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ORIGINAL CONTRIBUTION

Morphology, rheological, thermal, and mechanical properties of high-density polyethylene toughened by propylene-ethylene random copolymers

Yi Li[1,2] · Dongdong Li[2] · Hongda Cheng[2] · Yancun Yu[3] · Ye Zhang[3] · Hechang Shi[3] · Changyu Han[3]

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Abstract

Propylene-ethylene random copolymer (PEC) elastomer was used to toughen high-density polyethylene (HDPE). The morphology, thermal behavior, rheological, and mechanical properties of HDPE/PEC blends were investigated. Scanning electron microscopy (SEM) results revealed that PEC-rich domains dispersed evenly in the HDPE matrix, and HDPE/PEC blends were immiscible. Rheological measurements indicated that the HDPE/PEC blends exhibited higher melt strength and viscosity than neat HDPE, which was beneficial to foaming and blow molding of HDPE. From the results of DSC, PEC elastomer inhibited the crystallization and reduced the degree of crystallinity of HDPE. It was noteworthy that the significant enhancement of the toughness of HDPE was obtained by adding PEC elastomer. The maximum elongation at break (684.6%) and impact strength (35.3 kJ m^{-2}) were achieved for the HDPE/PEC blends, which increased by 110% and 240%, respectively, compared with neat HDPE. Therefore, PEC elastomer could be used as an effective impact modifier for HDPE.

Keywords High-density polyethylene · Propylene-ethylene random copolymers · Blends · Toughening

Introduction

High-density polyethylene (HDPE) is a widely used thermoplastic polymer due to its light weight, low price, non-toxic, well humidity resistance, and good mechanical. HDPE is commonly applied in rotational molded, extruded pipes, and blown containers. Large-sized plastic products will be subject to more complex loads and greater shocks during transport and work [1, 2]. This requires polymers to have more excellent fracture resistance and low temperature toughness. At present, the methods of HDPE toughening are mainly divided into two categories, chemical modification and physical modification [3]. The former includes crosslinking and graft modification [4], and the latter includes blending with elastomer and adding fillers [5–10].

Among them, compounding HDPE with elastomers, such as natural rubber powder (NRP) [6], ethylene-octylene copolymer (POE) [11–13], scrap rubber powder (SRP) [5, 14], ethylene-propylene-diene terpolymer (EPDM) [15, 16], and ethylene vinyl acetate (EVA) [17, 18], is a simple and effective way to fabricate HDPE-based materials with high performance. For example, Sae-Oui et al. [6] found the improvements of elasticity, softness, and tensile strength of NRP/HDPE blend with the increasing NRP content. Wang et al. [19] investigated the brittle-ductile transition of POE toughened HDPE and concluded that the minimum modulus loss of HDPE/POE blends decreased significantly with increasing HDPE molecular weight. Sonnier et al. [20] compatibilized HDPE/NBR blends by using peroxide. The impact energy and elongation at break of the resulting blends were better than those of uncompatibilized blend. Stelescu et al. [16] prepared HDPE/EPDM blends and found that the crystal structure of HDPE in the blends did not change, and the blends containing compatibilizing agent showed increased hardness and elongation at break. In another study, it was found that the storage modulus of HDPE/EVA blends decreased with the increasing EVA content [21].

✉ Changyu Han
cyhan@ciac.ac.cn

[1] Key Laboratory for Comprehensive Energy Saving of Cold Regions Architecture of Ministry of Education, Jilin Jianzhu University, Changchun 130118, China

[2] School of Materials Science and Engineering, Jilin Jianzhu University, Changchun 130118, China

[3] Key Laboratory of Polymer Ecomaterials, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, China

Recently, a propylene-ethylene random copolymer (PEC) with precisely controlled performance and structure was prepared by ExxonMobil Chemical Co. by using special metallocene catalysis. As toughening modifier for olefin polymers, PEC has gradually attracted attention due to its good mechanical properties and processability [22]. Chen et al. [23] studied the influence of PEC with various ethylene-unit contents on the crystallization of isotactic-polypropylene (iPP). It was found that the decline of the crystallization temperature for iPP became smaller with an increase in the ethylene unit content of PEC. In previous work, we prepared PP/PEC blends and found that, as the PEC content was increased, the impact strength of the blends increased, while the viscosity and modulus decreased. Therefore, in this paper, we investigated phase morphology, crystallization behavior, mechanical, and rheological properties of HDPE toughened by PEC elastomer and established the relationship between composition, morphology, and properties. It is hoped that this investigation can contribute to the preparation of new HDPE-based thermoplastic polymers with excellent properties.

Experimental

Materials

HDPE (M20056) with a melt flow index (MFI) of 20 g $(10\ \text{min})^{-1}$ (190 °C, 2.16 kg) was purchased from SABIC, Saudi Arabia. PEC elastomer (Vistamaxx™ 6202) with MFI of 9.1 g $(10\ \text{min})^{-1}$ (190 °C, 2.16 kg) and ethylene content of 15 wt% was supplied by ExxonMobil, USA.

Sample preparation

HDPE and PEC were dried at 80 °C under vacuum for 12 h. HDPE/PEC blends with different PEC contents were prepared by using an internal mixer (LKOI-03, Guangzhou Putong Experimental Analysis Instrument Co., Ltd.) at 160 °C with a screw speed of 60 rpm. The melt compounding time was 10 min. After that, the blended pellets were hot-pressed at 180 °C to a 1-mm thick sample sheet, subsequently cold-pressed at room temperature. HDPE/PEC blends with PEC weight content of 10 wt%, 20 wt%, and 30 wt% were abbreviated as HDPE/PEC-X, where X represented the weight percentage of PEC in the blends.

