Wet adhesive wear characteristics of untreated oil palm fibre-reinforced polyester and treated oil palm fibre-reinforced polyester composites using the pin-on-disc and block-on-ring techniques

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Abstract: This article presents an investigation on the wear and friction characteristics of oil palm fibre-reinforced polyester (OPRP) composites sliding against a polished stainless steel counter-face under wet contact conditions. Two different types of OPRP composites were fabricated, which were based on treated and untreated oil palm fibres (treated oil palm fibre-reinforced polyester (T-OPRP) and untreated oil palm fibre-reinforced polyester (UT-OPRP), respectively). The experiments were conducted using two different techniques, pin-on-disc (POD) and block-on-ring (BOR), integrated into the same tribo-machine. The tests were conducted at different rotational speeds (500 and 700 r/min) and 50 N applied load for different durations (10–60 min). The specific wear rate ($W_s$) and the friction coefficient were presented as a function of sliding distance. The morphology of the worn surfaces was observed using scanning electron microscopy (SEM) and the damage features were characterized. The results revealed that treating oil palm fibres has a significant effect on the wear and frictional performance of OPRP composites. Treating the oil palm fibres enhanced the wear properties of polyester by about 35–52 and 65–75 per cent in the case of the POD and BOR techniques, respectively. The observations on the worn surfaces showed various features of the damages such as debonding and breakage of fibres in the UT-OPRP composite.

Keywords: oil palm fibres, treatment, wear and friction, wet

1 INTRODUCTION

Nowadays, efforts are put by many researchers to substitute synthetic fibres with natural ones that are compatible with new regulations on the environment and depletion of petroleum resources. Natural fibres exemplify environmentally friendly alternatives for use in the reinforcement of polymers [1–4]. They have many advantages over the synthetics such as abundantly available renewable resources (non-toxic), biodegradable, low cost, flexibility in usage, high specific strength, and low density. Polymeric composites based on natural fibres are becoming widely used in the production of several products such as construction materials, furniture, and automotive parts.

Recently, there has been a lot of research on the use of natural fibres in the reinforcement of tribo-polymerscomposites and the possibility of replacing the synthetics was discovered [1, 3]. It is known that the tribo-behaviour of the materials is subject to many factors such as contact conditions [5–9], test technique [10, 11], and operating parameters [8, 12]. On the other hand, reinforcing the polymers with fibres can significantly improve the tribo-performance of polymers or worsen them in some cases [13–15]. In polymeric composites based on natural fibres, Chand et al. [2] found that the interfacial adhesion characteristics of jute fibres played an important role in controlling the abrasive wear performance of polyester composites. These authors found that treated jute fibres give better
wear resistance when compared with untreated fibres. The formation of linkages at the interface of the matrix and jute fibres during deformation played a significant role in the wear process. In other words, interfacial adhesion between the fibres and the matrix controlled the wear performance of jute/polyester composites.

The contact condition (wet/dry) has an equally important role in the control of the tribo-performance of polymeric composites. It has been reported that the tribo-performance of some polymeric composites such as PA, UHMWPE [5], and epoxy [6] was improved under wet contact conditions compared with dry contact conditions. This was due to the advantages of using water, which served as a cleaner/polisher by removing the wear debris from the rubbing area and helped to absorb the heat generated by friction. In spite of that, the wear and frictional properties of some thermoplastic composites, such as PPS and PEEK, deteriorated or worsened under wet contact condition compared with dry contact condition [7]. This was due to the decrease in hardness of the surface layer of the composite. Furthermore, the wear mechanism could be transferred from adhesive into abrasive because of the absence of film transfer on the counterface, where the removed debris and fibres in the interface attacked both surfaces [6].

Recently, an attempt was made by Zhang et al. [11] to investigate the effect of the length of carbon fibres on the tribo-performance of epoxy composites sliding against steel using two different techniques, pin-on-disc (POD) and block-on-ring (BOR). In both techniques, longer fibres in the composites exhibited better wear resistance. However, variations in the wear results, in the same conditions, showed higher wear resistance with BOR compared with POD, i.e. wear properties were not intrinsic material parameters but were sensitive to the conditions applied.

In previous works by the present authors, the effect of untreated oil palm fibres on the abrasive and dry adhesive wear behaviour of polyester composites was studied [3, 4]. In the dry adhesive work, the poor adhesive characteristics of the fibres to the polyester matrix led to debonding of the oil palm fibres during the sliding, especially under severe conditions. In spite of that, the untreated oil palm fibres showed good support to the wear performance of polyester in low ranges of load and speed by forming a back-film transfer to the composite surface. However, the friction coefficient measured was high (about 0.7), i.e. the interface temperature was high. This softened the polyester regions and led to pullout of the fibres from the bulk to the surface.

