Tribological Properties of LDPE + Boehmite Composites

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Tribological properties of composites of low density polyethylene (LDPE) reinforced with 1, 5, 10, 20 wt% micrometric Boehmite (B) and/or preheated Boehmite (HB) powders were studied in combination with two silane coupling agents (SCAs): vinyltri(2-methoxyethoxy)-silane (VTMES)-SCA 972 and 3-(trimethoxysilyl)-propylmethacrylate (3MPS)-SCA 989. When coupling agents are used, the samples not subjected to preheating provide higher yields of grafting and more bonds introduced on surfaces of Boehmite particles than heated ones. We have determined sliding wear by multiple scratching along the same groove using a micro scratch tester. Instantaneous penetration depth is lowered by the filler addition to LDPE. However, there is less viscoelastic recovery and the residual (healing) depths increase with increasing Boehmite concentration. Friction was determined using a pin-on-disk tribometer and also a moving sled friction device. Addition of 20 wt% untreated B and/or HB particles to LDPE matrix reduces friction. However, a more significant improvement of tribological properties results from incorporation of grafted particles; this occurs because of an increase of the filler/matrix interfacial adhesion. Surface morphology seen in scanning electron microscopy confirms this explanation. Friction values for all samples decrease along with the filler concentration increase. POLYM. COMPOS., 31:417–425, 2010. © 2009 Society of Plastics Engineers

INTRODUCTION

The use of polymer-based composites can provide a combination of good mechanical and tribological properties. Although addition of fillers can be advantageous, it can also leads to loss in mechanical properties due to incompatibility between filler and polymer matrix. Properties of composite materials are influenced by shape of the filler, the morphology of the system, and the nature of the interface between the phases. Thus a great variety of properties can be obtained with composites just by alteration of one of these items.

The advantages that composite materials have to offer must balance against their undesirable properties, which include complex rheological behavior [1, 2] and difficult fabrication techniques [3–6].

There is a number studies in the literature dealing with characterization of interfaces and their influence on the mechanical properties of polymer composites [7–31]. Kopczynska and Ehrenstein [32] discuss the importance of interfacial tension. The challenges consist in obtaining significant improvement in the interfacial adhesion between polymer matrix and the inorganic particles and also to achieve a homogeneous dispersion of the filler in the polymer [33–41].

Several special techniques are in used for the preparation of polymer composites [42–53]. We know that, during the preparation process, formation of covalent bonding between organic polymers and inorganic components contributes to the enhancement of the compatibility between them. This can be achieved through grafting of polymerizable groups onto oxide surfaces via hydroxyl groups followed by copolymerization with organic monomers. Among others, the surface treatment of the particles can prevent agglomeration and achieve better compatibility of the additives to the polymers; such treatment has a large influence on the properties of the composites [47–53].

We have fabricated low density polyethylene (LDPE) samples filled with different content of micrometer-scale Boehmite particles and studied tribological properties of the resulting composites. To overcome the difficulties of particles dispersion and adhesion, particle surfaces have been modified by grafting [19, 53]. For comparison, properties of unfilled LDPE were also evaluated under identical conditions.

EXPERIMENTAL PART

Materials

Silane coupling agents (SCAs), namely vinyltri(2-methoxyethoxy)-silane (VTMES)-SCA 972 and 3-(trime-

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TABLE 1. Properties of the coupling agents.

Product name	SCA 972	SCA 989
Classification and structural formula	Vinylsilane $CH_2{=}CHSi(OC_2H_4OCH_3)_3$	Acryloxy $H_2C{=}C(CH_3)CO_2(CH_2)_3Si(OCH_3)_3$
Chemical name	vinyltri(2-methoxyethoxy)silane	3-(trimethoxysilyl)propylmethacrylate
Molecular mass (ρ/gcm^{-3})	280.4	248.4
Specific gravity at 25°C	1.04	1.00
Refractive index $(n_d)^{25}$	1.427	1.429
Boiling point (t/°C)	285	255
Flash point (t/°C)	146	125

thoxysilyl)-propylmethacrylate (3MPS)-SCA 989 and high purity Boehmite (under the HiQ alumina trade name) with particle size ~55 μm, were received as a gift from Struktol Company of America and Engelhard Co., respectively. Table 1 lists some properties of the SCAs.

Toluene was from Sigma Chemicals Co. LDPE pellets (with melt index 35 g/10 min, density 0.923 g/cm^3, and softening point 98°C) were supplied by Huntsman Co. All reagents were analytical grade and were used as received.

