Characterization of Three-Dimensional Braided Polyethylene Fiber–PMMA Composites and Influence of Fiber Surface Treatment

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ABSTRACT: Three-dimensional (3D) braided polyethylene (PE) fiber-reinforced poly(methyl methacrylate) (PMMA), denoted as PE_{3D}/PMMA, composites were prepared. Mechanical properties including flexural and impact properties, and wear resistance were tested and compared with those of the corresponding unidirectional PE fiber–PMMA (abbreviated to PE_L/PMMA) composites. Both untreated and chromic acid-treated PE fibers were used to fabricate the 3D composites in an attempt to assess the effect of chromic acid treatment on the mechanical properties of the composites. Relative changes of mechanical properties caused by fiber surface treatment were compared between the PE_{3D}/PMMA and PE_L/PMMA composites. The treated and untreated PE fibers were observed by scanning electron microscopy (SEM) and analyzed by X-ray photoelectron spectroscope (XPS). SEM observations found that micro-pits were created and that deeper and wider grooves were noted on the surfaces of the PE fibers. XPS analysis revealed that more hydroxyl (—OH) and carboxyl (—COOH) groups were formed after surface treatment. The physical and chemical changes on the surfaces of the PE fibers were responsible for the variations of the mechanical properties of the PE/PMMA composites. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 99: 949–956, 2006

Key words: polyethylene (PE) fibers; composites; poly(methyl methacrylate) (PMMA); surface treatment; mechanical properties

INTRODUCTION

At the present time, thermoset-based (such as epoxies, polyimides, and bismaleimides) composites dominate the advanced composites market. Nevertheless, certain deficiencies including limited shelf life, insufficient toughness, low strain to failure, long and rigid multi-step processing, and moisture sensitivity of these thermoset polymers make them less suitable as matrix materials in certain circumstances. Composites based on thermoplastic polymeric matrix potentially offer several advantages like excellent biocompatibility, good processibility, high chemical stability, greater toughness and impact resistance, recycling and repairability over those based upon thermosetting resins.[1] Accordingly, thermoplastics and their com-

posites have found more medical applications ranging from low risk, noninvasive devices such as blood bags and surgical gloves to high risk applications for orthopedic and cardiovascular implants, catheters, etc.[2,3] Nevertheless, difficulty in processing is a major barrier in finding more applications for thermoplastic composites. Much effort has been made and various methods such as solution processing, slurry processing, film stacking, fiber commingling, dry powder impregnation, etc. have been employed to prepare unidirectional, multi-directional, and three-dimensional (3D) fiber-reinforced thermoplastic polymeric matrix composites.[4,5] In our previous research,[6,7] resin transfer molding technique was employed to prepare 3D braided carbon fiber-reinforced monomer casting nylon composites in an attempt to find promising alternatives to 3D carbon–epoxy composites for orthopedic applications. In the present work, poly(methyl methacrylate) (PMMA) was chosen to prepare 3D braided fabric-reinforced thermoplastic polymer composites. The PMMA was selected based upon the fact that it has long been used in medical field as bone cement to secure orthopedic implants to the skeleton or as the matrix material for various bioactive composites.[8–10] The 3D PMMA composite is expected to be a promising material for osteosynthesis devices.

Tensile strength	2420 (MPa)
Tensile modulus	≥ 86.4 (GPa)
Density	970 (kg m^{-3})
Strain at break	≤ 4 (%)
Diameter	36 (μm)
Melting point	152 (°C)
Molecular weight	1.2×10^6

It is recognized that carbon fiber–polymer composites offer high strength and stiffness and find wide engineering and medical applications. However, since higher stiffness of the composites than host bones is believed to cause osteoporosis, a composite with a modulus close to human bones is favored. Compared with carbon fibers that have been widely used in advanced composites, the advantages of polyethylene (PE) fibers are good biocompatibility, hydrophobia, chemical resistance, low density, excellent ductility and superior resistance to impact, wear, moisture, and chemical agents, and high specific strength and modulus, which has encouraged the translation of these properties to high-performance composites.

