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ORIGINAL PAPER

Flexural and impact properties of POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites

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Abstract The Polyformaldehyde (POM)/ethylene–vinyl acetate copolymer (EVA)/high-density polyethylene (HDPE) blends and POM/EVA/HDPE composites filled with nanometer calcium carbonate (nano-$CaCO_3$) were prepared using a twin-screw extruder. The effects of the HDPE weight fraction on the flexural and impact properties of the blends and composites were investigated at room temperature. The results showed the flexural modulus and strength of the blends and composites decreased slightly with increasing the HDPE weight fraction. The values of the flexural modulus of the composites were higher than those of the blends, while the values of the impact fracture strength of the blends were higher than those of the composites under the same HDPE weight fraction. This indicated that the impact fracture toughness of the POM could be improved with the HDPE, while the flexural stiffness of the POM blends could be enhanced with the nano-$CaCO_3$ to some extent.

Keywords Polymer-matrix composites · POM · HDPE · EVA · Flexural properties · Impact fracture

Introduction

Polyformaldehyde (POM) is an engineering resin and is used widely in many fields, such as in construction, transportation, electronic applications, and in general household materials due to its favorable performance in processing including high strength and stiffness, good dimensional stability, excellent corrosion resistant, wear

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resistant, self-lubricating and anti-creep [1–7]. However, some shortcomings such as low notch sensitivity, bad heat resistance and a high price hinder its application scope. Therefore, how to modify POM resin to expand its applications has been one of the research hot-points in recent years. To improve the physical and mechanical properties of resins, blending with other resins or filling with inorganic particles is one of the important modification methods for polymer materials [8, 9].

High-density polyethylene (HDPE) is a comprehensive resin with high performance in processing and low price, hence it has also a wide range of industrial applications such as automobiles, electronic appliances, vessels and tube, and spinning and film [10, 11]. However, the compatibility between POM and HDPE is not good under general conditions, and it is necessary to improve their compatibility. Ethylene–vinyl acetate copolymer (EVA) is a thermoplastic resin made of ethylene monomer and vinyl acetate monomer; it has wide range of applications used for adhesives, blow molding, wire and cable manufacturing, foam products, extrusion, lamination, hot melt, as well as injection molding products mainly due to its excellent flexibility, impact resistance, optical transparency, adhesion, environmental stress cracking resistance, weather resistance, chemical resistance, sealing ability, and good compatibility [12]. Because the compatibility between POM and HDPE is poor, EVA is usually chosen as the compatibilizer for POM/HDPE blends.

Nanometer calcium carbonate (nano-$CaCO_3$) is a kind of new high-grade functionality filler with low cost, which is widely used in rubber, plastics, paint and other industrial fields [13]. POM can be modified by blending with HDPE and filling with nano-$CaCO_3$ to fabricate a new kind of polymeric composite material with good mechanical and processing properties and low cost.

In the past few decades, the studies on many aspects of POM composites have been extensively reported, including the phase morphology [14], mechanical properties [14–16], friction [17], crystallization [18, 19], processing [7], etc. Unfortunately, there have been few reports on the impact and flexural properties of POM/HDPE/EVA nanocomposites during the past 20 years. More recently, Liang and his colleagues [20, 21] studied the melt extrudate swell and flow properties during capillary extrusion of POM/EVA/HDPE nanocomposites, and found some interesting phenomenon. The objectives of this paper are to investigate the effects of HDPE content on the impact and flexural properties of the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites to understand mechanical behavior as well as the reinforcing and toughening mechanisms of the blends and the nanocomposites under experimental conditions.

Experimental

Raw materials

The POM with trademark of ZPR supplied by Baotailing Engineering Plastics Co., Ltd. (Nantong city, China) was used as the continuous resin in this work. The density in the solid state and the melt index (MI) of the resin were 1,410 kg/m^3 and

20 g/10 min, respectively. The HDPE with trade mark of 5000S, supplied by Petro China Daqing Petrochemical Company Ltd. (Daqing city, China) was used as the dispersive phase resin, and the density in the solid state and the MI of the resin were, respectively, 954 kg/m^3 and 0.9 g/10 min.