Grafting of SCA onto Ceramic Particles

Introduction of reactive groups onto the commercially obtained and preheated (for 3 hr at 550°C) ceramic surfaces were achieved by a reaction of SCA with the hydroxyl groups of alumina. Two different types of SCA were used, and the temperature dependence of the process was characterized. Details have been described earlier [53].

Blending and Sample Preparation

LDPE samples reinforced with micrometric Boehmite particles were prepared via melt mixing followed by compression molding.

Pellets of dried LDPE and Boehmite powders were melt mixed in a C.W. Brabender D-52 preparation station at the rotation speed of 80 rpm at 150°C. The resulting blends were pelletized and dried. The blends contained in turn 1, 5, 10, and 20 wt% Boehmite.

Subsequently, the blends were dried for 8 hr at 100°C before compressing them in a Carver compression molding machine at 160°C at the compression pressure 20.7 × 10^3 kPa.

Characterization Techniques

Environmental Scanning Electron Microscopy. Micrographs of all samples were taken using a FEI quanta environmental scanning electronic microscope (ESEM). A small fraction of the samples were cut and/or fractured in liquid nitrogen, mounted on a copper stub, and coated with a thin layer of gold to avoid electrostatic charging during examination.

Sliding Wear Determination. The scratch test equipment used was a micro-scratch tester from CSEM, Neuchatel. For each sample 15 scratch runs were performed along the same groove at room temperature (23°C). The parameters applied were: load 5.0 N, scratch length 5.0 mm, scratch velocity 5.0 mm/min. A conical diamond intender with 200 μm of diameter and the cone angle of 120° was used.

Friction Measurements. Friction tests were conducted on the Nanoevea pin-on-disk tribometer from Micro Photonics Inc. The tribometer provides friction results under rotation conditions. Silicon nitride ceramic balls (NBD200), made by Saint-Gobain Ceramics, with the diameter of 3.2 mm were used. The balls were cleaned in acetone and thoroughly dried. Each test was performed under the following conditions: temperature 20 ± 2°C, speed 100 rpm, radius 2.0 mm, weight 5.0 N. The test durations ranged between 50 and 55 min.

The universal testing machine MTS-QTEST TM/5 with a friction device was used to measure static and dynamic friction of the composites on a Teflon and polypropylene (PP) surfaces. The polymer surfaces were cleaned with acetone and thoroughly dried. Each test was performed under the following conditions: temperature 20 ± 2°C, speed 150 mm/min, weight 10.0 N.

SCRATCH BEHAVIOR OF LDPE COMPOSITES

In the present study, three parameters were considered relevant to determine scratch resistance: (a) maximum or penetration depth R_p; (b) residual depth R_h; and (c) viscoelastic recovery φ.

The viscoelastic recovery φ has been defined [54–58, 13] as:

$$\varphi = \frac{R_p - R_h}{R_h} \cdot 100\% \qquad (1)$$

From each run, we obtain a diagram of the depth as a function of the scratch number. The scratch length was 5.0 mm. For detailed analysis, we have used the middle point 2.5 mm of the scratching range. Results are presented in Figs. 1–3. F1–F3

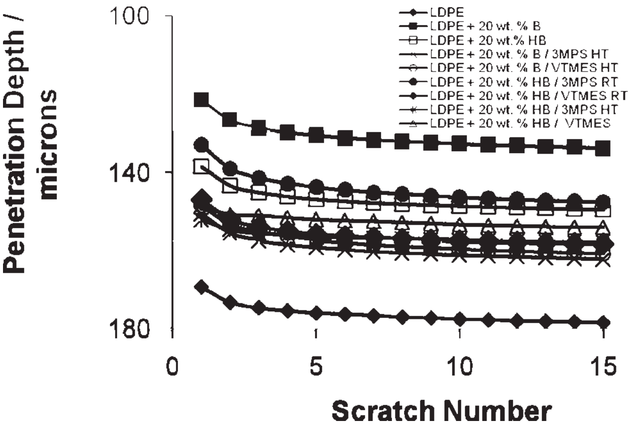


FIG. 1. Penetration depth R_p values to LDPE composites with 20 wt% filler at the constant force 5.0 N.

In Figs. 1 and 2, we show the curves of the penetration R_p and the residual depth R_h, respectively, as a function of the number of scratch tests performed.