The aim of this study was to prepare and characterize the PMMA composites reinforced with treated and untreated 3D braided PE fibers and to compare the 3D with the unidirectional PE fibers reinforced PMMA (denoted as PE$_{3D}$/PMMA and PE$_L$/PMMA, respectively) composites in terms of mechanical properties and their changes with fiber surface treatment.

EXPERIMENTAL

Materials

PE fibers supplied by the Beijing Tongyi Unique Fiber Development Co., Limited, Beijing, China were used as the reinforcement in this work. The physical properties of the PE fibers are given in Table I. The preforms, 3D four-directional fabrics with a braiding angle of 20° were prepared by the Nanjing Fiberglass R and D Institute, Nanjing, China.

Fiber surface treatment

Liquid oxidation was employed to modify the surface of the PE fibers in the unidirectional and fabric forms. The medium used for oxidation treatment was a chromic acid solution ($K_2Cr_2O_7$ + H_2SO_4 + H_2O). The fibers were utterly immersed in the solution at ambient temperature for 10 or 15 min, removed, rinsed with water and acetone successively, and finally dried in a vacuum oven at ambient temperature for 24 h.

Composite fabrication

Methyl methacrylate (MMA) monomer was mixed and stirred with the initiator, then heated to a temperature of 90°C, and partially polymerized for a stipulated time period to obtain a liquid with a suitable viscosity.

Either braided fabrics or longitudinal fiber tows were placed in a mold with five separate cavities of 160 mm $\times$ 12 mm $\times$ 2 mm dimensions. The partially polymerized MMA was introduced into the mold under a certain pressure. The introduction of resin continued until no bubbles were observed within the resin flowing from the exit of the mold. The prepreg was then heat polymerized at 45°C for 16 h and placed in a 90°C oven for an hour to ensure complete polymerization. The resulted composite specimens were demolded and kept for characterization. The fiber volume fraction (V_f) of the composites used in the present study was controlled at 40%.

Measurement of mechanical properties

Measurement of flexural properties and impact strength was carried out at ambient temperature. A three-point bending fixture was chosen to test the flexural strength and modulus of the 3D composites. The testing procedures of the flexural properties were identical to those described elsewhere.[11] The flexural strength and modulus were calculated following ASTM D 790. Load-displacement curves were recorded during flexural tests. The impact strength was tested using an XCJ-500 Impact Tester (pendulum type) and the notched specimens were employed. The sample dimensions were 80 mm $\times$ 12 mm $\times$ 2 mm with a support span of 40 mm.

Mechanical properties were tested along either the braiding direction for the 3D composites or the fiber direction for the unidirectional composites. For each sample group, five specimens were tested from which the mean values and the standard deviations were obtained and analyzed using ANOVA.

Scanning electron microscopy observations

Scanning electron microscopy (SEM) was used to observe the surfaces of the PE fibers untreated and treated in chromic acid solution. The instrument used in this study was an XL30 scanning electronic microscope. The fiber surfaces were coated with a thin layer of gold to eliminate the charging effect.

X-ray photoelectron spectroscope analyses

The untreated and treated fibers were characterized with an X-ray photoelectron spectroscope (XPS). Analyses were performed on a PHI 1600 model surface analysis system with a 250 W MgK$_\alpha$ X-ray (1253.6 eV) source at a base pressure ranging from 10^{-8} to 10^{-9} Torr. All XPS spectra were averaged results from a surface area of $\sim$0.8 mm^2. The overall spectrum was
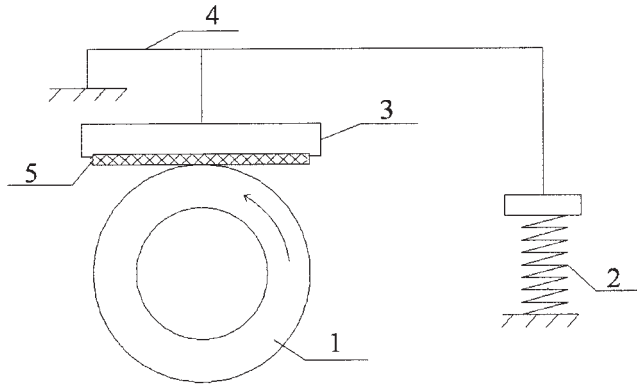
Figure 1 Schematic diagram of the wear tester: 1, steel ring; 2, spring; 3, specimen holder; 4, lever; 5, specimen.

taken at low resolution and the spectra of elements of interest were taken at high resolution. The surface functional group contents were determined by deconvoluting selected spectra after subtracting the linear background. Both mean values from three replicate tests and standard deviations were presented and ANOVA analysis was performed.