Characterization

Scanning electronic microscopy (SEM)

The phase morphology of cryo-fractured and impact fracture surface of HDPEL/PEC blends were observed on a field-emission SEM (Sigma 300, ZEISS Co., Germany) with 10 kV accelerating voltage. Before SEM examination, all samples were fractured in liquid nitrogen and then were sputtered with gold. The size and size distribution of dispersed PEC-rich domains in the HDPE/PEC blends were analyzed by nano-measure software.

Rheological measurements

Dynamic shear rheological measurements of neat HDPE and HDPE/PEC blends were carried out on a rotational rheometer (AR2000ex, TA Instruments, USA). The tests were run with a 1-mm gap and 25-mm parallel plate at 180 °C. The viscoelastic properties were determined with strain value of 1.25% and frequency range from 0.05 to 100 rad/s.

Differential scanning calorimetry (DSC)

To examine thermal behaviors and crystallization of HDPE/PEC blends, DSC measurements were carried out on a DSC Q20 (TA Instruments, USA) under a nitrogen atmosphere. All samples (5–8 mg) were heated from 40 to 150 °C with a heating rate of 10 K min^{-1}, held for 2 min at 150 °C to eliminate thermal history, prior to being cooled to 40 °C at a cooling rate of 10 K min^{-1}. Then, the samples were reheated to 150 °C at a heating rate of 10 K min^{-1}. The crystallization temperature (T_c) and melting temperature (T_m) of HDPE/PEC blends were obtained from the first cooling and the second heating thermograms, respectively. The crystallization enthalpy (ΔH_c) and melting enthalpy (ΔH_m) were calculated from the area of crystallization and melting peaks, respectively. The degree of crystallinity (X_c) of HDPE matrix in the blends was estimated by the following:

$$X_c = \frac{\Delta H_m}{\Delta H_m^0 \alpha} \times 100\% \tag{1}$$

where $\Delta H^0{}_m$ is the theoretical melting enthalpy of 100% HDPE crystal, and its value is 287.3 J g^{-1} [24]. α is the mass percentage of HDPE in the blends.

Dynamic mechanical analysis (DMA)

DMA measurements of neat HDPE and HDPE/PEC blends were performed on a dynamic mechanical analyzer (Q800, TA Instruments, USA) in tensile mode. The dimensions of the rectangular sample were 20.0 mm in length, 4.0 mm in width, and 1.0 mm in thickness. The test frequency was 1 Hz, and temperature range was −100 to 100 °C with heating rate of 3 K min^{-1}.

Tensile tests

The tensile properties of HDPE/PEC were determined by using a tensile testing machine based on ISO 527–1:2012 at room temperature. The crosshead speed was fixed at 100 mm/min. Each measurement was repeated 5 times, and the average value was taken as the mechanical properties of the samples and expressed in the form of standard deviation.

Notched Izod impact strength

Notched Izod impact strength tests were carried out on an Izod impact tester (XJUD-5.5, Chengde Jinjian Testing Instrument Co. Ltd., China) at room temperature. Each group of tests was repeated 5 times. The cross section of the samples after impact was observed by SEM.

Results and discussion

Phase morphology

We know that the properties of blends composed of multiphase polymers are highly dependent on the phase structure. Therefore, studying the phase morphology of the blends in the present work is an important method to establish the relationship between morphology and resulting properties. The SEM micrographs of neat HDPE and HDPE/PEC blends are shown in Fig. 1. For neat HDPE in Fig. 1a, obvious smooth and typical continuous phase morphology was observed. For the HDPE/PEC blends, as shown in Fig. 1b–d, PEC phase was uniformly dispersed as spheres in the HDPE continuous matrix. The surface of PEC-rich domains was smooth, and the boundary was clear, indicating that the immiscibility and weak interfacial adhesion between these phases. The average particle size and distribution of PEC-rich domains are shown in Fig. 2. It can be seen from Fig. 2 that as the PEC content increased from 10 to 30 wt%, the average diameter of the PEC-rich domains increased from 1.38 to 3.02 μm. Moreover, the size distribution of PEC-rich domains was gradually broadened with the increase of PEC content. Similar phase morphology development was also observed in the immiscible HDPE/POE [19] and HDPE/EVA [25] blends.

Rheological properties

Melt strength and viscosity are important characterizations of the processing properties of polymers, especially for polymer foaming and blow molding. Thus, it is very important to study the effect of PEC elastomer on the dynamic rheological properties of HDPE. Figure 3a, b, and c show the frequency dependence of storage modulus (G'), loss modulus (G''), and complex viscosity ($|\eta^*|$) of neat HDPE and HDPE/PEC blends at 180 °C. According to Fig. 3a, G′ of the HDPE/PEC blends was higher than that of neat HDPE. The gradual increase in PEC elastomer content from 10 to 30 wt % increased G' of the HDPE/PEC blends within the test frequency range. It was worth noting that in the low frequency region, the increase in G' was more significant. As shown in Fig. 3b, the G'' of the blends was also higher than that of neat HDPE, but its increase with PEC content was small. This may be because G'' behaves as a viscous effect of polymer and was less affected by the relaxation process.