In view of the above, there is a necessity to investigate the effect of water on the adhesive wear behaviour of the oil palm fibre-reinforced polyester (OPRP) composites in different conditions and techniques. In the present work, the adhesive wear performance of treated oil palm fibre-reinforced polyester (T-OPRP) and untreated oil palm fibre-reinforced polyester (UT-OPRP) composites was studied under the wet contact condition using two different techniques. The tests were conducted using a tribo-machine that combined the POD and BOR techniques working simultaneously against the identical counterface surface (stainless steel). The experimental tests were carried out for different test durations (10–60 min) at different rotational speeds (500 and 700 r/min) and 50 N applied load.

2 EXPERIMENTAL DETAILS

2.1 Preparation of samples

2.1.1 Treated and untreated oil palm fibres

A bunch of fruits of oil palm was collected from a farm in Muar, Johor state, Malaysia. The procedure for preparing oil palm fibres (5–10 mm length) is described in detail elsewhere [3]. The oil palm fibres have a diameter of about 350 μm. In the treatment process, the prepared oil palm fibres (10–15 mm length) were soaked in 6 per cent NaOH solution in a water bath, where the temperature was maintained throughout at 26 ± 2 °C for 48 h. The fibres were rinsed and left to dry at room temperature before being put in an oven for 5 h at 45 °C. In reference [3], because of the treatment of the fibres, the outer layer (skin) of the oil palm fibres was washed out and a bundle of fine fibres was clearly seen.

2.1.2 Preparation of OPRP composites

A metal-closed mould (120 × 120 × 20 mm³) was used to fabricate the T-OPRP and UT-OPRP composites. Before the fabrication, internal surfaces of the mould were greased with a thin layer of wax as a release agent. The prepared fibres were placed randomly in the mould and pressed into a mat to a thickness of 25 mm. The ends of the fibres keep facing the walls of the mould during the process. Polyester resin, mixed with 1.5 per cent of hardener, was poured into the mould until the fibres were totally impregnated. A pressure of about 50 kPa was applied on the top of the mould to force out the bubbles. The prepared composite block was cured for 24 h at room temperature (24 °C). Furthermore, blocks of neat polyester (NP) were prepared by the same method as above but without reinforcement. Some of the mechanical properties of the composites are given elsewhere [3]. The fibre content in the composites was about 48 vol%. In addition, the interfacial adhesion characteristics of the fibres to the polyester matrix have been reported in reference [3], showing that untreated oil palm fibres have poor interfacial adhesion to the polyester matrix, where the pullout process took place during the tests. On the other hand, the interfacial adhesion of treated

oil palm fibres to the polyester matrix was very high, that is, no pulling out took place.

2.2 Experimental tests

The tests were conducted on a tribological machine combining the POD and BOR techniques [8, 16]. In this machine, both POD and BOR run simultaneously against the same surface with identical characteristics. This gives a more accurate comparison of both techniques. The specimens of both techniques are immersed in a container filled with tap water (hardness of 120–130 mg/l).

For each test, a stainless steel counterface (AISI 304; 50BH hardness and 0.09 μm Ra roughness) and composite specimens (0.353 μm Ra) were polished using an SiC abrasive paper (grad 1500). The lubricant container was filled with fresh tap water for each test. The tests were conducted under ambient conditions for various durations (10–60 min) at different rotational speeds (500 and 700 r/min). In the machine, the track radii of both techniques are different. The equivalent sliding velocities are 2.8 and 3.9 m/s for POD and 5.6 and 7.8 m/s for BOR. The tests were carried out at an applied load of 50 N with an equivalent contact pressure of 0.5 MPa in POD, whereas in BOR, at the beginning of the test, the contact pressures were equivalent to 35 MPa, and after tests, the average of the contact pressures was determined to be about 1.31–1.42 MPa. This was because of the variations in the apparent contact area in the case of BOR. At the beginning of the test, the contact was almost in line and then increased as the wear process continued. Meanwhile, in POD, the apparent contact area remained the same throughout the test duration.

The weight of the specimens was determined before and after each test using ±0.1 mg balances (Shimadzu AW120) and then the specific wear rate ($W_s$; mm$^3$/m N) was calculated. After the test, the specimens were kept in an oven for 24 h at 30°C for the drying process. The roughness of the wear track of the counterface (the disc and the ring) and the sliding surface of the specimens were measured before and after the tests using a Mahr Perthometer S2. The worn surfaces of the composites were coated with a thin layer of gold using an ion sputtering device (JEOL, JFC-1600) to observe the microstructure of the composite using scanning electron microscopy (SEM; JEOL, JSM 840). The tests were repeated at least three times, and the typical values of the standard deviation of $W_s$ are listed in Table 1.