Sliding wear tests

Comparative lubricated sliding experiments (periodical dripping of phosphate buffer solution at a rate of 100 mL h^{-1}) were carried out to evaluate the tribological behaviors of the neat PMMA, unidirectional, and 3D composites using an MM200 wear tester (see Fig. 1). The fiber direction for the unidirectional composite specimens or the braiding direction for the 3D specimens was parallel to the sliding direction. The counterface used was a medium carbon steel ring with a hardness of HRC 52. Wear tests were performed at a normal load of 50 N and a sliding velocity of 0.42 m s^{-1}. Both the unidirectional and 3D composite specimens were removed and cleaned after an hour of sliding experiment (at the steady-state period) to measure the width of the wear scratches with a 3D profilometer. The volume loss, ΔV, of the specimens were calculated by

$$\Delta V = B\left[\frac{\pi R^2}{180}\arcsin\frac{b}{2R} - \frac{b\sqrt{R^2 - \frac{b^2}{4}}}{2}\right] \quad (1)$$

The coefficient of friction, μ, and the specific wear rate, W_S determined as specific volume loss, were calculated from

$$\mu = \frac{T}{RN} \quad (2)$$

$$W_S = \frac{\Delta V}{NS} \quad (3)$$

In the above three equations, R is the radius of the counterpart (carbon steel ring) in mm, b and B are the width (in mm) of the wear scratches and width of the testing coupons, respectively, μ is the coefficient of friction, T stands for the frictional torque (N m), N the normal load (N) applied to the wear specimens, W_S represents the specific wear rate in mm^3 N^{-1} m^{-1}, ΔV the volume loss (mm^3), and S the total sliding distance (m).

Three coupons cut in dimensions of 25 mm × 7 mm × 2 mm were tested for each sample group and the average values were collected and standard deviations were calculated. ANOVA analysis was also carried out. The coupons were fixed to a metal base during wear tests (see Fig. 1).

RESULTS AND DISCUSSION

Mechanical properties

Mechanical properties (flexural strength and modulus and impact strength) of the PE$_{3D}$/PMMA composites obtained in this work are presented in Table II. A comparison with the PE$_L$/PMMA composites is included in this table. As expected, the PE$_{3D}$/PMMA composites showed lower flexural strength and modulus than the unidirectional counterparts. The difference is caused by their different fiber architectures—fibers are aligned without any curvature in the loading direction and resin-rich areas that cause strain inhomogeneities are avoided in the unidirectional composites; whereas in the 3D composites, fiber undulation caused by crosslinking of fiber tows is unavoidable. It was found that the 3D composites exhibited much higher impact strength than their unidirectional counterparts (with values of 204.1 ± 23.1 kJ m^{-2} and 90.1 ± 17.6 kJ m^{-2}, respectively). A similar result was observed for carbon fiber-reinforced nylon composites[12] and carbon fiber–polylactic acid composites.[13] The impact strength of the composites is related to the ability of the components to absorb applied energy. Unlike unidirectional fibers, the entwining of fiber tows in the 3D fabrics is believed to increase the resistance to crack initiation.[14] Hence, the 3D compos-

TABLE II
Mechanical Properties of the PE/PMMA Composites

Properties	PE$_L$/PMMA	PE$_{3D}$/PMMA
Flexural strength (MPa)	267.4 ± 12.3	187.3 ± 8.8
Flexural modulus (GPa)	12.2 ± 0.8	9.0 ± 1.0
Impact strength (kJ m^{-2})	90.1 ± 17.6	204.1 ± 23.1

TABLE III
Mechanical Properties of Various 3D Braided
Composites and Human Cortical Bone

| Materials	Flexural strength (MPa)	Flexural modulus (GPa)
Cortical bone	180	20
Ti-Al-V	380	120
Stainless steel	280	200
Co-Cr	480	240
PE_{3D}/PMMA	187 ± 8.8	9 ± 1.0
C_{3D}/EP	756	47
C_{3D}/MC	395	21
K_{3D}/MC	205	14

ites showed higher impact strength than the unidirectional ones.