As can be seen from complex viscosity in Fig. 3c, neat HDPE exhibited a Newtonian melt behavior with a Newtonian plateau over the entire range of angular frequency. The complex viscosity of the blends was higher than that of neat HDPE

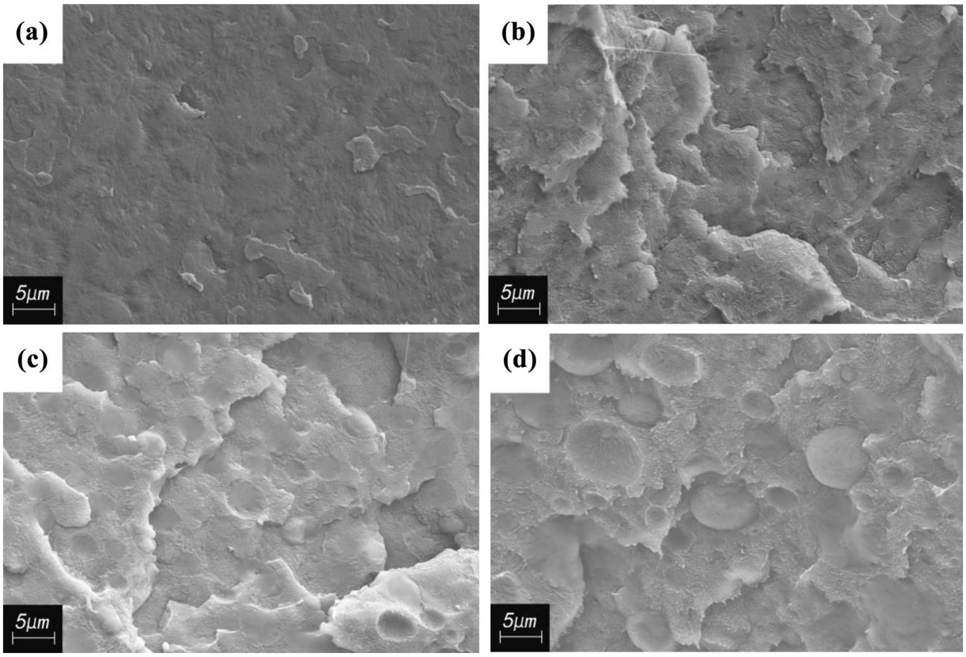

Fig. 1 SEM micrographs of fractured surface of neat HDPE and HDPE/PEC blends: **a** neat HDPE, **b** HDPE/PEC-10, **c** HDPE/PEC-20, and **d** HDPE/PEC-30

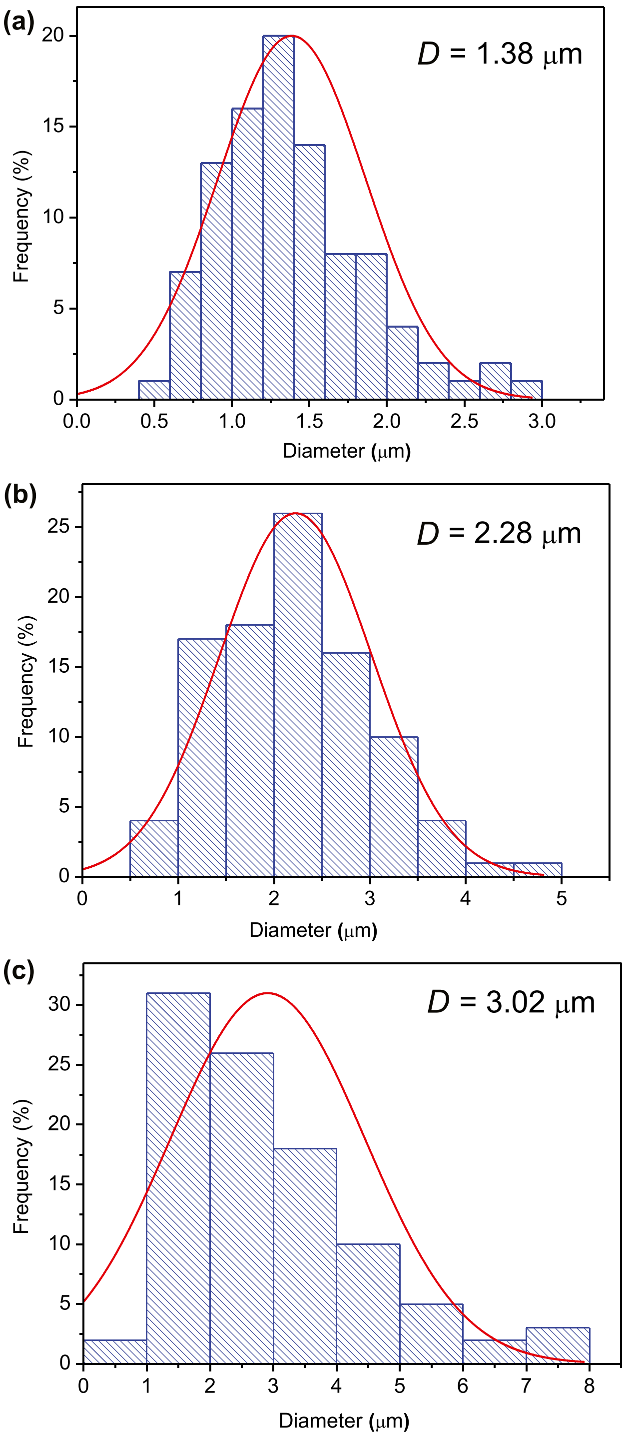

Fig. 2 Quantitative analysis of the PEC-rich domains size: **a** HDPE/PEC-10, **b** HDPE/PEC-20, and **c** HDPE/PEC-30. *D* is the average diameter of the dispersed PEC-rich domains

and increased with the increase of PEC content. In fact, for the blends with PEC content of 20 wt% and 30 wt%, the Newtonian behavior of HDPE disappeared and a significant shear-thinning behavior was observed.

