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Preparation and interfacial behavior of superhydrophobic TPV/graphite fluoride composites

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Abstract

The High-density Polyethylene/Ethylene Propylene Diene Monomer/Graphite Fluoride (HDPE/EPDM/GF) composites are prepared by dynamic vulcanization technology. The composites with sea-island structure which contributes to excellent mechanical properties can be easily recycled and re-thermoplastized. A unique processing technology is designed to make graphite fluoride distributed on the phase interface of the material. The prepared phase interface with aggregated GF has very low interfacial energy, and this unique phase interface greatly improves the hydrophobicity of the material. The static WCA reaches 153° with a WSA 12° for composite containing 15 phr graphite fluoride. What’s more, the composite material has relatively good wear resistance, and when the surface of the composite material is damaged, the composite material can still maintain excellent hydrophobicity. Therefore, the superhydrophobic material prepared by this convenient method can be used for potential industrial application.

1. Introduction

The wettability of a solid surface is a very important characteristic property, which is mainly determined by the chemical composition and geometric microstructure of the surface [1, 2]. Surfaces with water contact angle (WCA) greater than 150° and water sliding angle (WSA) less than 10° are called superhydrophobic surfaces. The surfaces make it difficult for water droplets to adhere and have the abilities of self-cleaning and corrosion-prevention [3–5]. The materials with self-cleaning and corrosion-prevention are applied in industrial processes, bring great convenience in daily life, and have attracted great attention of scientific researchers [6–8].

There are two main ways to improve the hydrophobicity of the material surface: increasing the surface roughness of the material and using materials with low surface free energy. A class of superhydrophobic hybrid films was prepared through layer-by-layer self-assembly of silica nanoparticles and ionic liquids [9]. Bio-inspired superhydrophobic coating were prepared by chemical vapor deposition using gas phase silica [10]. Electrochemically deposited metal layer was prepared to form a superhydrophobic surface [11]. Superhydrophobic surfaces were etched by peeling off the surface material [12, 13]. The water-based hybrid coating was prepared by the sol aggregation method [14, 15]. Some superhydrophobic and oleophobic surfaces were prepared using photolithography technology [16–19]. The regularly densely packed arranged carbon nanotubes or TiO₂ nanorods formed superhydrophobic surface [20]. Fiber materials with super-hydrophobic properties are also obtained by preparation methods such as electrospinning [21, 22]. However, the above-mentioned methods of improving the surface hydrophobicity of materials have relatively shortcomings, such as the fragile material surfaces [14, 15], the expensive equipment [16, 21, 22], the harsh manufacturing processes [20, 23, 24] and so on. Therefore, it is necessary to explore a method for preparing more universal superhydrophobic materials to meet the increasingly demanding requirements.

In our previous work, the superhydrophobic properties of PP/PDMS/GF have been studied [25]. Through dynamic vulcanization technology, it is convenient to mix polymers and other inorganic fillers together [26].
PDMS with lower surface free energy is selected as the matrix, however, due to the poor compatibility of PP and PDMS, the tensile strength and elongation at break of the material are not sufficient for the application.

Therefore, in this paper, based on the previous work, new matrix EPDM (the ethylene segment content is 70 wt%) and HDPE are selected as the raw materials, HDPE/EPDM/GF composites were prepared through dynamic vulcanization technology and rough mold, and their superhydrophobic properties are studied. The outcomes of this work are expected to provide impactful insights into the design and preparation of composites with superhydrophobicity and mechanical stability.

2. Experimental

2.1. Materials
EPDM (3745 P) was purchased from Dow Inc. HDPE (60550AG) was purchased from China Oil and Gas Group Co. Ltd. GF was provided by Wuhan YuanCheng Technical Co. Ltd. Antioxidant RD (poly(1,2-dihydro-2,2,4-trimethyl-quinoline)) was purchased from Shanghai Linling Chemical Co. Ltd. Rubber accelerator TMTD and sulfur was purchased from Sinopharm Chemical Reagent Co. Ltd. ZnO was a commercial product of Shanghai Titan Scientific Co. Ltd. Stearic acid (analytic purity) was purchased from Shanghai Longfeng Chemicals Co. Ltd.