To be a promising material for osteosynthesis devices, the candidate material should meet certain physical and medical demands to be safe for clinical applications. Physically, it should offer sufficient strength and suitable stiffness (close to human bones). Table III lists the flexural strength and modulus of typical biomedical metals, cortical bone, and the 3D PMMA composites prepared in this study. As can be seen from this table, the PE_{3D}/PMMA composites showed a slightly higher flexural strength (mean value) than the cortical bone. Compared to the typical biomedical metals, Ti–Al–V, stainless steel, and Co–Cr, the modulus of the PE_{3D}/PMMA composites was much lower, which helps eliminate osteoporosis and possibility of refracture when these materials are used for osteosynthesis devices.

It was noted that the modulus of the 3D PMMA composites was lower than that of the cortical bone, which is likely to be a concern during the early stage of the healing process of the fractured bones. To this end, effort should be made to improve modulus and flexural strength of the PE_{3D}/PMMA composites to ensure stability at early healing stage. Many approaches are available to enhance the mechanical properties of these 3D composites such as fiber surface treatment, hybridization, increase of fiber loading, and so forth. In this work, only study on fiber surface treatment is presented.

## Changes of fiber surface conditions by liquid oxidation

It is well accepted that fiber/matrix adhesion at interfaces is a big concern for PE fiber composites owing to its low surface energy and chemical inertness. Several approaches such as chemical and plasma treatments have been adopted to improve the interfacial adhesion of the PE fiber composites.[15,16] Among various methods, chemical etching is a cost-effective and popular method.[17,18] Generally speaking, modification of or-
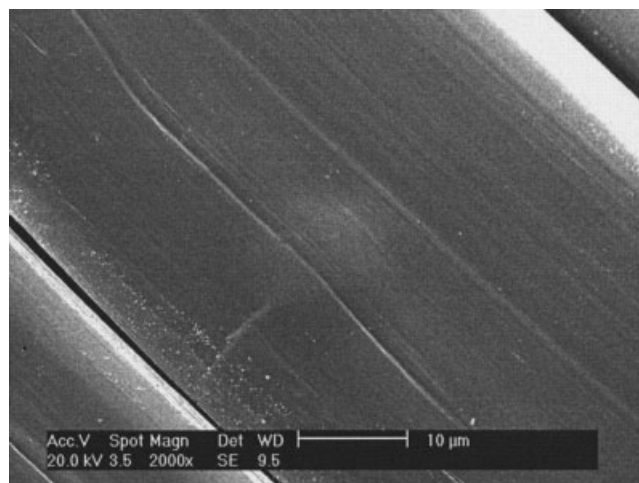
(a)
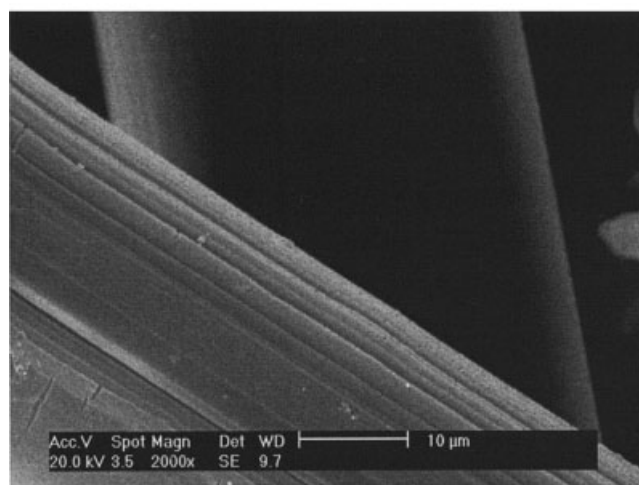
(b)
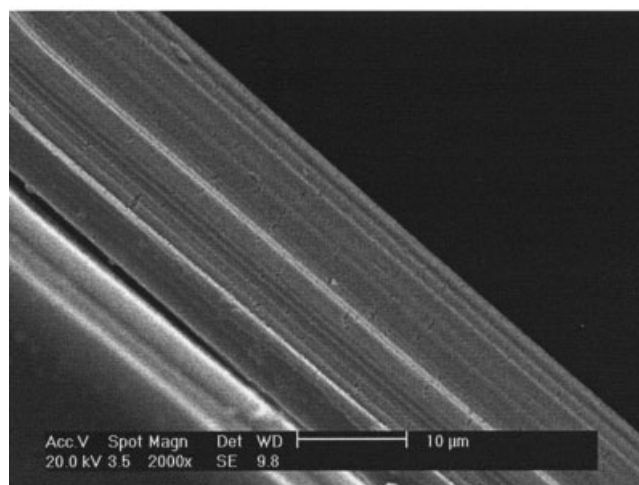
(c)