It is clearly seen in Fig. 1 that composites are more resistant to instantaneous deformation by microscratching. At the force equal to 5.0 N, composites show the penetration depths R_p in range of 120–150 μm while LDPE has $R_p \approx 170$ μm. However, the situation is reversed for the residual depth R_h. All composites had relatively higher residual depths than LDPE. These results can be explained by increased brittleness [19] of the Boehmite (B) and preheated Boehmite (HB) reinforced composites. We recall that the definition of brittleness [59, 60] involves the reciprocal of the elongation at break.

In Fig. 3, we see the viscoelastic recovery as defined by *Eq. 1* as a function of the number of tests.

The viscoelastic recovery of the composites is ≈65 and 85% range while that of the LDPE is between ≈83% and 86%. The ceramic powder hinders the recovery.

In Fig. 4, we show variation of penetration depth R_p values of LDPE composites with varying amounts of untreated and treated Boehmite powders.

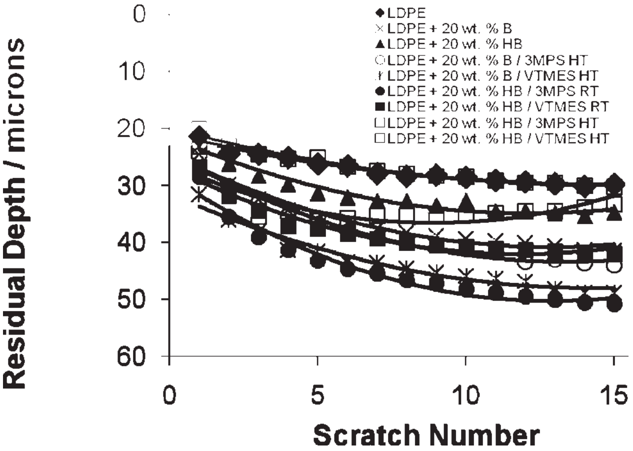


FIG. 2. Residual depth R_h values to LDPE composites with 20 wt% filler at the constant force 5.0 N.

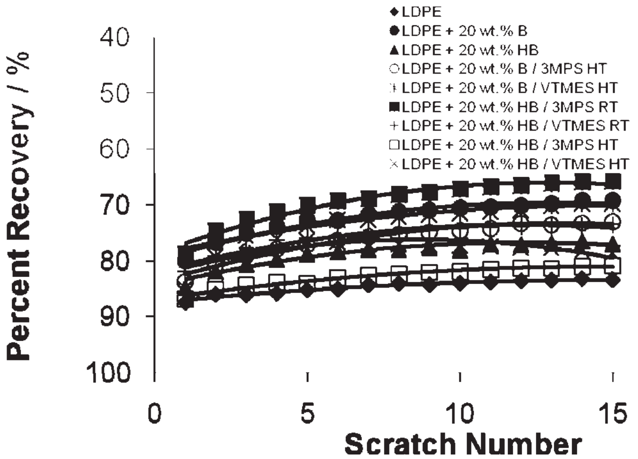


FIG. 3. Viscoelastic recovery *Eq. 1* of LDPE composites with 20 wt% filler at the constant force 5.0 N.

On the basis of penetration depth, residual depth, and viscoelastic recovery in particular, we may conclude that resistance to scratch deformation follows the sequence LDPE < LDPE + 1 wt% B < LDPE + 5 wt% B < LDPE + 10 wt% B < LDPE + 20 wt% B; scratch resistances of LDPE + untreated B/or HB and LDPE + treated B/or HB are comparable. We infer from these results that the coupling agents do not show significant effect on the scratch resistance or filler particle dispersion in to polymer. The agents do favor a better polymer-filler interaction [19]; this is due to replacement of hydroxide groups on surfaces of ceramic oxide powders with polymerizable organic groups [41, 53].

FRICTION OF NEAT AND REINFORCED LDPE COMPOSITES

The dynamic and static friction values of the LDPE composites against Teflon and PP surfaces are compared in Fig. 5a and 5b.

It is seen that dynamic and static friction of the composites incorporated with treated and untreated Boehmite particles is almost the same as that of neat LDPE on the Teflon surface. The effect is limited in magnitude, while