The plots of G' as a function of G'' (Han plot) were also used to analyze the miscibility of the components of the blends, as shown in Fig. 3d. In the low frequency region, the Han curves of the blends have the same slope as the neat polymer, indicating that the two components are miscible, otherwise immiscible [26]. It can be seen from Fig. 3d that with the increase of PEC content, the slopes of Han curves for HDPE/PEC blends in the low frequency region decreased, suggesting immiscibility between HDPE matrix and PEC elastomer. This result was consistent with the observation from SEM and the subsequent dynamic mechanical properties. The Cole–Cole curves (plot of η' against η'') of neat HDPE and HDPE/PEC blends are shown in Fig. 3e. Neat HDPE presented an arc on the Cole–Cole curve, corresponding to the time-dependent relaxation of neat HDPE. However, two half-arcs appeared on the Cole–Cole curves of the HDPE/PEC blends, related to two different relaxation mechanisms of HDPE, local dynamic relaxation and long-range restricted relaxation, respectively. Furthermore, the half-arcs at high-viscosity region became larger with the increase of PEC content, which was indicative of a longer relaxation process of the restrained HDPE chains. This could be attributed to the larger diameter of the dispersed phase with the increase of PEC content, resulting in a longer relaxation process of the PEC phase [27]. In short, the dynamic viscoelastic properties discussed above revealed that PEC elastomer affected the rheological properties of the HDPE/PEC blends. The increase in G' and $|\eta^*|$ of blends melt was beneficial to their processability as it implied the reinforcement of bubble stability and melt strength.

Thermal and crystallization behaviors

HDPE is a typical semi-crystalline polymer, and its degree of crystallinity will highly affect the physical and mechanical properties. Accordingly, the study of the influence of PEC elastomer on the thermal and crystallization behavior of the HDPE matrix is of great importance. Figure 4 shows the DSC cooling and heating thermograms at a rate of 10 K min^{-1} for neat HDPE and the HDPE/PEC blends. The detail results from DSC are listed in Table 1. As can be seen from Fig. 4a, neat HDPE showed a sharp exothermic peak at about 116 °C corresponding to the crystallization of HDPE. The crystallization peak temperature (T_c) of HDPE/PEC blends was about 114 °C. Incorporation of PEC elastomer caused little change in crystallization temperature of HDPE. Because the phases were physically separated in immiscible polymer blends, the heterogeneous structure that could nucleate homopolymer under T_c might nucleate the blend matrix. Consequently, when the polymer blends were cooled from the melt, their T_cs had little difference with that of the neat component [28]. The slight drop in