The EVA with VA (vinyl acetate) content of 14 % was used as compatibilizer, produced by Beijing Dongfang Petrochemical Co., Ltd (Beijing, China). The MI and density in solid state were 2 g/10 min and 935 kg/m^3, respectively. The nano-$CaCO_3$ with trade mark of CC-A supplied by Anyuan Science and Technology Chemical Industrial Co. Ltd (Jiangxi province, China) was used as the filler. The mean diameter and the density of the particle were 40 nm and 2,500 kg/m^3, respectively. The particle surface was pretreated with stearic acid.

Fabrication

First, the POM resin was mixed with EVA, HDPE (the mass ratio between EVA and HDPE was 1:10) and the nano-$CaCO_3$ with 3 phr (per hundred parts of POM resin by weight) in a high speed compounder with model of SHR-10A, and then the mixtures were molten-blended in a co-rotation twin-screw extruder (model SHJ-SC-58) under conditions of temperature ranging from 170 to 180 °C and screw speed of 100 rpm, the extrudate of the POM/EVA/HDPE blends and the POM/EVA/HDPE/nano-$CaCO_3$ composites was granulated. The weight fraction (ϕ_{HDPE}) of HDPE were 3, 6, 9, 12 and 15 %, respectively. The diameter and length–diameter ratio of the screw were 35 mm and 40, respectively. Finally, pellets of the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites were dried for 4 h at 80 °C before the specimens for impact test and flexural test were fabricated by means of a plastics injection machine (model SLQ100-T), and the injection temperature range was from 200 to 220 °C.

Apparatus and methodology

The flexural properties of the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites were measured at room temperature by means of a universal materials testing machine (model CMT4104) supplied by Newsans Co. Ltd. (Shenzhen, China). The tests were conducted at the crosshead speed of 2 mm/min. The impact tests including Izod impact tests and Charpy impact tests were conducted at room temperature using the LCD type plastic pendulum impact-testing machine with model of PIT501B-2 supplied by Wance test equipment limited company (Shenzhen, China). Each group specimens contained 5 pieces, and the average values of the measured mechanical properties were used from the measured data.

The fracture surfaces of the specimens from the impact tests were examined by means of a scanning electron microscope (SEM, model S-3700N) supplied by Hitachi Co. Ltd. (Tokyo, Japan) to observe the impact fracture surface, interfacial debonding, interlayer structure morphology, and the dispersion or distribution of the dispersion phase or nano-$CaCO_3$ particles in the POM resin. The specimens were gold coated before SEM examination.

Results and discussion

Flexural curves

Figure 1 shows the curves of the flexural stress versus the flexural strain of the POM/EVA/HDPE blends. It can be seen that the flexural stress increases with increasing the flexural strain when the flexural strain is lower than 7 %, and the maximum flexural stress for the blends is reached at a flexural strain about 7 %; and then the flexural stress increases with decreasing the flexural strain. Moreover, the maximum flexural stress decreases relatively obviously with increase of the HDPE weight fraction (ϕ_{HDPE}). This indicates that the HDPE resin is not beneficial to improve the flexural strength of the blends under these experimental conditions. This is because that the flexural strength of the HDPE resin is lower than that of the POM resin, leading to reduction of the blends with increasing the HDPE content.

Figure 2 displays the curves of the flexural stress and strain of the POM/EVA/HDPE/nano-$CaCO_3$ composites. Similar to the results shown in Fig. 1, the flexural stress of the composites increases with increasing the flexural strain when the flexural strain is lower than 7 %. In addition, the maximum flexural stress decreases relatively obviously with increase of the HDPE content; when the flexural strain is about 7 %, the flexural stress reaches the maximum value, and then the flexural stress increases with decreasing the flexural strain. The reason could be that the flexural strength of the HDPE resin is lower than that of the POM resin, resulting in reduction of the blends with increasing the HDPE content, even though the flexural strength can be improved somewhat by the nano-$CaCO_3$ particles.