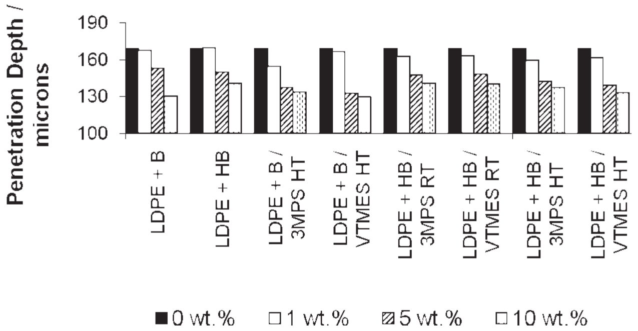
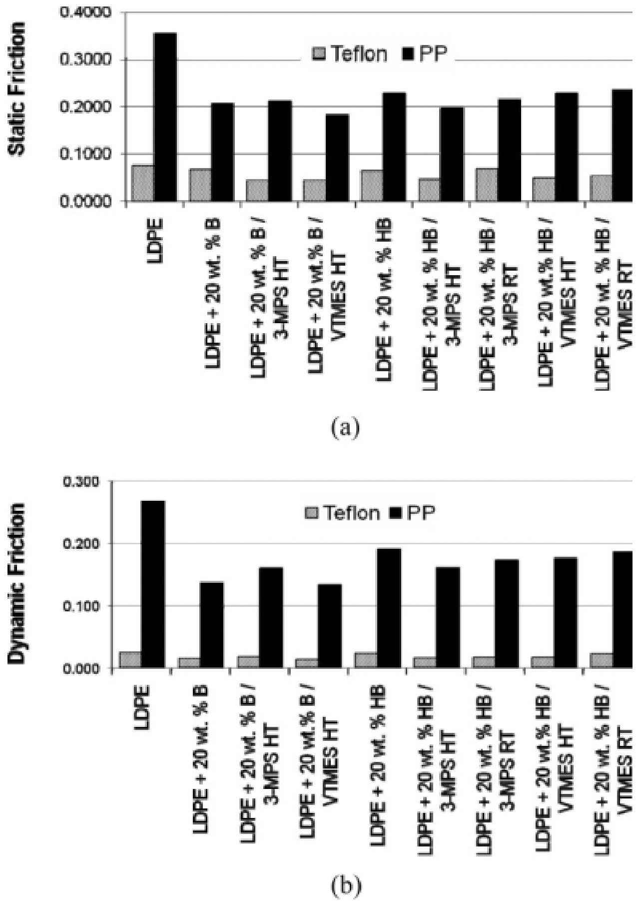


FIG. 4. Variation of penetration depth R_p values of LDPE composites with varying amounts of untreated and treated Boehmite powders.



(a)

(b)

FIG. 5. (a, b) Static and dynamic friction of LDPE composites filled with 20 wt% of untreated and treated B and/or HB powders.

all composites show 1.5 times reduced value of dynamic and static friction with respect to PP surface. Comparatively, LDPE + 20 wt% B is more effective than LDPE + 20 wt% HB in decreasing the friction of the composites. In HB containing samples there is more plastic deformation than in B containing composites. We explain this by lubrication effect of —OH groups of the B surfaces. On the other hand, a larger decrease in friction is seen for LDPE composites filled with grafted Boehmite particles—as compared with LDPE samples filled with grafted HB. As was reported in our earlier study [53], the high temperature heating of B powder causes the lowering of the concentrations of —OH groups on the powder surface and the grafting yields, respectively. Since grafting increases the interfacial adhesion between particles and polymer matrix, the difference in wear resistance between the untreated and grafted particles filled composites implies that the presence of the modified particles might increase the shear strength of the matrix, and hence prevent occurrence of large-scale destruction of the polymer matrix during sliding tests.

As shown in Fig. 5a and 5b, LDPE composites filled with 3MPS grafted B and/or HB exhibit improved tribological performance in comparison with VTMES treated composites. This fact can be explained by difference between [19, 53] organic group reactivity in the grafting process. As discussed before, we have used VTMES and 3MPS with vinyl and methacryl groups, respectively. The experiments show clearly more chemical activity of the methacryl groups and formation of larger amounts of organic–inorganic hybrid macromonomers. However, VTMES has lower activity and exhibits nearly the same grafting efficiency for both Boehmite powders. On the other hand, both SCAs provide higher yields of grafting onto surface of the Boehmite particles as compared with preheated samples.

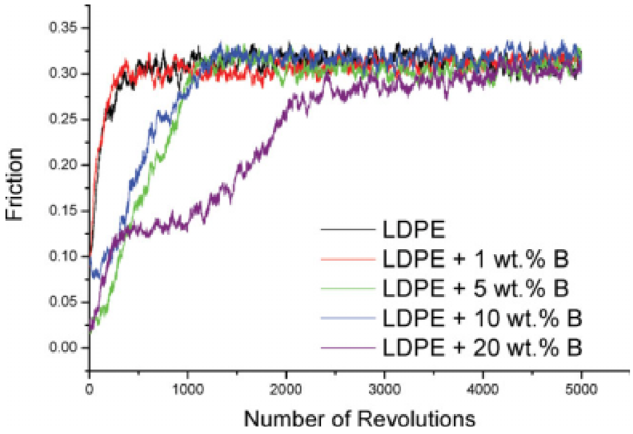


FIG. 6. Variation of friction of LDPE composites filled with varying amount of B powders. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Friction values were also measured using a pin-on-disc machine. We recall that in this case the partner surface is a silicon nitride ball. The friction as a function of the number of rotation cycles is shown in Figs. 6–11. F6–F11

LDPE displays a very stable friction value 0.3 in whole 5,000 cycles range.