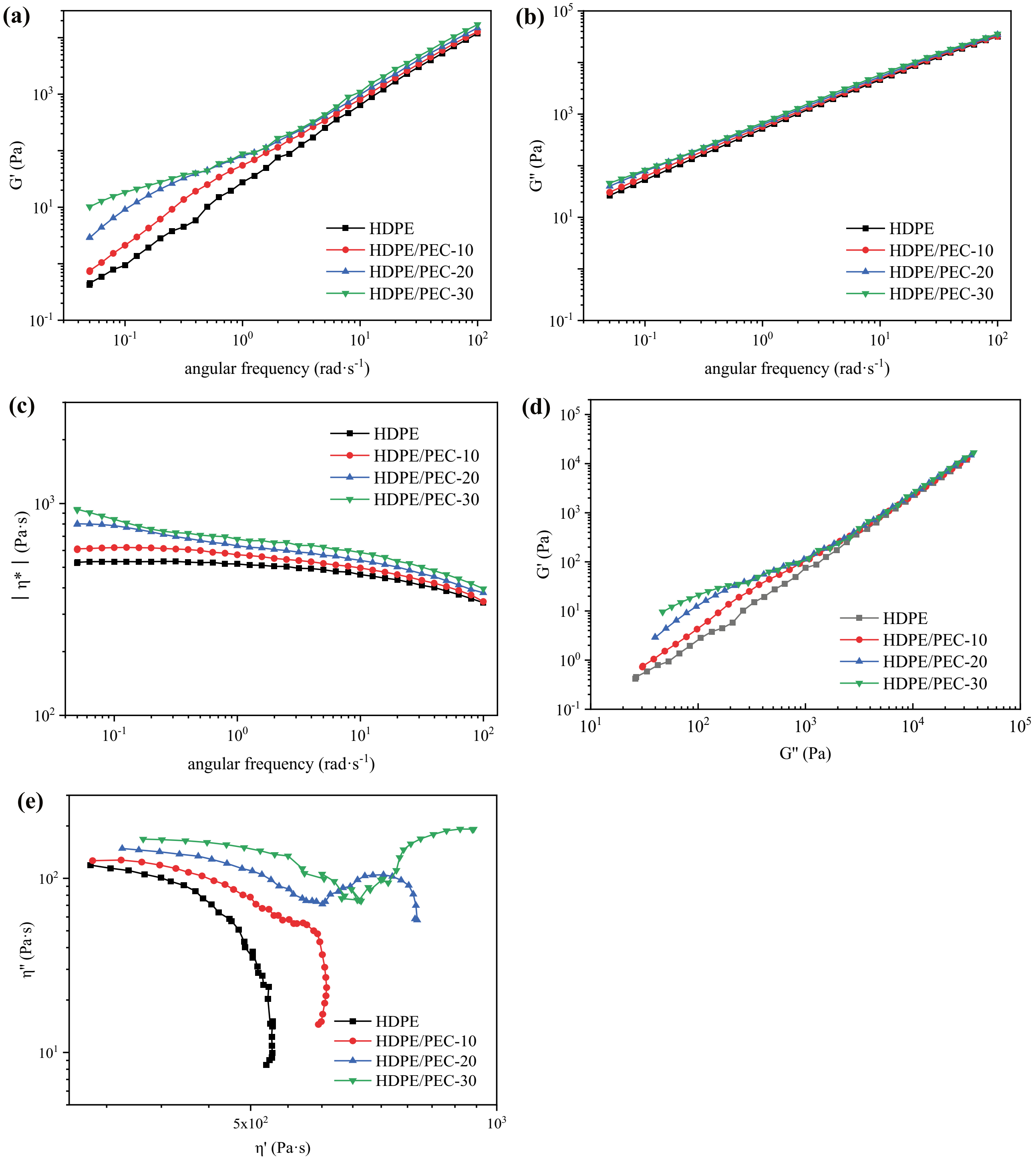

Fig. 3 Angular frequency dependence of **a** storage modulus *G*′, **b** loss modulus *G*″, and **c** complex viscosity |η*| of neat HDPE and HDPE/PEC blends at 180 °C, and **d** Han plots and **e** Cole–Cole plots

T_c of HDPE in the HDPE/PEC blends might be attributed to the migration of heterogeneities from HDPE matrix to PEC-rich domains, where PEC was in the molten state during HDPE crystallization (the melting temperature of PEC was 107 °C [14]). The phenomenon of decreasing T_c has been reported in ethylene–propylene copolymers (EPDM) toughening HDPE blends [29].

From Fig. 4b, an obvious endothermic peak at about 131 °C could be observed for neat HDPE. The melting peak temperature (T_m) of HDPE in the blends was found to rise to about

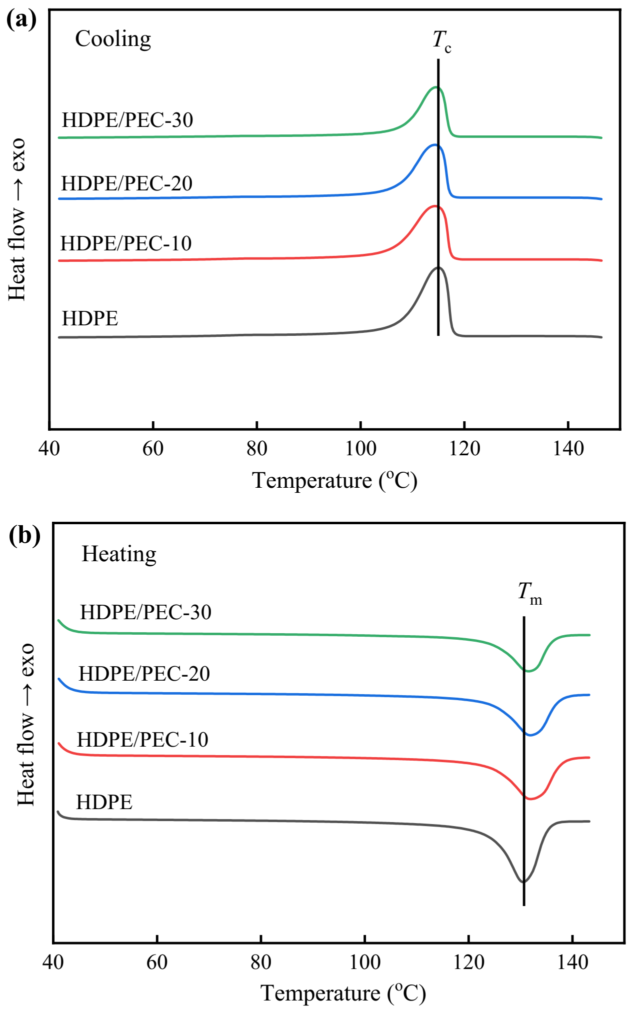

Fig. 4 **a** First cooling and **b** second heating DSC thermograms at rate 10 K min^{-1} for neat HDPE and the HDPE/PEC blends

132 °C. It can be seen that the addition of PEC had little effect on the T_m of HDPE. This was due to the PEC-rich domains did not affect the normal crystallization behavior of the HDPE matrix, attributed to the physical separation of HDPE matrix and PEC-rich domains [28]. In addition, the degree of crystallinity (X_c) of all samples was calculated and listed in Table 1. The X_c of neat HDPE was 67.5%, and the incorporation of PEC elastomer decreased the X_c of HDPE. By increasing PEC elastomer content, the larger droplets of PEC-rich domains were formed due to coalescence of droplets, as shown in Fig. 2. As a result, the gradual coarsening PEC-rich domains inhibited the growth of HDPE crystals, leading to the decline of the X_c of HDPE in the blends. Although the changes of crystallization and melting behavior of the HDPE/PEC blends were not significant, they also had influence on the mechanical properties of HDPE to some extent.