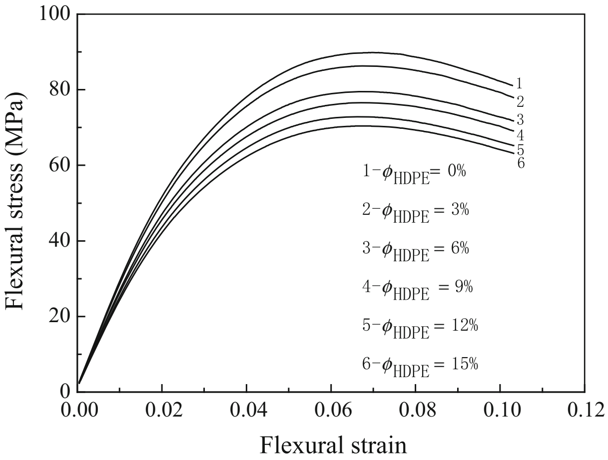

Fig. 1 Flexural stress–strain curves of POM/EVA/HDPE blends

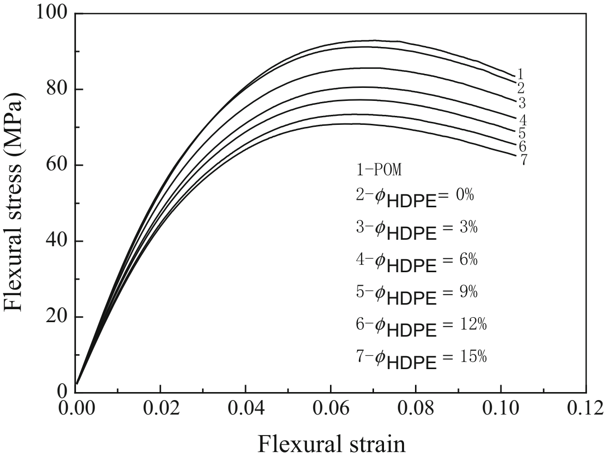

Fig. 2 Flexural stress–strain curves of POM/EVA/HDPE/nano-CaCO$_3$ composites

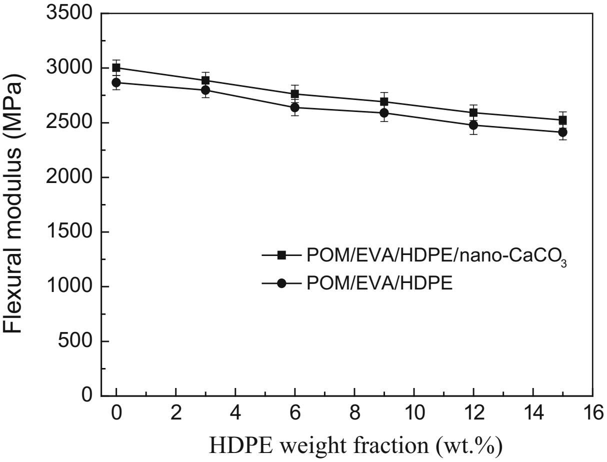

Fig. 3 Relationship between Flexural modulus and HDPE weight fraction

Dependence of flexural modulus on HDPE content

Figure 3 illustrates the dependence of the flexural modulus on the HDPE weight fraction for the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-CaCO$_3$ composites. With the increase of the HDPE weight fraction, the flexural modulus decreases nonlinearly, this is because that the flexural stiffness of the HDPE resin is lower than that of the POM resin, leading to decreasing the flexural modulus of the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-CaCO$_3$ composites with an addition of the HDPE concentration. This is similar to the results from the electrospun polyoxymethylene/polyurethane blend fiber mats [14]. Furthermore, when the HDPE weight fraction is constant, the flexural modulus of the POM/EVA/HDPE blends is lower than that of the POM/EVA/HDPE/nano-$CaCO_3$ composites. It indicates that nano-$CaCO_3$ is beneficial to improve the flexural stiffness of the POM/EVA/HDPE blends, even though in the case of low concentration of the nano-$CaCO_3$. This is because that the nano-$CaCO_3$ particles could play the role of skeleton material in the POM/HDPE matrix, and they could block the movement of the macromolecular chains of the POM and HDPE resins. Consequently, the flexural stiffness of the composites is improved correspondingly.

Relationship between flexural strength and HDPE content

Flexural strength is an important parameter for characterizing the functional performance of materials. Figure 4 presents the relationship between the flexural strength and the HDPE weight fraction for the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites. It can be seen that the flexural strength decreases nonlinearly with increasing the HDPE weight fraction; when the HDPE weight fraction is constant, the flexural strength of the POM/EVA/HDPE blends is lower than that of the POM/EVA/HDPE/nano-$CaCO_3$ composites, and the difference between the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites is very small. The reason should be that the flexural strength of the HDPE resin is lower than that of the POM resin, leading to reduction of the flexural strength of the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites with increasing the HDPE weight fraction. This is similar to the results found by Wang and Cui [15], that was, the tensile strength of polyoxymethylene/methyl methacrylate–styrene–butadiene copolymer blends decreased with increasing the ionomers weight fraction.