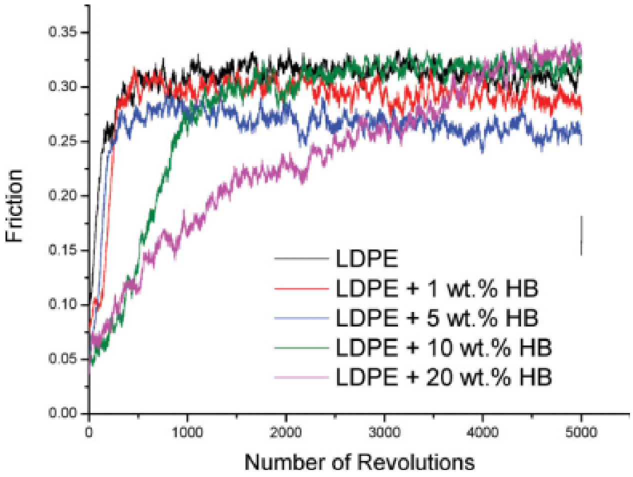


FIG. 7. Variation of friction of LDPE composites filled with varying amount of HB powders. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

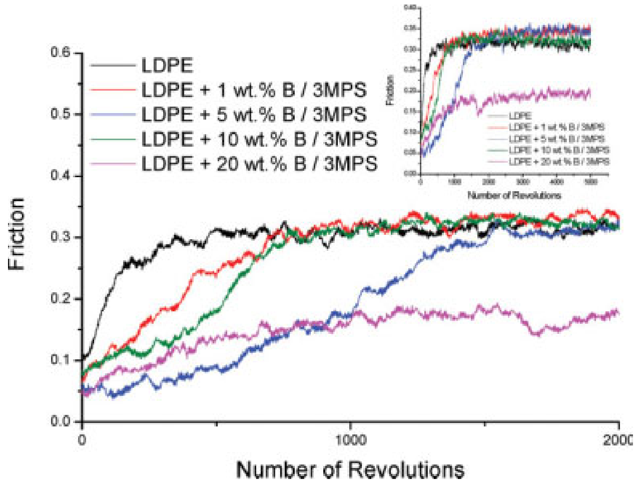


FIG. 8. Variation of friction of LDPE composites filled with varying amount of 3MPS treated B powders. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

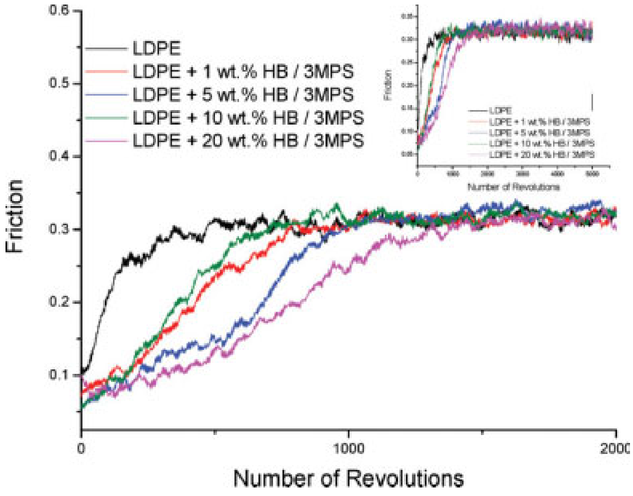


FIG. 10. Variation of friction of LDPE composites filled with varying amount of 3MPS treated HB powders. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

All composites demonstrate lower friction values (<0.3) than neat LDPE. An initial decline of friction is observed from pin-on-disk tribometer in the first hundreds of cycles followed by changes in different manners depending upon the materials composition. Both treated and untreated Boehmite filled composites produce a lower value of friction in the first 2,000 cycles, followed by some fluctuations and increases to about 0.25 and 0.30, respectively.