Table 1 Thermal properties of neat HDPE and the HDPE/PEC blends obtained from the DSC

Samples	First cooling		Second heating		
	T_c (℃)	ΔH_c (J·g^{-1})	T_m (℃)	ΔH_m (J·g^{-1})	X_c (%)
HDPE	116.42	183.2	130.54	194.0	67.5
HDPE/PEC-10	114.35	171.1	132.01	188.7	65.6
HDPE/PEC-20	114.33	178.0	131.95	182.8	63.6
HDPE/PEC-30	114.53	174.4	131.59	179.3	62.3

Dynamic mechanical properties

Figure 5a shows the storage modulus (E') plots of neat HDPE and HDPE/PEC blends. The E' of neat HDPE decreased gradually with the increase of temperature in the range of temperature from −100 to 100 °C. For neat PEC, at its glass transition temperature (T_g) of about −20 °C, the E' decreased sharply.

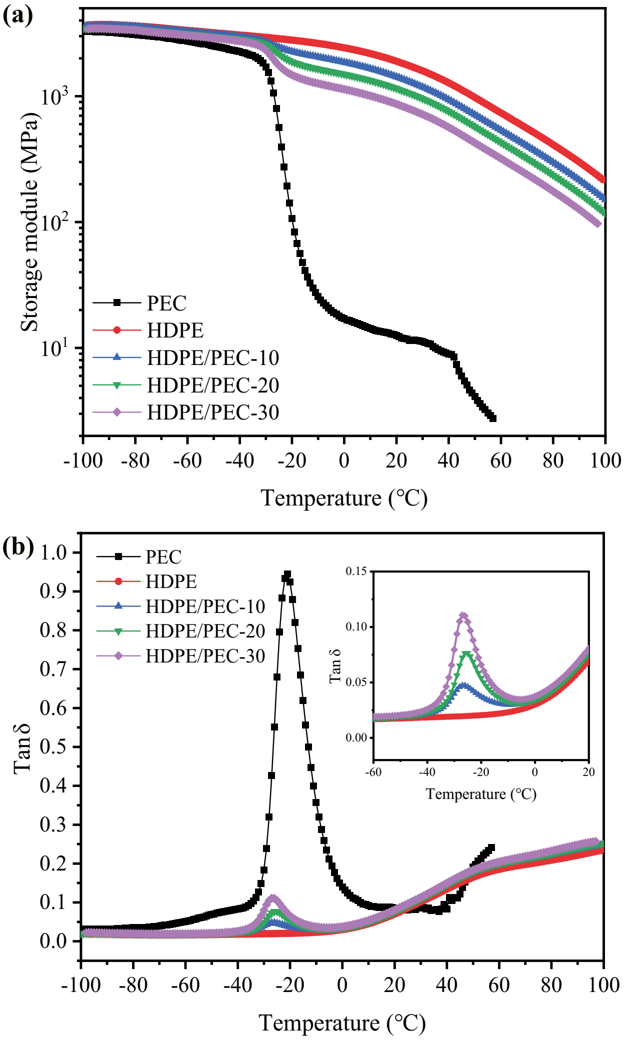

Fig. 5 **a** Dependence of storage modulus (E') and **b** damping factor (tan δ) on the temperature for neat HDPE and the HDPE/PEC blends

The E' of PEC exhibited a slightly lower value than that of HDPE at temperatures below T_g of PEC. When the temperature was raised to room temperature, the E' of PEC elastomer decreased more than 2 orders of magnitude, reaching about 13 MPa. In the case of HDPE/PEC blends, the PEC content had little influence on the E' of the blends at temperature below T_g of PEC. This was attributed to the fact that both HDPE and PEC were glassy. However, above the T_g of PEC elastomer, HDPE/PEC blends with higher content of PEC exhibited lower E' attributed to low E' of PEC compared to HDPE.

Tan delta (tan δ), the ratio of loss modulus to storage modulus, of neat HDPE and HDPE/PEC blends was shown in Fig. 5b. On the tan δ curve of neat PEC, a sharp peak corresponding to the glass transition was located at about −20 °C. For the HDPE/PEC blends of different PEC contents, a single damping peak at about −25 °C was visible in the tan δ curves, corresponding to the glass transition of PEC elastomer. Moreover, the T_g of PEC elastomer in the blends hardly changed with the increase of the PEC content. These results suggested that PEC elastomer was not thermodynamically miscible with HDPE, which was in agreement with the results of SEM and rheological tests. However, the T_g of PEC elastomer in the blends was shifted towards lower temperatures compared with neat PEC. The depression in T_g of PEC elastomer in the blends could be attributed to the existence of the phase interface originated from immiscibility of HDPE matrix and PEC elastomer. The state of polymer chain near the interface was different from that of the bulk in the heterogeneous blend system. The excess free volume ascribed to the aggregation of small molecules, short chains, and molecular chain end groups at the interface could improve the mobility of polymer chains and segments at the interface, resulting in a decrease in the T_g [30, 31].