It is generally believed that the mechanical properties of crystalline polymers are related closely to the crystallization and crystalline type [22–27]. POM is a type of

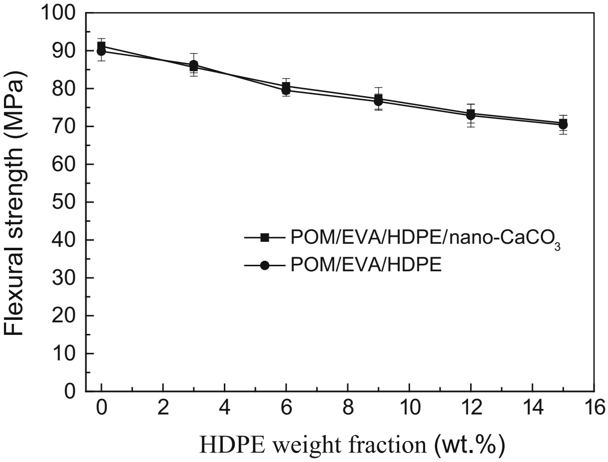

Fig. 4 Correlation between flexural strength and HDPE weight fraction

linear chain resin with no side chain, high density and high crystallinity. The mechanical properties of the POM blends and POM composites could be weakened if their crystallization properties and crystal type are not improved by the components or fillers. The flexural modulus and flexural strength decrease with increasing the HDPE weight fraction, therefore, it means that the crystallization behavior and the crystal type for the POM blends and composites are not improved by the HDPE [18, 19].

Dependence of impact strength on HDPE content

Figure 5 displays the correlation between the V-notched Charpy impact strength (σ_{VCI}) and the HDPE weight fraction for the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites. With an addition of the HDPE weight fraction, the V-notched Charpy impact strength decreases slightly for both the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites; when the HDPE weight fraction is less than 9 %, the V-notched Charpy impact strength of POM/EVA/HDPE blends is higher than that of the POM/EVA/HDPE/nano-$CaCO_3$ composites, and then the difference in the V-notched Charpy impact strength between the blends and the composites decreases with increasing the HDPE weight fraction. The reason could be that the nano-$CaCO_3$ particles could form some defects in the POM matrix; these defects should generate cracks and develop under impact load; and the notch sensitivity should increase, leading to weakening of the impact fracture toughness of the composites.

Figure 6 shows the dependence of the unnotched Izod impact strength (σ_{II}) on the HDPE weight fraction for the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites. For the POM/EVA/HDPE blends, the unnotched Izod impact strength increases when the HDPE weight fraction is less than 6 %, and then