Figure 2 SEM micrographs of PE fibers: (a) untreated; (b) treated in chromic acid for 10 min; (c) treated in chromic acid for 15 min.

TABLE IV
Functional Groups on Different Carbon Fiber Surfaces and Their Contents

Functional group	—CH$_2$	—OH	—COOH
Untreated	85.4 ± 1.8	9.4 ± 1.7	5.2 ± 0.3
Treated	80.7 ± 1.7	13.0 ± 1.3	6.2 ± 0.4

ganic fibers *via* chemical etching involves the mechanisms of the abstraction of hydrogen atoms from the polymer back-bone and their replacement with polar groups (hydroxyl, carbonyl, carboxylic acid groups) from the oxidizing agents and change of surface roughness.[18,19] The change of physical condition of the PE fibers as a result of chromic acid etching is displayed in Figure 2. The difference was discernible among the fibers untreated and treated under different conditions.

Certain shallow grooves on the surfaces of the untreated fibers were noticeable (Fig. 2(a)). After surface treatment, more longitudinal grooves were observed and the grooves became deeper and wider as surface treatment processed (see Figs. 2(b) and 2(c)). Moreover, treatment in chromic acid introduced micro-pitting on fiber surfaces (see Fig. 2(c)). Undoubtedly, these micro-pits, together with the deepened and widened grooves, acted to enhance interfacial adhesion by mechanical interlocking between the PE fibers and the PMMA matrix. Similar micro-pits were reported by Moon and Jang[20] who declared that micro-pits improved the interfacial adhesion in PE fiber/vinylester composites through the mechanical interlocking between the micro-pits and the vinylester resin.

It was expected that the change of chemical condition also played a role in improving the interfacial adhesion between the PE fibers and the PMMA. As revealed by XPS analysis, the chemical composition on fiber surfaces changed after fiber treatment. From the XPS results given in Table IV, one could see that no new functional groups formed on fiber surfaces after surface treatment. Two functional groups, hydroxyl (—OH) and carboxyl (—COOH), were detected on the surfaces of the untreated and treated PE fibers. However, after liquid oxidation, the amount of each group measured by the deconvolution of C_{1s} peak changed significantly ($P < 0.05$ in all cases). The amount of the —OH and —COOH increased from (9.4 ± 1.7)% to (13.0 ± 1.3)% and from (5.2 ± 0.3)% to (6.2 ± 0.4)%, respectively.

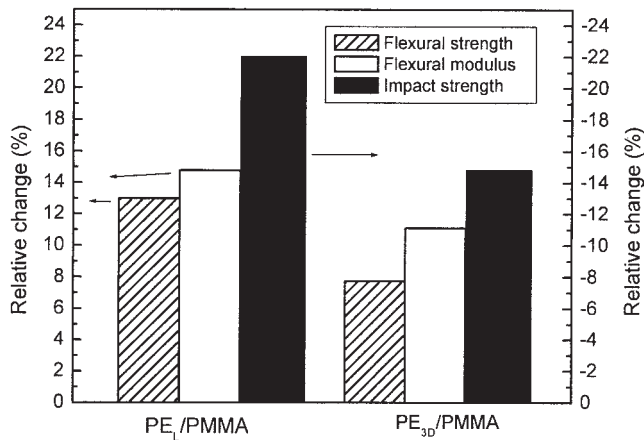### Changes of mechanical properties of the composites by surface treatment