2.2. Preparation of TPVs and the formation of sea-island structure
Table 1 is the composites blended at different temperatures and their corresponding codes. At room temperature, EPDM and additives were added to a kneading machine and kneaded for 25 min to obtain EPDM premixed rubber. The EPDM premixed rubber and HDPE was taken with a mass ratio of 6: 4 and added to the Hakke (SU-200A, SuYan Technical Co. Ltd.). The blends were blended at 125 °C, 135 °C, 145 °C, 155 °C, 165 °C at a rotating speed of 60 rpm for 15 min and they were recorded as B1-1, B1-2, B1-3, B1-4, B1-5, respectively. The blends were added to Hakke and dynamically vulcanized at 175 °C for 8 min to obtain TPVs. The corresponding composites were recorded as D1-1, D1-2, D1-3, D1-4, D1-5, respectively. The thermoplastic dynamic vulcanizates (TPVs) were pressed in press vulcanizer to form the thickness of 2 mm boards: the TPVs were posed in the mold, preheated for 5 min, pressed at 175 °C for 5 min, and finally in a cold press for 10 min.

2.3. Preparation of TPV/GF composites
Figure 1 is the illustration of preparation process for TPV/GF composites. The EPDM premix rubber and HDPE were taken with a mass ratio of 6:4. The HDPE and fluorinated graphite was added to Hakke and mixed at 125 °C for 10 min, then the EPDM premixed rubber was added into the Hakke and melting blended at 125 °C at a rotation speed of 60 rpm for 15 min to obtain blended rubber. The boards were fabricated by the same operation as above.

The composites were prepared using sample saw and pounding apparatus. After that they were exposed in the air for 24 h for the following test.

2.4. Fabrication of the molded TPV/GF composites
Before the composites were posed in the press vulcanizer, a piece of W10 sandpaper (grit no.800) was inserted between the mold and the composite. This piece of the W10 sandpaper was used as a rough mold. And then, the molded composite boards were fabricated by the same operation as shown in 2.3.

2.5. Abrasion test of the TPV/GF composites
The abrasion test was performed by placing a sand paper (grit no.320) on the molded surface with 100 g weight on it and dragged for 1000 cm with speed of 10 mm s⁻¹.

| Table 1. The composites blending at different temperatures. |
|-----------------|-------|-------|-------|-------|-------|
| Blending Temper- | 125   | 135   | 145   | 155   | 165   |
| ature/°C        |       |       |       |       |       |
| Blends          | B1-1  | B1-2  | B1-3  | B1-4  | B1-5  |
| TPVs            | D1-1  | D1-2  | D1-3  | D1-4  | D1-5  |
The intensely abrasion test was performed by placing a sand paper (grit no. 320) on the molded surface with 10 kg weight on it and dragged for 1000 cm with speed of 50 mm s\(^{-1}\) to destroy the surface of composites.

2.6. Fluorine content analysis for GF
EDS analysis (QUANTAX 400-30, BRUKER AXS GMBH) and FT-IR testing (MAGNA-IR 560, Nicolet Company) were used to test fluorine content of GF.

2.7. Mechanical properties of blends and TPV/GF composites
Universal tensile testing machine CMT 2203 was used to test mechanical properties of blends and TPV/GF composites.

2.8. Hydrophobic properties of TPV/GF composites
The water static contact angle (WCA) and sliding angle (WSA) were tested by JC2000D3 Contact Angle Test Meter. Water droplet volume of static contact angle or WSA was 3 ~ 5 \(\mu\)l or 15 ~ 20 \(\mu\)l, respectively.

2.9. Micro-morphology analysis of TPV/GF composites
The microstructure of the material surface was analyzed with Field Emission Scanning Electron Microscope (FESEM) (S-4800, Hitachi Co. Ltd.).