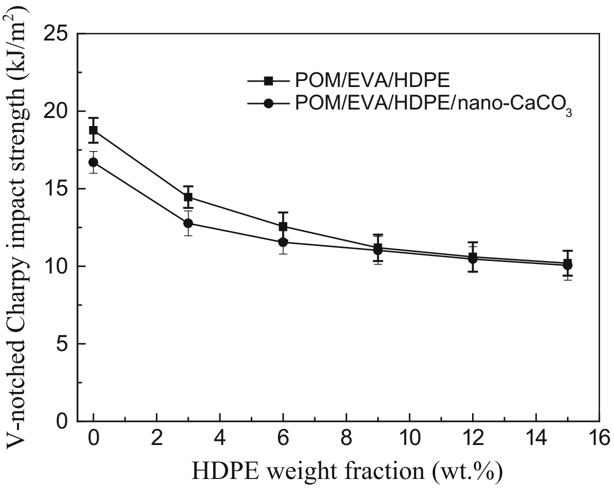

Fig. 5 Dependence of V-notched Charpy impact strength on HDPE weight fraction

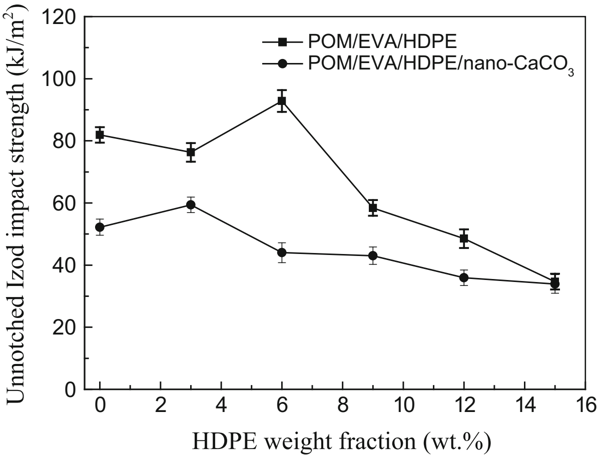

Fig. 6 Dependence of unnotched Izod impact strength on HDPE weight fraction

it decreases nonlinearly with increasing the HDPE weight fraction. For the POM/EVA/HDPE/nano-$CaCO_3$ composites, the unnotched Izod impact strength increases when the HDPE weight fraction is less than 3 %, and then it decreases nonlinearly with increasing the HDPE weight fraction. This indicates that there is certain toughening effect for the blends and composites in the case of low HDPE concentration. Moreover, the unnotched Izod impact strength of the POM/EVA/HDPE blends is higher than that of the POM/EVA/HDPE/nano-$CaCO_3$ composites under the same conditions, and the difference in the variation of the values of the impact strength with the HDPE weight fraction between the blends and the composites could be attributed to that the introduction on the HDPE resin and the nano-$CaCO_3$ would increase the notch sensitivity of the POM blends and POM composites.

Figure 7 illustrates the dependence of the V-notched Izod impact strength (σ_{VII}) on the HDPE weight fraction for the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites. It can be seen that the notched Izod impact strength decreases nonlinearly with increasing the HDPE weight fraction for the POM/EVA/HDPE blends; for the POM/EVA/HDPE/nano-$CaCO_3$ composites, the notched Izod impact strength decreases nonlinearly when the HDPE weight fraction is less than 6 %, and then it increases with increasing the HDPE weight fraction. Similarly, the notched Izod impact strength of the POM/EVA/HDPE blends is higher than that of the POM/EVA/HDPE/nano-$CaCO_3$ composites except to individual data point.

Moreover, both the notched Charpy impact strength and notched Izod impact strength for the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites decrease nonlinearly with an addition of the HDPE weight fraction; and the notched Charpy impact strength is higher than that of the notched Izod impact strength under the same HDPE weight fraction (see Figs. 5, 7), it should be attributed to the difference in load form between the two tests. This is because that the specimen is undergone shear load during Izod impact testing, while the

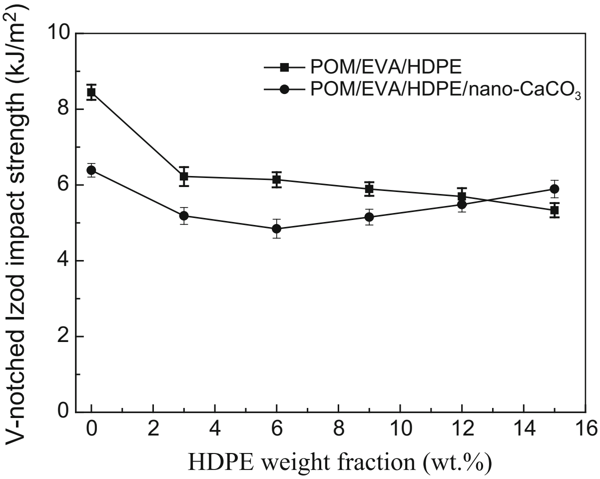

Fig. 7 Dependence of V-notched Izod impact strength on HDPE weight fraction

specimen is undergone flexural load during Charpy impact testing; the capacity undergoing flexural load is usually higher than that of undergoing shear load for most polymeric materials. In addition, the unnotched Izod impact strength increases in the case of low concentration of the HDPE resin, it should be attributed to the high notched sensibility of the POM and HDPE resins.