Flexural properties

Changes of the flexural strength and modulus of the PE/PMMA composites are displayed in Figures 3(a)

and 3(b). As expected, fiber surface oxidization in chromic acid improved the flexural strength (see Fig. 3(a)) and modulus (see Fig. 3(b)) of the PE$_L$/PMMA ($P < 0.02$ for strength and $P < 0.01$ for modulus) and the PE$_{3D}$/PMMA ($P < 0.05$ for both strength and modulus) composites. ANOVA analysis demonstrated that the changes in flexural strength and modulus were statistically significant at a significance level of 0.05. Figure 4 presents the relative improvements of the flexural properties of the two composites as a result of fiber surface oxidation. After liquid oxidation in chromic acid for 15 min, the mean values of the flexural strength and modulus of the 3D composites increased by 7.7 and 11.1%, respectively; while the corresponding values for the unidirectional composites were 13.0 and 14.8%. This result may demonstrate that fiber surface treatment is more effective for the unidirectional composites than the 3D composites. The same result has been obtained from the study of carbon/epoxy composites.[21] The explanation is that the effective fiber–matrix interface area (measured interface
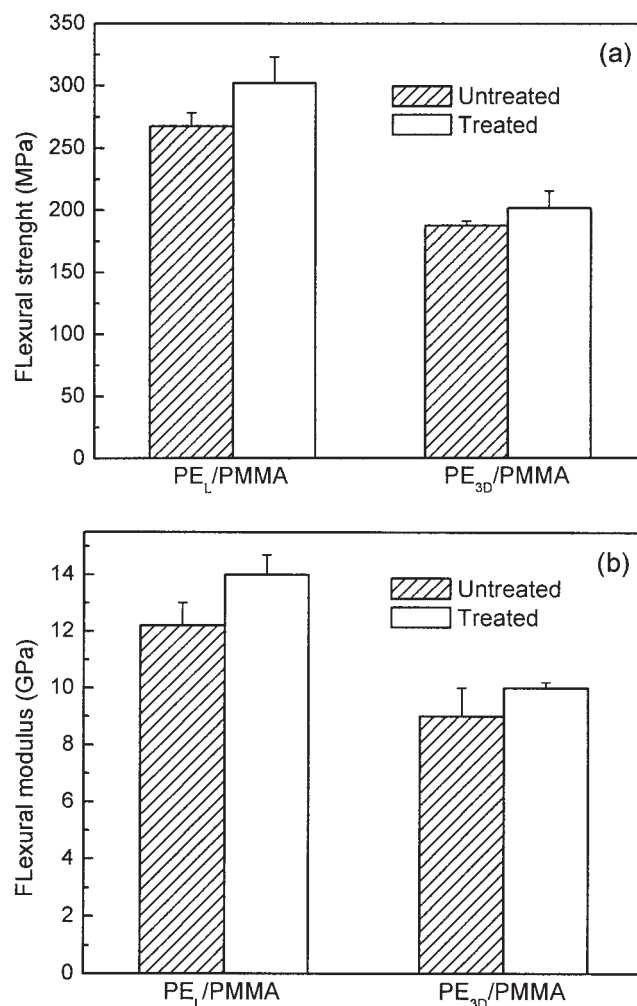
Figure 3 Effect of PE fiber surface treatment on flexural strength and modulus of the PE/PMMA composites.

Figure 4 Relative changes of mechanical properties of the PE/PMMA composites by fiber surface treatment.

area times $\cos\alpha$, α is the braiding angle of a 3D fabric) of a 3D composite is smaller than a unidirectional composite under identical fiber loadings.[21] It should be mentioned that the 3D PMMA composites exhibited the same trend as the unidirectional ones, suggesting that fiber architecture does not exert any effect on the trend of variations of flexural properties with interfacial adhesion strength.