2.10. The calculation of wetting coefficient \(\omega_a\) and interfacial tension
The wetting coefficient \(\omega_a\) (equation (1)) is derived from the Young’s equation and it is a widely accepted index that can analyze the distribution of fillers in the matrix when they reach a thermodynamic equilibrium state [27].

\[
\omega_a = \frac{\gamma_{epdm-gf} - \gamma_{hdpe-gf}}{\gamma_{epdm-hdpe}}
\]  
(1)

Therefore, the \(\omega_a\) of GF with HDPE/EPDM/GF system was calculated, where \(\gamma_{epdm-gf}\), \(\gamma_{hdpe-gf}\) and \(\gamma_{epdm-hdpe}\) are the interfacial tension between EPDM and GF, HDPE and GF, EPDM and HDPE, respectively. They are calculated based on the harmonic mean equation (equation (2)) [27].

\[
\gamma_{AB} = \gamma_A + \gamma_B - 4 \left[ \frac{\gamma_A^d \gamma_B^d}{\gamma_A^d + \gamma_B^d} + \frac{\gamma_A^p \gamma_B^p}{\gamma_A^p + \gamma_B^p} \right]
\]  
(2)

Where in A and B represent the two components of the compound, their surface tension values are \(\gamma\). The \(\gamma^d\) and \(\gamma^p\) are the dispersion and polar components, respectively.

3. Results and discussion

3.1. The preparation of phase reversal composites with dynamic vulcanization technology
Figures 2 and 3 is the tensile strength and elongation at break of composites prepared at different blending temperatures. Composites B1-1, B1-4 and B1-5 are blended at 125 °C, 155 °C, and 165 °C, respectively. According to figures 2 and 3, the tensile strength of B1-1, B1-4 and B1-5 are over 13 MPa and the elongation at break of them are over 470%. In contrast, the mechanical properties of the composites B1-2 and B1-3 which are...
mixed at the temperature of 135 °C and 145 °C are poor. After dynamic vulcanization, the tensile strength of composites D2-2 and D2-3 has increased significantly, which is higher than that of composites D2-1, D2-4 and D2-5.

Figure 4 is the illustration of phase reversal during HDPE/EPDM TPV processing. The rubber EPDM has better fluidity and low viscosity, while the plastic HDPE has a high viscosity after the melting point 125 °C, resulting in a sea-island structure: the rubber phase is the continuous phase while the plastic phase is the dispersed phase. The composite D2-1 is dynamically vulcanized from composite B1-1. During the dynamic vulcanization process, the rubber gradually cross-links and the viscosity increases rapidly while the plastic becomes fluid due to the high temperature. Therefore, the change in viscosity causes phase reversal, resulting in a new sea-island structure with plastic phase as continuous phase and rubber phase as dispersed phase.

The composites B1-2 and B1-3 were blended at 135 °C and 145 °C, respectively. At these temperatures, the rubber phase and the plastic phase do not show the sea-island structure, but a kind of interweaving and mixing structure with no obviously 'sea' phase. This structure causes more defects between the two phases, leading to relatively poor mechanical properties. However, after dynamic vulcanization, the interwoven rubber phases formed a small amount of spatial network structure. The tensile strength and elongation at break of EPDM after vulcanization are both greater than HDPE. Thus, the mechanical properties of composites D2-2 and D2-3 have been significantly improved.

During the preparation of composites B1-4 and B1-5, the fluidity of HDPE has improved due to the higher blending temperature. Therefore, HDPE becomes the continuous phase. At 155 °C and 165 °C, EPDM become
vulcanized rubbers, and the viscosity of them is higher than HDPE, so they become the dispersed phases. After dynamic vulcanization, the viscosity difference between EPDM and HDPE doesn’t change, there is no phase reversal phenomenon in composites D2-4 and D2-5.

In summary, by analyzing the mechanical properties of the blends B1-1 ~ B1-5 and their dynamic vulcanization products D2-1 ~ D2-5, we found that the composites blended at 125 °C with dynamically vulcanization show obvious phase reversal. The reversed composite has dispersed phases with higher degree of dispersion, and this structure is of great significance for improving the mechanical properties and the surface roughness of the composite materials.

3.2. Distribution of GF in TPV/GF composites
3.2.1. Elemental analysis of fluorinated graphite
In GF, the surface of the carbon layer is covered with C–F bonds which greatly reduces the surface free energy of GF and the interaction force between particles becomes very weak. Therefore, GF has excellent hydrophobicity and lubricity [28]. Figure 5 is the FT-IR spectrum of GF, it shows a typical intensive C–F vibrating peak at 1211 cm⁻¹ in the spectrum of GF, indicating that the GF used in the experiment has successfully grafted fluorine atom.
Figure 6 is the micro-morphology and elemental analysis spectrum of GF. As shown in figure 6, the molar ratio of carbon atoms and fluorine atoms is 56:44. It indicates that the GF used in the experiment is sufficiently fluorinated, which is consistent with the conclusion of the FT-IR analysis.