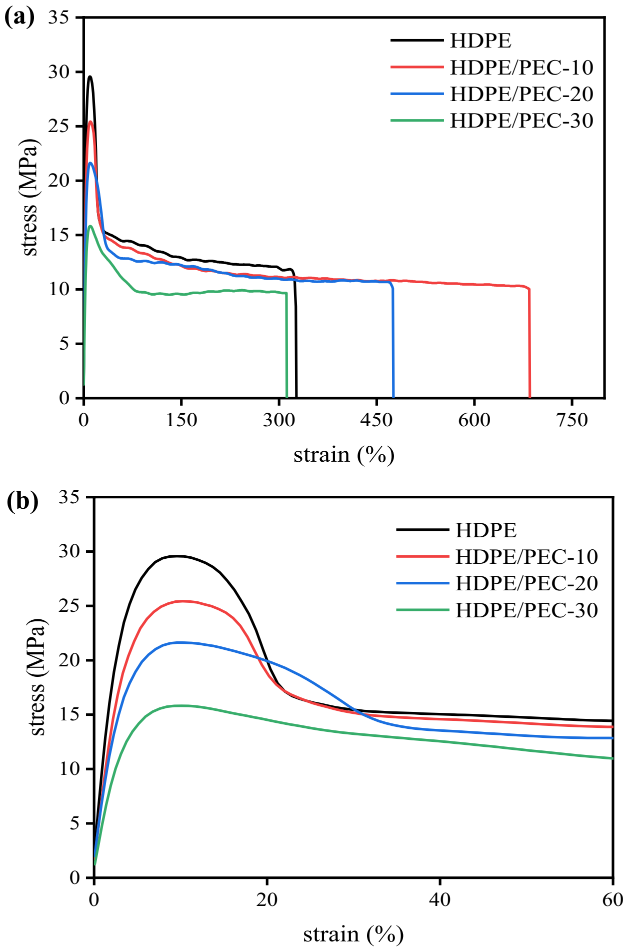

Fig. 6 **a** Tensile stress – strain curves of neat HDPE and the HDPE/PEC blends. **b** Details of stress–strain curves at low strain

Tensile properties

Figure 6 shows the strain–stress curves of neat HDPE and HDPE/PEC blends. The relevant mechanical properties are summarized in Table 2. As can be seen in Fig. 6a, neat HDPE exhibited a significant yield point and a stable necking process and behaved in soft manner with yield strength of 30.9 MPa, elongation at break of 326%, and Young's modulus of 899 MPa. With the increase of PEC content, yield strength and Young's modulus of the HDPE/PEC blends decreased as expected, whereas the elongation at break increased significantly compared to neat HDPE. When PEC content was 10 wt%, yield strength and Young's modulus of the blend were 25.3 and 698 MPa, respectively, which were lower 18.1% and 22.3% than those of neat HDPE. However, the elongation at break was increased from 326 to 685%, a 110% improvement compared to neat HDPE. The reduction of Young's modulus of blends with the increase of PEC content was attributed to the introduction of PEC soft phase to the HDPE matrix.

It can be determined that phase morphology and interfacial strength between dispersed and matrix phase are the

Table 2 Tensile properties of neat HDPE and HDPE/PEC blends

Sample	Young's modulus (MPa)	Yield strength (MPa)	Breaking strength (MPa)	Elongation at break (%)
HDPE	899.3 ± 65.3	30.9 ± 0.3	11.2 ± 2.6	325.9 ± 47.4
HDPE/PEC-10	697.9 ± 23.8	25.3 ± 0.2	10.0 ± 2.8	684.6 ± 56.2
HDPE/PEC-20	586.9 ± 48.0	21.1 ± 0.6	10.6 ± 0.6	474.9 ± 13.4
HDPE/PEC-30	411.6 ± 13.7	15.9 ± 0.3	9.6 ± 2.7	311.5 ± 9.9

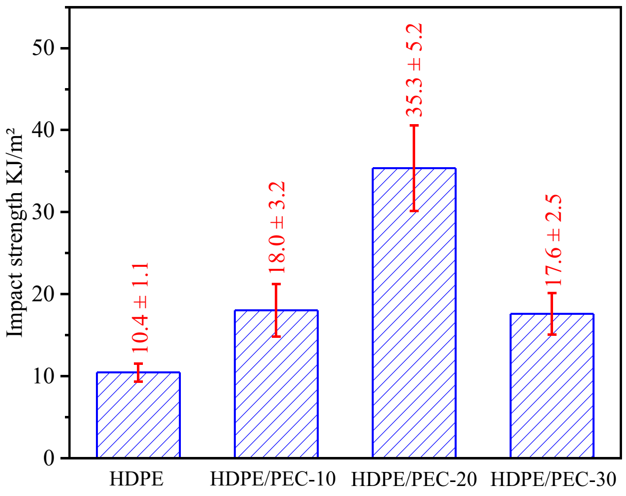

Fig. 7 Impact strength of neat HDPE and the HDPE/PEC blends

main factors affecting yield strength [32]. The PEC-rich domains in the blends acted as stress concentration points due to the different elastic performance of HDPE from that of PEC elastomer. When the tensile stress was applied to the samples in the tensile test, the PEC-rich domains were debonded from HDPE matrix, resulting in large interfacial microvoids in the blends. The interface microvoids caused the triaxial stress state of HDPE matrix around the pores to be reduced, thus producing a stress state conducive to inducing multiple matrix shear yield [33]. The higher the PEC content in the HDPE/PEC blends, the bigger the dispersed phase size, and as a result, the easier it was to debond at the particle – matrix interface on account of insufficient interfacial adhesion. Consequently, the yield strength would decrease as the PEC content in the blends was increased. Additionally, the elongation at break of the HDPE/PEC blends decreased with the increasing PPC content. This result was also caused by coarsening of PEC-rich domains.