Morphology

It is generally believed that the morphology of impact fracture surface reflects, in a certain degree, the toughness of materials. Figure 8 is the SEM photograph of the

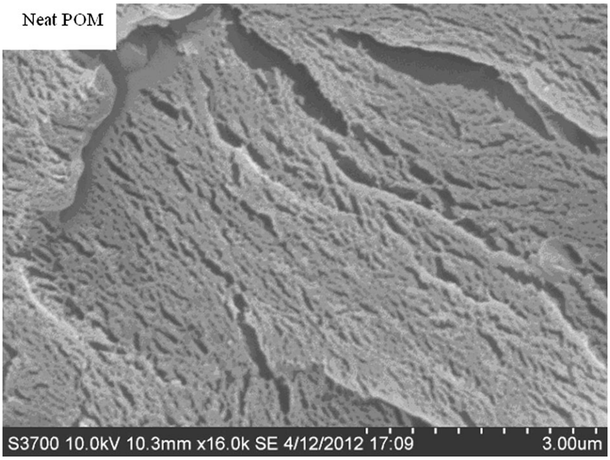

Fig. 8 SEM photograph of impact fracture surface of neat POM nesin

impact fracture surface of the neat POM resin. It can be seen that the fracture surface is rough, and there are some platforms on the surface. This indicates that the impact fracture of the POM resin is roughly ductile fracture, and the notch sensitivity is low, thus the impact fracture strength is relatively high comparing with the blends and composites (see Figs. 5, 6, 7).

Figure 9 is the SEM photograph of the impact fracture surface of the POM/EVA/HDPE blend with ϕ_{HDPE} of 3 %. It can be observed that the dispersion phase resin (EVA) distributes uniform in the continuous phase resin (POM), and the interfacial layer between the continuous phase and dispersive phase is not clear. This presents that the compatibility between them is relatively good. Moreover, the fracture surface is relatively smooth, leading to the impact fracture strength decreases (see Figs. 5, 6, 7). Figure 10 is the SEM photograph of the impact fracture surface of the POM/EVA/HDPE/nano-$CaCO_3$ composite with ϕ_{HDPE} of 3 %. Similarly, the dispersion phase resins (EVA and HDPE) and the nano-$CaCO_3$ particles distribute uniform in the continuous phase resin (POM), and the interfacial layer between them is not clear. Furthermore, the fracture surface is rougher than that of the blend with ϕ_{HDPE} of 3 % shown in Fig. 9. The reason should be that the matrix around the nano-$CaCO_3$ particles will generate the stress concentration under impact load, it will produce shear yield to form relevant plastic deformation, leading to rough fracture surface. On the other hand, the nano-$CaCO_3$ particles might form some defects in the POM matrix; these defects should generate cracks and develop under impact load; and the notch sensitivity increases, leading to weakening of the impact fracture toughness of the composites. As a result, the impact fracture strength of the blend is higher than that of the composite under the same conditions (see Figs. 5, 6, 7).

Figure 11 is the SEM photograph of the impact fracture surface of the POM/EVA/HDPE blend with ϕ_{HDPE} of 6 %. The dispersion phase resins (HDPE and EVA) distribute roughly uniform in the continuous phase resin (POM), and the

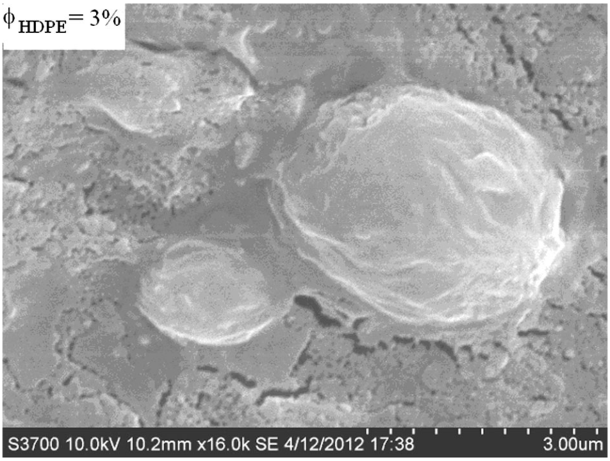

Fig. 9 SEM photograph of impact fracture surface of POM/EVA/HDPE blends ($\phi_{\mathrm{HDPE}} = 3$ %)