We note that the friction of neat LDPE is comparable with that obtained with sled moved against Teflon or PP surfaces. However, friction values of the composites obtained from pin-on-disk tribometer do not show the same effect, except for a lower number (lower than a 2,000 or so) of revolutions. All the composites reach the friction asymptote of pure LDPE after 2,000 cycles—except for LDPE +20% B/3MPS. In this case the friction is about one half of that for pure LDPE.

The testing configuration and the type of interacting partner exert significant influence on frictional process. This is why we perform different types of friction determination. Our sliding friction configuration (universal testing machine MTS - QTEST TM/5 with a friction device) is considered as "pure linear" sliding. Under this condition, the transfer film of the polymer to the counter face might be produced more effectively than in the case of pin-on-disk configuration [61]. When neat LDPE is tested on a pin-on-disk machine, the same value of friction, namely 0.3, is obtained. However, the results are different for the filled composites. A large number of revolutions increase the probability of abrasive wear; such wear results in fine debris and/or particles pulled out from the

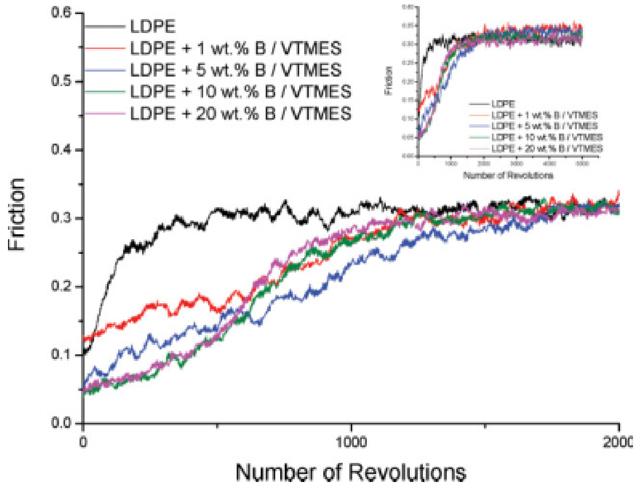


FIG. 9. Variation of friction of LDPE composites filled with varying amount of VTMES treated B powders. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

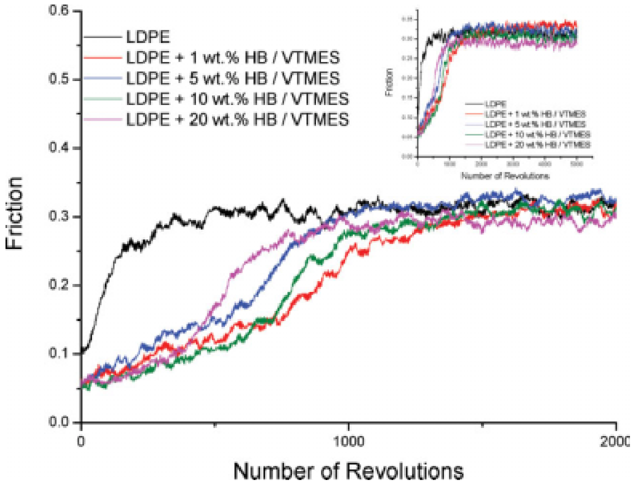


FIG. 11. Variation of friction of LDPE composites filled with varying amount of VTMES treated HB powders. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