### 3.2.2. Thermodynamic prediction

The distribution of inorganic filler particles is closely related to the microstructure of the blend and the compatibility of the two phases, which also profoundly affects the mechanical properties of the blend [29]. According to the analysis of thermodynamic theory, the distribution of nanoparticles is closely related to the interfacial tension between the components.

Table 2 is the surface tension and its dispersion and polar component of materials used in this investigation.

#### Table 2. Surface tension and its dispersion and polar component of materials used in this investigation.

| Sample | Dispersion component $\gamma^d$ (N·m/m) | Polar component $\gamma^p$ (N·m/m) | Total $\gamma$ (N·m/m) |
|--------|-----------------------------------------|-----------------------------------|------------------------|
| HDPE   | 33.20                                   | 0                                 | 33.20                  |
| EPDM   | 28.00                                   | 0.68                              | 28.68                  |
| GF     | 21.50                                   | 0.84                              | 22.34                  |

Table 3 is the values of interfacial tension as calculated based on harmonic-mean equation.

#### Table 3. The values of interfacial tension as calculated based on harmonic-mean equation.

| Component couples | Interfacial tension (N·m/m) |
|-------------------|-----------------------------|
| HDPE/EPDM         | 1.12                        |
| HDPE/GF           | 3.34                        |
| EPDM/GF           | 0.87                        |

Figure 6 is the micro-morphology and elemental analysis spectrum of GF. As shown in figure 6, the molar ratio of carbon atoms and fluorine atoms is 56:44. It indicates that the GF used in the experiment is sufficiently fluorinated, which is consistent with the conclusion of the FT-IR analysis.

### 3.2.3. Design of the processing technology according to the thermodynamic properties of each component

According to the analysis of the thermodynamic properties, in the HDPE/EPDM/GF ternary system, GF tends to be distributed in EPDM. Figure 7 is the illustration of GF migration in composite materials. Therefore, in order to make the composite material have better hydrophobicity, a processing method is designed: During
blending process, GF is added into HDPE to prepare a HDPE/GF mixture, and then the mixture is blended with EPDM and dynamically vulcanized. During dynamic vulcanization, GF will gradually migrate from HDPE phase to EPDM phase, which is shown in Figure 7. However, the viscosity of EPDM becomes particularly large due to cross-linked structure, and it is difficult for GF to migrate into the EPDM phase. At the same time, there is a phase interface between HDPE and EPDM, on which GF will be delayed. Therefore, a considerable part of GF will be distributed on the phase interface of the composites. When the composite is molded by rough mold, it will be easier for GF to be exposed to the surface of the composite. This processing method makes the utilization rate of GF higher and can effectively improve the hydrophobicity of the material.

3.3. Hydrophobicity analysis of TPV/GF composites

3.3.1. The microstructure of TPV/GF composites

Figure 8 is the surface morphology of TPV/GF composites containing 15 phr GF under different conditions. Observation from figure 8(A) indicates that the molding process form the micrometer dimensional protrusions and gulfs. Figure 8(B) is the surface morphology of TPV/GF composites containing 15 phr GF after sandpapering. W40 sandpaper (grit no.320) was used and the composites were sanded under conditions in 2.5 in order to test the hydrophobic properties of the composites when the surfaces of molded composites are destroyed. From figure 8(B), there are many 5–20 μm irregular tree branches in the micro-structures of the composite, because of their overlapping with each other, a lot of multidimensional combinations are produced. Due to these structures, Cassie model is formed with air filled in the slots. According to figures 8(B) and (C), the surface structure of the composite is richer after being molded and abrasion, thus greatly increases surface roughness.