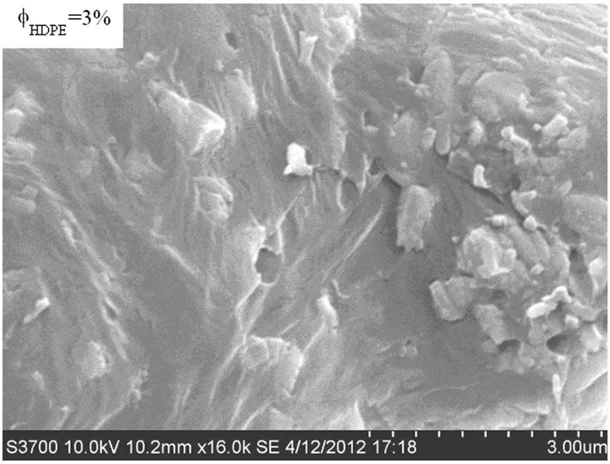

Fig. 10 SEM photograph of impact fracture surface of POM/EVA/HDPE/nano-$CaCO_3$ composites ($\phi_{HDPE} = 3$ %)

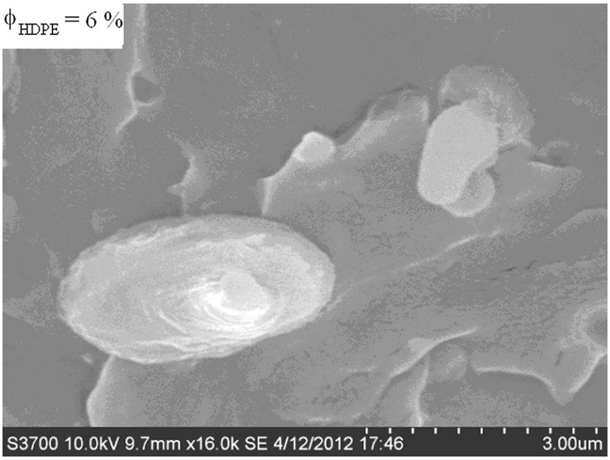

Fig. 11 SEM photograph of impact fracture surface of POM/EVA/HDPE blends ($\phi_{HDPE} = 6$ %)

interfacial layer between them is not clear. Moreover, the fracture surface is relatively rough; the reason should be that the dispersion phase HDPE would yield first due to its low modulus under impact load, to produce large shear deformation to absorb relevant impact deformation energy, resulting in improving significantly the impact toughness of the blend (see Fig. 6). Figure 12 is the SEM photograph of the impact fracture surface of the POM/EVA/HDPE/nano-$CaCO_3$ composite with ϕ_{HDPE} of 6 %. It can be seen that there is somewhat aggregation of the dispersion phase in the continuous phase, and the fracture surface is smoother than that of the POM/EVA/HDPE/nano-$CaCO_3$ composite with ϕ_{HDPE} of 3 %. As stated above, the

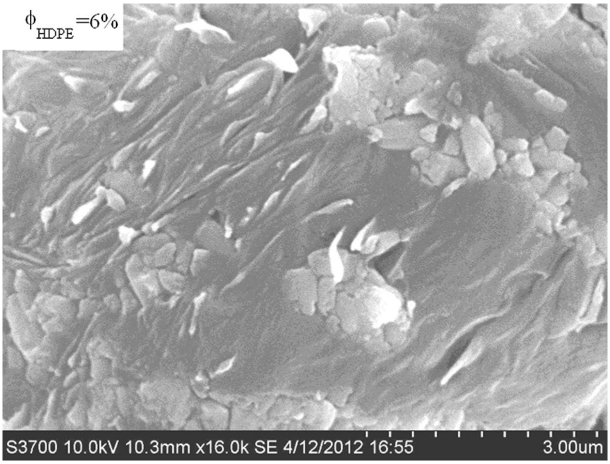

Fig. 12 SEM photograph of impact fracture surface of POM/EVA/HDPE/nano-CaCO$_3$ composites ($\phi_{\mathrm{HDPE}} = 6$ %)