The observed improvements in flexural strength and modulus are attributed to the enhanced mechanical interlocking between the fibers and the matrix as a result of the increase of fiber surface roughness (see Fig. 2), improved van der Waals forces and dipole–dipole interaction caused by cleansing effect of oxidization, promoted wettability, and strengthened chemical bonding between the fibers and the matrix due to the increased reactive functional groups on fiber surfaces.

It is a fact that the relative improvements in flexural strength and modulus were not tremendous for the PE_{3D}/PMMA composites. The PE_{3D}/PMMA composites with the treated PE fibers showed a modulus of 10 ± 0.2 GPa, which is still lower than human cortical bone. Further improvement in modulus is needed to meet physical requirements for fixation of load-bearing bones. This will be given in a further report.

Impact strength

The effect of liquid oxidation on the impact strength of the composites differs from that on the flexural properties. As can be seen from Figure 5, surface treatment led to significant reduction of the impact strength for both the PE_L/PMMA ($P < 0.05$) and the PE_{3D}/PMMA ($P < 0.05$) composites. The relative changes of the impact strength for both composites are also depicted in Figure 4. A reduction of 22.0% in impact strength was observed for the PE_L/PMMA composites as a

result of fiber surface treatment. Likewise, the PE_{3D}/PMMA composites showed a reduction of 14.8% after fiber surface treatment. This trend contrasted with that of the flexural properties, which is, however, understandable. The interpretation is that a stronger interface usually results in higher flexural strength and modulus, as strengthened interface improves the efficiency of load transfer from the matrix to the fibers. In the case of the impact strength, the interface affects the energy absorption in a different way. It is believed that the following energy absorption mechanisms work during impact process in a thermoplastic fiber-reinforced thermoplastic matrix composite: (1) fiber–matrix debonding, (2) fiber pull-out and post friction between fiber and matrix, (3) fiber elastic and plastic deformations and breakage, (4) matrix elastic and plastic deformations and fracture. Interfacial adhesion is considered to affect all these mechanisms. On the one hand, a strengthened fiber/matrix interface means limited fiber debonding and fiber pull-out, and therefore, reduced absorbed energy during the impact process. Similar mechanisms have been proposed by other researchers.[22,23] On the other hand, the increased interfacial adhesion restricts fiber and matrix deformations. The decreasing trend of the impact strength with increasing interfacial adhesion is common in literature. For example, Long et al. found that surface treatment of their PE fibers by plasma etching increased maximum stress and ILSS, and decreased peak absorbed energy of PE/epoxy composites made by structural reaction injection molding.[24] Woods et al. noted that the increase in interfacial adhesion led to the improvement in modulus and strength, but reduction in impact energy.[25] Similar phenomenon was also reported by Brown and coworkers.[26]
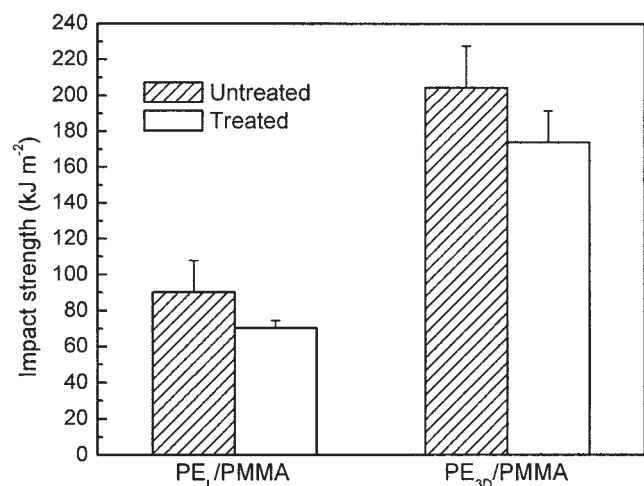
Figure 5 Effect of PE fiber surface treatment on impact strength of the PE/PMMA composites.
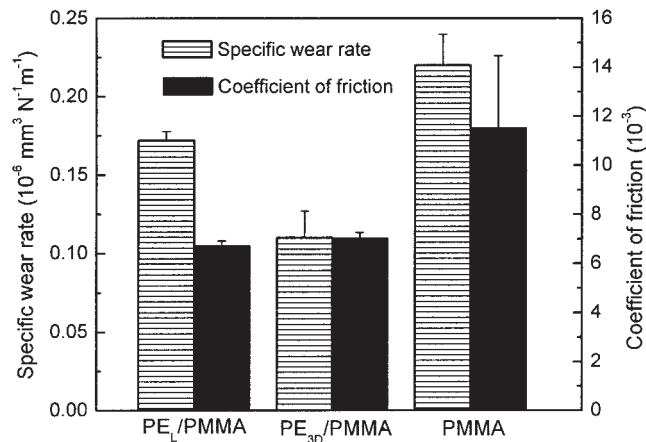
Figure 6 Coefficients of friction and specific wear rates of various materials.