Figure 9 is high-magnification surface morphology of composite with different content of GF. The protrusions with a diameter of about 10 μm are directly obtained by molding the composite material with a rough mold. In addition, it is obvious that many dendritic structures with a diameter of 1 μm can be observed in figure 9. Large numbers of the dendritic structures appear in the composite when 15 phr GF is added. During the processing, a large amount of GF aggregate on the phase interface of the composite, reducing the interface energy of the phase interface, leading to a more exposable phase interface. What’s more, the internal stress caused by the large temperature difference makes the rubber phase which is enriched with GF generate deformation, leading to a more complex micro-structure. These tiny rubber phases enriched with GF not only reduce the surface energy of the composites, but also increase the surface roughness of the composites. Therefore, big static WCAs and small WSAs are displayed on the composites surface with the increase of GF content.

3.3.2. The influence of GF contents on composites’ hydrophobic properties

Figure 10 is the influence of GF contents on the WCAs of TPV/GF composites under different conditions. As shown in figure 10(A), the static WCAs of untreated composites varied with the contents of GF, ranging from 102° to 124° for composites containing 0 to 15 phr GF, respectively, exhibiting slight hydrophobicity. GF showed an increase in the hydrophobicity of the composite material, but it did not make the material superhydrophobic.

In addition, the composite was molded to fabricate a rough surface. The hydrophobicity was measured and shown in figure 10(B). Surprisingly, the WCAs were improved dramatically. The addition of GF enhanced
Figure 8. Morphology of composites containing 15 phr GF with (A) untreated surface; (B) surface after sandpapering and (C) molded surface.
Figure 9. High-magnification surface morphology of composite (A) without GF; (B) containing 15 phr GF.

Figure 10. The influence of GF contents on the WCAs of TPV/GF composites: (A) untreated composites; (B) molded composites.
hydrophobicity apparently. When the content of GF in the composite reaches 12 phr and 15 phr, the corresponding WCAs exceed 150°. It is shown that the rough mold has successfully prepared a complex microstructure on the composite material, giving the composite material a super-hydrophobic performance.

In the literature, a superhydrophobic coating on the glass has been fabricated by PVDF and candle soot. This coating gave 171° WCA and 3° WSA. But after abrasion, the WCA of the PVDF coating is reduced by 53°. The abrasion test was performed by placing a W40 sandpaper (grit no.320) on the coated glass slide with 50 g weight on it and dragged for 1000 cm (20 cm per cycle, 50 cycles) with speed of 5 mm s^{-1} [30].

As a comparison, the abrasion test of molded composite was performed according to 2.5. Figure 11 is the influence of abrasion on the WCAs of molded TPV/GF composites with different content of GF. Obviously, compared with the literature, the abrasion environment in this paper is more severe. According to figure 11(B), the WCAs of the molded composites after abrasion decreased slightly, ranging from 122° to 152°. The abrasion test shows that the molded composites have high mechanical stability.

For further, the molded composites were rubbed by W40 sandpaper to destroy the original surface. The surface state of the composite after intense abrasion is very similar to the surface of the destroyed sample. According to figure 11(C), the WCAs of the composites after intense abrasion range from 111° to 141°. When compared with the untreated composites, hydrophobicity of the composites after intense abrasion has been greatly improved. Therefore, it can be concluded that even if the specially prepared hydrophobic layer is lost, the composite material can maintain good hydrophobicity.

Figure 12 is the influence of GF contents on the WSAs of TPV/GF molded composites and composites after intense abrasion. Figure 13 is the static WCA and WSA of composite containing 15 phr GF. For the composite without GF, the WSA is 69°, whereas, surprisingly, the WSA is dramatically decreased with the addition of GF. Composite with 6 phr GF gave 23° WSA, and composite with 15 phr GF furnished the minimum WSA of 12°. For the composites after intense abrasion, the WSAs are gradually decreased with the addition of GF, and composite with 15 phr GF displayed the WSA of 23°.