Impact strength

From the point of view of industry and academia, the improvement of impact strength, like tensile strength, is also very important and has become the subject of a lot of research work. The notched Izod impact strength of all samples is shown in Fig. 7. The impact strength of neat HDPE was only about 10.4 kJ m^{-2}, and the introduction of PEC elastomer increased the impact strength of HDPE. The increase of the PEC content in the HDPE/PEC blends from 10 to 20 wt % improved the impact strength of the resulting blends. When the PEC content was increased to 30%, the impact strength of the blend was decreased. The blend containing 20 wt% PEC had the highest impact strength. The impact strength of HDPE/PEC blend with 20 wt% PEC elastomer was about 35.3 kJ m^{-2}, which was more than 3 times higher than that of HDPE.

As we all know, toughness means energy absorption, which can be achieved by adding granular second flexible phase. After the cavitation process, the dispersed phase particles cause a large degree of stress concentration, resulting in a wide range of shear deformation, which is a high energy absorption mechanism [34]. In order to further investigate the toughening mechanism of the HDPE/PEC blends, the cross-section morphology of impact samples was studied by SEM and then is shown in Fig. 8.

The fracture surface morphology can provide direct information about crack propagation stability and deformation

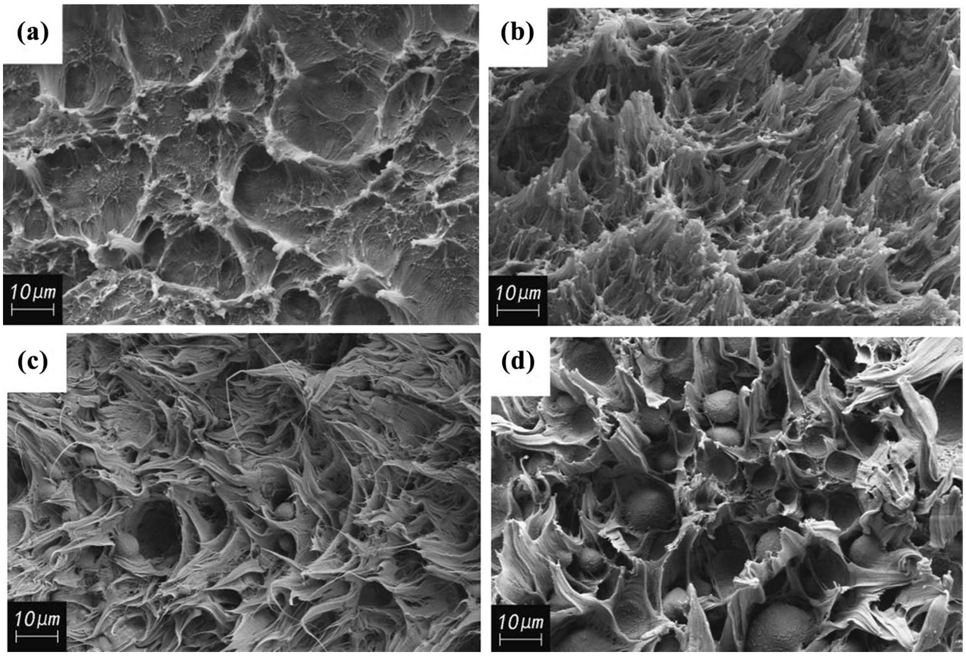

Fig. 8 SEM images of impact-fracture surface of neat HDPE and the HDPE/PEC blends: **a** neat HDPE, **b** HDPE/PEC-10, **c** HDPE/PEC-20, and **d** HDPE/PEC-30

mechanism. Although neat HDPE showed a relatively coarse fracture surface, it had no much deformation, corresponding to HDPE's low impact strength. With the addition of PEC elastomer, rough surface structure and many long stretches of fibrils caused by plastic deformation were observed. With the increase of PEC content to 20 wt%, the fracture surface morphology became more rugged and the fibers became thinner and longer, suggesting large matrix shear yielding. Such large plastic deformation could effectively dissipate the impact energy, leading to significantly improved impact strength [35]. Such fibrillated structure had been observed in other elastomer toughened polymers [36, 37]. When PEC content was up to 30 wt %, the fiber structure coarsened obviously and consisted of less deformed material. The larger dispersed PEC-rich domains were clearly visible. The large PEC-rich domains and weak interfacial interactions triggered the unstable crack propagation of the blend with 30 wt% PEC, thus reduced the impact strength.

Conclusions

In this study, PEC elastomer-toughened HDPE blends were successfully prepared by melt mixing with different PEC contents. The morphology, rheological properties, thermal and crystallization behaviors, and mechanical properties of HDPE/PEC blends were systematically investigated. SEM result showed a phase-separated morphology and the increased size of dispersed PEC-rich domains with the PEC content, indicating that HDPE and PEC were immiscible. Rheological measurements revealed that the addition of PEC elastomer improved the melt elasticity and viscosity of HDPE. It can be seen from the DSC results that the presence of PEC elastomer inhibited the nucleation of HDPE and reduced the degree of crystallinity. Dynamic mechanical property analyses suggested that HDPE/PEC blends with higher content of PEC exhibited lower E' due to low E' of PEC compared to HDPE. Mechanical analysis revealed that PEC elastomer improved remarkably the elongation at break and impact strength with reducing yield strength and elastic modulus of the HDPE matrix. The SEM micrographs of the impact-fractured surfaces of the blends revealed that PEC-rich domains acted as stress concentrators, and a large amount of shear yielding of HDPE matrix was the main way of absorbing and dissipating energy during the impact test.

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Declarations

Conflict of interest The authors declare no competing interests.

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