Wear properties

As a potential material for orthopedics, wear behavior is also a critical property. For this reason, extensive investigation has been carried out for biomedical stainless steel,[27] bearing materials made from ultra-high molecular weight polyethylene (UHMWPE),[28] stainless steel/UHMWPE combination,[29] carbon–PEEK composite,[30] and so forth. The results from our preliminary sliding experiments on the PE/PMMA composites are presented in Figure 6. Usually, the addition of a reinforcing fiber to a matrix improves the tribological properties of a composite. The current results from the PE/PMMA composites also support this mechanism, *viz.*, the PE_L/PMMA and PE_{3D}/PMMA composites exhibited significantly lower coefficients of friction ($P < 0.005$) and specific wear rate ($P < 0.05$) than the matrix material. Interestingly, compared to their unidirectional counterparts, the 3D composites showed an insignificantly higher coefficient of friction ($P > 0.05$). However, the specific wear rate was substantially lower ($P < 0.005$) than the unidirectional ones, showing an advantage over the unidirectional composites. The specific wear rates were $(0.22 \pm 0.02) \times 10^{-6}$, $(0.172 \pm 0.01) \times 10^{-6}$, and $(0.11 \pm 0.02) \times 10^{-6}$ mm^3 N^{-1} m^{-1} for the PMMA, the PE_L/PMMA composites, and the PE_{3D}/PMMA composites, respectively. The corresponding coefficients of friction were 0.067 ± 0.002, 0.070 ± 0.003 and 0.120 ± 0.030. It can be concluded that fiber architecture exerts an effect on the wear behavior of the PE/PMMA composites. Similar results were reported by other researchers for composites with different fabric geometries.[31,32] The detailed results regarding the wear mechanisms of the PE/PMMA composites with various reinforcement structures will be presented in a further report.

CONCLUSIONS

The main conclusions can be summarized as follows:

1. The 3D braided PE fabric–PMMA composites were significantly superior to the long PE fiber-reinforced composites in terms of impact property and sliding wear performance. However, the flexural strength and modulus of the PE_{3D}/PMMA composites tested along braiding direction were lower than those of the PE_L/PMMA composites when tested along fiber direction.
2. The amount of the hydroxyl group (—OH) and carboxyl group (—COOH) on the surfaces of the PE fibers increased after oxidation in chromic acid, which was likely to cause enhanced chemical adhesion between the PE fibers and the PMMA matrix. Additionally, surface treatment created micro-pits and deepened and widened grooves on the PE fiber surfaces. These micro-pits and grooves were believed to improve the interfacial adhesion through the mechanical interlocking. The flexural strength and modulus of the PE/PMMA composites were enhanced after fiber treatment as a result of stronger fiber/matrix adhesion. Nevertheless, the impact strength of the two composites decreased after fiber surface treatment.
3. Compared with the PE_L/PMMA composites, the PE_{3D}/PMMA composites displayed lower relative changes in flexural and impact properties caused by PE fiber surface treatment, suggesting the 3D composites are less sensitive to fiber surface treatment in comparison to the unidirectional composites.
4. Wear tests demonstrated that the PE_{3D}/PMMA composites were superior to the PE_L/PMMA composites in terms of the specific wear rate, which is another advantage of the 3D composites over their unidirectional counterparts.

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