests.

4. Conclusions

We investigated the failure mechanisms of LDPE and GTR blends with acoustic emission analysis, focusing on the effect of EVA compatibilization. Our findings showed that the incorporation of EVA improved the interphase in LDPE/GTR blends, which resulted in increased strain at break and decreased tensile strength and modulus. In addition, the impact of compatibilization was also observed in the SEM images: the incorporation of EVA resulted in a considerable improvement in the connection between the GTR and the thermoplastic phase.

Based on the amplitude of the acoustic emission signals, we could define three distinct stages in the tensile curves LDPE/GTR blends: an elastic deformation, a plastic deformation and a failure stage. We found that the effect of EVA is most prominent in the plastic deformation stage: both the number and the amplitude of signals increase due to compatibilization. We confirmed that the signals in this stage are connected to matrix-GTR separation during the examination of the matrices. We have also performed a detailed analysis of the main properties of AE signals and found a shift connected to improved interphases in all of them.

We examined the fracture mechanics of the blends with tear tests and found that the number of AE signals increases with the incorporation of GTR as the cracks can proceed through the weak interphases. The results of the tear tests confirmed that EVA facilitated more stable crack propagation, as evidenced by the disappearance of downward steps in the tear curve and the reduced number of AE events as well. We believe that our work is an important step in understanding and improving the failure process of rubber-filled thermoplastic polymers, especially for GTR filling.

CRediT authorship contribution statement

Ákos Görbe: Writing – original draft, Visualization, Formal analysis, Data curation, Conceptualization. **Gergő Zsolt Marton:** Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Tamás Bárány:** Writing – review & editing, Supervision, Resources, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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