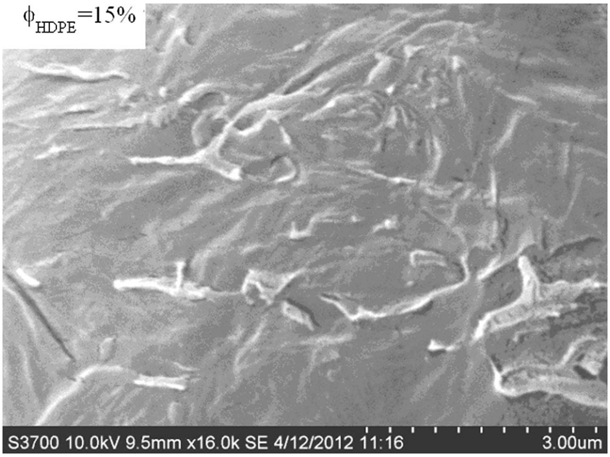

Fig. 13 SEM photograph of impact fracture surface of POM/EVA/HDPE blends ($\phi_{\mathrm{HDPE}} = 15$ %)

nano-CaCO$_3$ particles might form some defects in the POM matrix; these defects should generate cracks and develop under impact load, leading to weakening of the impact fracture toughness. Therefore, the impact fracture strength of the composite decreases correspondingly.

Figure 13 is the SEM photograph of the impact fracture surface of the POM/EVA/HDPE blend with ϕ_{HDPE} of 15 %. It can be observed that the fracture surface is smoother than that of the POM/EVA/HDPE blend with ϕ_{HDPE} of 6 %, and the dispersive phase HDPE resin is extended as ribbons. The reason should be that the sea–island structure between the continuous phase and dispersion phase is difficult

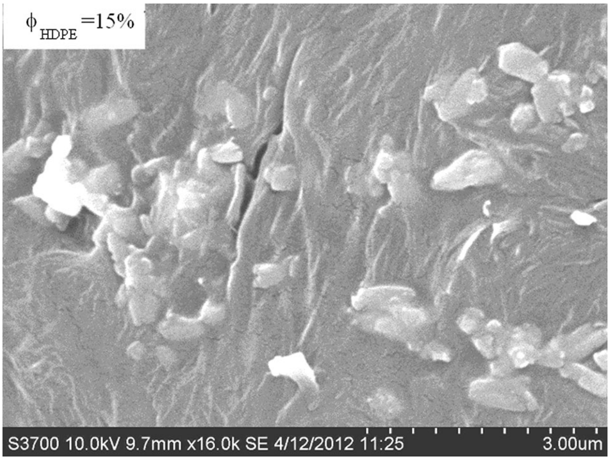

Fig. 14 SEM photograph of impact fracture surface of POM/EVA/HDPE/nano-$CaCO_3$ composites ($\phi_{HDPE} = 15$ %)

to form in the case of higher concentration of the HDPE; the toughening effect is weakened with an addition of the HDPE content, leading to the reduction of the impact fracture strength of the blends. Figure 14 is the SEM photograph of the impact fracture surface of the POM/EVA/HDPE/nano-$CaCO_3$ composite with ϕ_{HDPE} of 15 %. Similarly, the fracture surface is smoother than that of the POM/EVA/HDPE/nano-$CaCO_3$ composite with ϕ_{HDPE} of 6 %. As discussed above, the sea–island structure between the continuous phase and dispersion phase is difficult to form with an increase of the HDPE concentration, and the stress concentration of the matrix around the nano-$CaCO_3$ particles is not great enough to generate large plastic deformation due to low content of the nano-$CaCO_3$. Consequently, the impact fracture strength of the POM/EVA/HDPE/nano-$CaCO_3$ composite decreases in this case.

Conclusions

There were some effects of the HDPE weight fraction on the flexural and impact properties of the POM/EVA/HDPE blends and POM/EVA/HDPE/nano-$CaCO_3$ composites. It was found that the flexural modulus and flexural strength of both the blends and composites decreased slightly with increasing the HDPE weight fraction; the V-notched impact strength decreased slightly with increasing the HDPE weight fracture, while the unnotched impact strength increased in the case of low concentration of the HDPE, it should be attributed to the notched sensibility of the POM and HDPE resins. The values of the flexural modulus of the composites were higher than those of the blends; while the values of the impact fracture strength of the blends were higher than those of the composites under the same HDPE weight fraction. The results indicated that the impact fracture toughness of the POM resin could be improved with the HDPE resin, while the flexural stiffness of the POM/HDPE/EVA blends could be enhanced with the nano-$CaCO_3$ to some extent. Furthermore, the reinforcing and toughening mechanisms of the blends and composites were discussed.

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