In the literature, Nilsson and Song has used a range of sandpaper which grit designations between 40 and 600 to sand the surface of Teflon, and the static contact angles of samples are measured [31, 32]. It is found that sandpaper of different grain sizes can significantly affect the hydrophobic properties of the material surface. The contact angle and contact angle hysteresis of the Teflon samples sanded with 320 grit sandpaper are 150° and 31°, respectively. For comparison, the contact angle and sliding angle of the composites sanded with 320 grit sandpaper in this paper are 141° and 23°, respectively. It is shown that both the TPV/GF composites and Teflon display big static contact angle, but their sliding angle and contact angle hysteresis are slightly larger than the standard of superhydrophobic surface. This is because that the insufficient transition from Wenzel state to Cassie-Baxter state on the surfaces of TPV/GF composites and Teflon after sandpapering (grit no.320).

The WSC of the molded composite without GF is very large. After added GF, the micron-scale protrusions formed by the rough mold effectively improve the surface roughness of the composite material. The tiny rubber phases enriched with GF are exposed on the surface of the material, generating many dendritic structures. These dendritic structures not only reduce the surface energy of the composite material, but also increase the surface roughness of the composites, which results lead to a low WSA.

![Figure 11. The influence of abrasion on the WCAs of molded TPV/GF composites with different content of GF: (A) molded composites; (B) molded composites after abrasion; (C) composites after intense abrasion.](image-url)
3.3.3. Theoretical model of superhydrophobic materials
The static WCA of the material was described by two models: Wenzel Model[33] and Cassie-Baxter Model[34]. Figure 14 is the illustration of (A) Wenzel Model and (B) Cassie-Baxter Model.

Wenzel model points out the effect of roughness on the contact angle between water and the surface. But it believes that when the liquid contacts the rough solid surface, it will completely enter the gap. It was usually
described as the following relation (equation (3)):

$$\cos \theta' = r \left( \frac{\gamma_{s} \cdot \gamma_{l} - \gamma_{s} \cdot \gamma_{l}}{\gamma_{l}} \right) = r \cos \theta$$

(3)

where $r$ is the roughness factor, which is defined as the ratio of actual contact surface area over the vertical projected area. $\theta'$ is the apparent contact angle on a rough surface, $\theta$ is the ideal contact angle (Young’s angle) on a smooth surface of identical chemistry.

While Cassie-Baxter model believes that the size of the microstructure of the rough surface is very small, resulting in the presence of air between the solid surface and the droplet, so the droplet falls on the composite surface composed of air and solid. The relation between the apparent contact angle $\theta'$ and the ideal angle $\theta$ is in this case described as equation (4):

$$\cos \theta' = r_f \cos \theta + f - 1$$

(4)

Where $r_f$ is the roughness factor of the wetted area, and $f$ is the area fraction of the projected wet area.

In many research papers, blended Wenzel-Cassie-Baxter Model have been observed. Various effects occurring at the mesoscale influence the wettability of the rough solid surfaces such as the effect of the disjoining pressure and of crystal microstructure, grains, and defects. In the Wenzel regime, those effects have the potential to trap air pockets, resulting in Cassie-Baxter regime [35, 36].

Through the analysis of these two models, static WCA depends on the surface energy of the composite components and surface microstructure. In this composite, the intrinsic contact angles of all components including HDPE, EPDM and GF are greater than 90°, and they are all hydrophobic. Thus, the composites’ chemical components can meet the hydrophobicity requirements. Figure 15 is the illustration of the
superhydrophobic theory of TPV/GF composites, since there are more phase interfaces on the rough material surface, more GF is exposed, which has further improved the hydrophobicity. Therefore, the molded composite material has a higher static WCA and a lower WSA.

4. Conclusion

In this paper, the TPVs with sea-island structure as well as the TPV/GF composites with hydrophobic phase interface were prepared by dynamic vulcanization technology. GF was distributed on the phase interface of HDPE and EPDM, and the special internal phase structure determines the excellent hydrophobicity of the composites. When the composite contains 15 phr GF, the static WCA and WSA of the composite material reach 153° and 12°, respectively, meaning that the composites have outstanding hydrophobicity. After added GF, the surface microstructure changes from the Wenzel model to the Cassie-Baxter model. The WCAs of the molded composites after abrasion almost have no change, which means that the composites have excellent mechanical stability. Moreover, the static contact angle of the material surface still maintains 141° after intensely abrasion, meaning that the composites can keep hydrophobic even if the surface is damaged. All the above results suggest that the superhydrophobic composite prepared in this paper is durable, and has great application potential in industry and daily life.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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