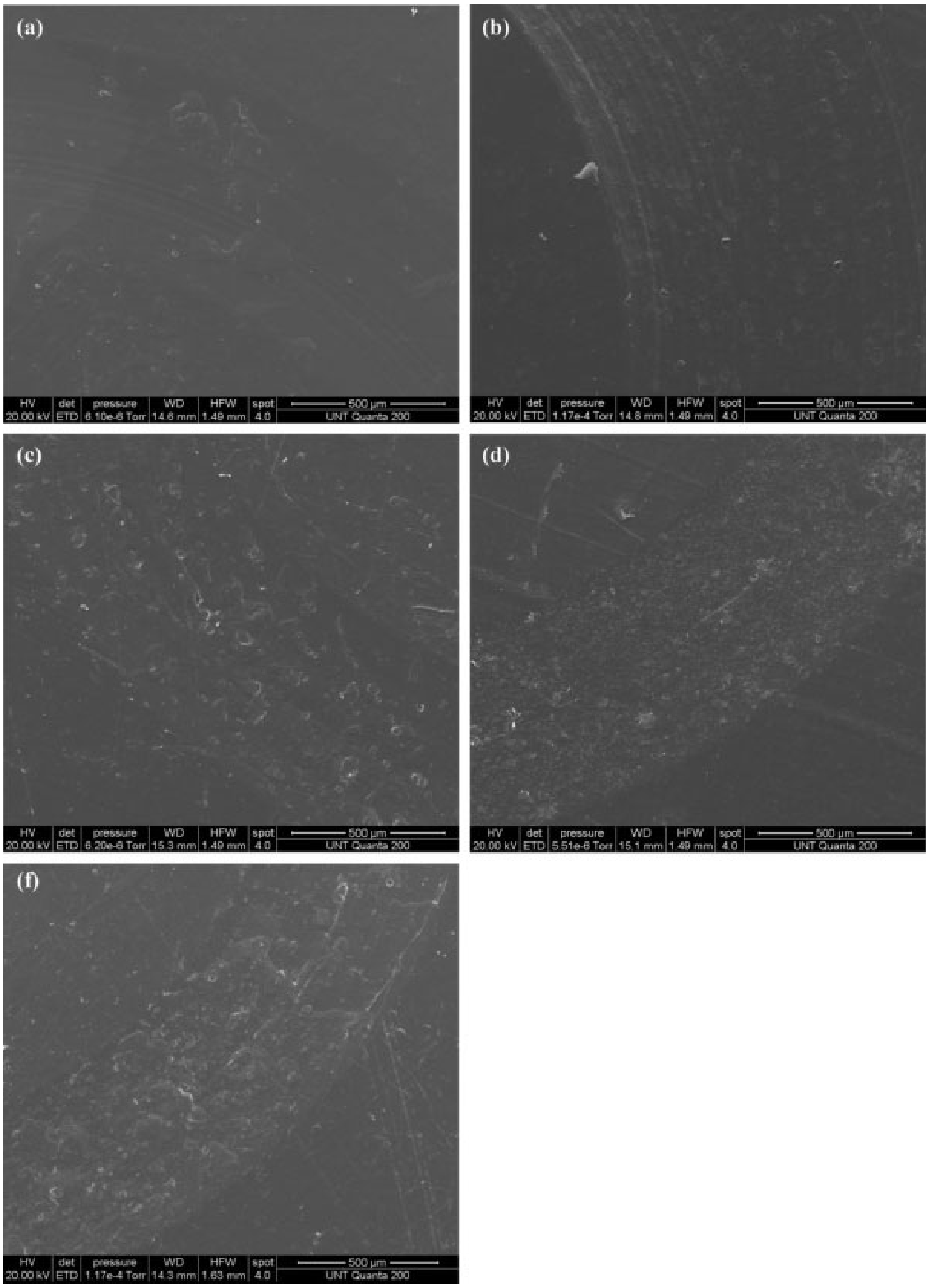


FIG. 12. Micrographs of the worn surfaces of LDPE (a), LDPE composites with 20 wt% filler:B (b), HB (c), B treated with 3MPS (d), and VTMES (f).

composites. Pin-on-disk data show improved tribological performance of the composites below 2,000 cycles. As a result, the sliding configuration can demonstrate more effectively the consequences of inclusion of Boehmite or HB particles on the tribological performance of LDPE.

WORN SURFACE MORPHOLOGY OF NEAT AND REINFORCED LDPE COMPOSITES

To further understand the effect of inorganic particles on the friction behavior of the composites, the worn surfaces of the samples have been studied by ESEM (Fig. 12a–f).

ESEM micrographs (see Fig. 12) show the worn surfaces of the neat LDPE and composites under identical conditions. LDPE and 20 wt% B or HB reinforced LDPE composite exhibit a distinct boundary between the wear track and none deformed regions. In 3MPS or VTMES treated Boehmite composite such a boundary is not clearly discernible (Fig. 12d–f).

Evident plucked marks appear also on the wear scars of the neat LDPE and 20 wt% B or HB reinforced LDPE

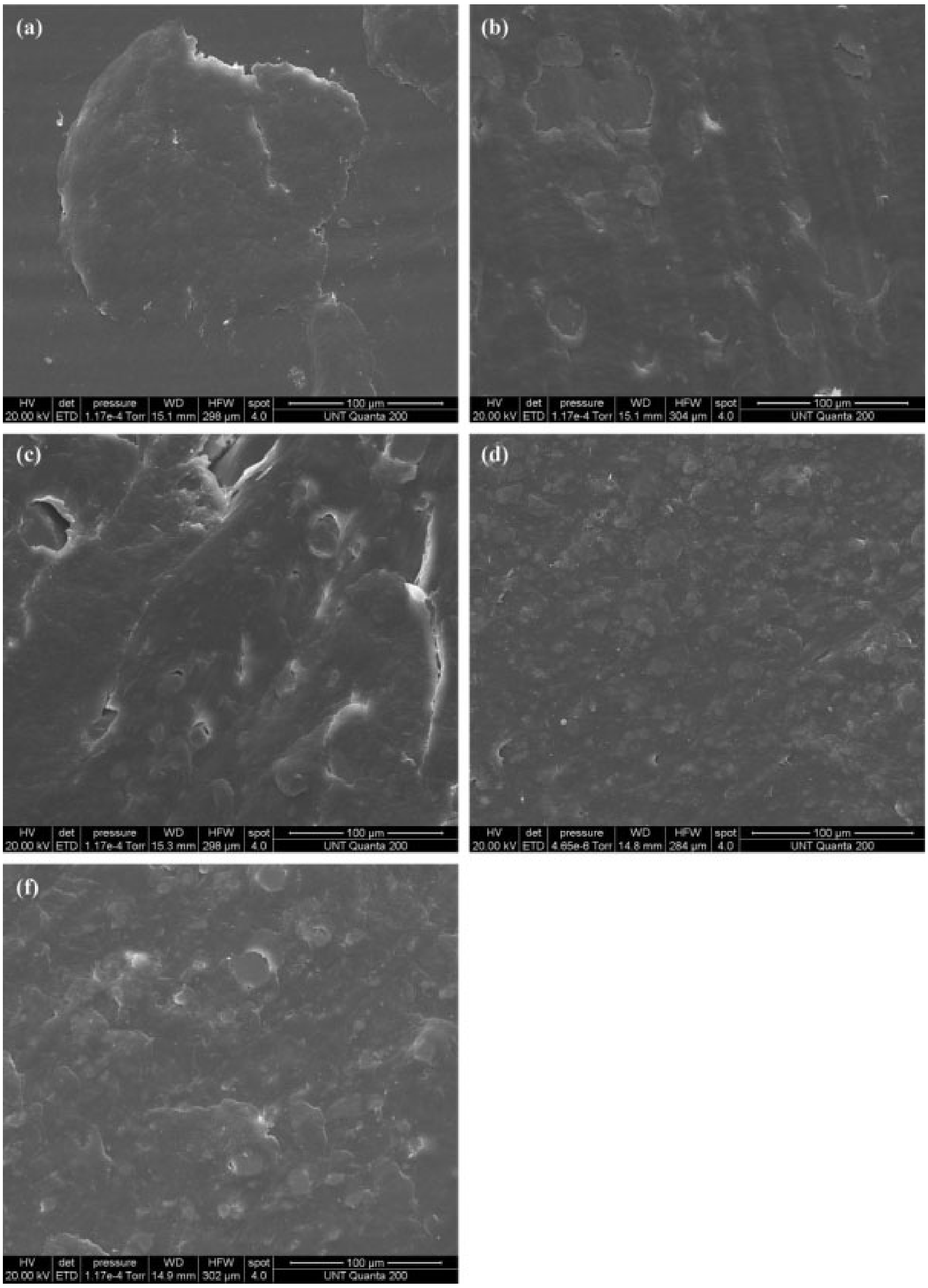


FIG. 13. Micrographs of the worn surfaces of LDPE (a), and LDPE composites with 20 wt% filler:B (b), HB (c), B treated with 3MPS (d), and VTMES (f).

surfaces—whereas the worn surfaces of the composites filled with treated Boehemite are characterized by slight scuffing.

In comparison with 20 wt% untreated B composites, the worn surfaces of the samples with 3MPS and VTMES-grafted fillers are quite smooth; this can be explained by changes in the adhesion properties and compatibility between particles and LDPE matrix after surface modification. These observations are consistent with the tribological data discussed earlier.

Detailed examination of the samples at a higher magnification is shown in Fig. 13a–f. The addition of the 3MPS and VTMES grafted particles remarkably enhances the tribological performance of the LDPE composite. The worn region of 3MPS-grafted Boehmite composite shows smooth surface without obvious grooves. The breakage of the LDPE matrix in filler/matrix interfacial region is much less pronounced, allowing the particles to remain in the composite and thus producing high load carrying capacity. The worn surface of the composites with untreated B particles shows microgrooves with traces of mild wear. In this case, the particles removal was aggravated since the non-polar LDPE matrix and resulting low adhesion with Boehmite could not effectively protect the particles from peeling off. These phenomena are clearly correlated with the role of the grafting. As expected, enhancing filler/matrix interfacial interaction improves the efficiency of the filler particles in reducing friction.

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