3 RESULTS AND DISCUSSION

The specific wear rate ($W_s$), friction coefficient results, and SEM micrographs of the worn surface of NP, UT-OPRP, and T-OPRP composites are presented in Figs 1 to 10.

Table 1 Typical values of the standard deviation variation of $W_s$

<table>
<thead>
<tr>
<th>Material</th>
<th>Pin-on-disc</th>
<th>Block-on-ring</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neat polyester</td>
<td>0.18–0.9</td>
<td>0.5–0.65</td>
</tr>
<tr>
<td>Untreated oil palm fibre-reinforced polyester</td>
<td>0.165–1.2</td>
<td>0.12–1.7</td>
</tr>
<tr>
<td>Treated oil palm fibre-reinforced polyester</td>
<td>0.13–1.15</td>
<td>0.13–0.9</td>
</tr>
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Fig. 1 $W_s$ versus sliding distance using the POD technique at 50 N applied load
T-OPRP shows lower $W_s$ when compared with UT-OPRP and NP. In other words, the presence of treated oil palm fibres enhances the wear properties of polyester. Furthermore, treated fibres give better support to polyester than untreated ones. Moreover, treated fibres reduced the $W_s$ of the wear polyester by about 35–52 per cent. Meanwhile, untreated oil palm fibres enhanced polyester by only about 15–29 per cent at a longer sliding distance.

Figures 2(a) and (b) show the friction coefficient behaviour versus sliding distance. The friction coefficient values of all the materials, especially at low sliding velocity, are almost the same. Steady state is reached after 5–8 km sliding distance for all the tested materials and the range of the friction coefficient values is between 0.1 and 0.17. At high velocity (3.9 m/s), the UT-OPRP composite shows a lower friction coefficient when compared with T-OPRP and NP (see Fig. 2(b)). The presence of treated oil palm fibres does not have any significant effect on the frictional behaviour of polyester.

The wear properties of the composites seem to be controlled by the interfacial adhesion characteristics of the fibres to the matrix. In other words, the lower $W_s$ values of the T-OPRP composite compared with UT-OPRP is because of the better interfacial adhesion characteristics of the former. In addition to that, the outer layer of the treated fibres was washed away and this could allow the polyester resin, in liquid form (before hardened), to enter the bundles and fill it. This assists in stabilizing the surface characteristics of the composite at the sliding during the test.

In contrast, untreated fibres are empty bundles and have poor interfacial adhesion properties [3]. This weakens the bonds between the fibres and the matrix. Because of the mechanical loading condition, during the sliding, the possibility of debonding and pulling out the fibres is high and this weakens the composite surface at the sliding. It has been reported that high porosity fillers (CaCO$_3$) worsened the wear performance of polyester composites [17]. This could explain the lower wear performance of the high-porosity untreated oil palm fibres. Further explanation is given with the assistance of SEM observations of the worn surface of T-OPRP, UT-OPRP, and NP (Figs 3 to 5). The arrows on the SEM micrographs represent the sliding direction of the counterface.

For the T-OPRP composite, at a short sliding distance (Fig. 3(a)), a smoothened (polished) surface can be observed in the polyester regions. Moreover, the treated oil palm fibre still adheres well to the polyester. At the end of the test, the roughness of the composite was not highly varied (from 0.21 $\mu$m before the test...
to 0.25 μm after the test). At a longer sliding distance (10 km), a slight debonding of fibres occurred without damages to both the regions (fibrous and resinous). Moreover, cracks appeared in the cross-section of the bundle, which could be because of the mechanical loading. On the other hand, the UT-OPRP composite shows debonding of fibres at a short sliding distance and a gap between the fibres and the matrix (10 μm) (Fig. 4(a)). The debonding of the fibres at the beginning of the test leads to tearing of the empty bundle of fibres (Fig. 4(b)). Moreover, at a longer sliding distance, the worn surface seems to have deteriorated and abrasive wear nature appears on the worn surface (roughness increased, on average, from 0.25 to 0.78 μm). This could explain the low support of the untreated oil palm fibres to polyester when compared with the treated ones (Fig. 3).

In SEM micrographs of NP, Fig. 5 shows a rough surface especially at a longer sliding distance (10 km), where the average roughness increased from 0.19 to 1.2 μm. The rough surface could have been caused by two reasons. First, the polyester debris was washed and cleaned with water. Second, because of the cooling process via water, the possibility of generating film transfer is so weak. Nevertheless, the wear behaviour of the polyester could be similar to the published work on epoxy [6], where the adhesive wear was transferred into three-body abrasion because of the increase in the counterface roughness and the third bodies acted in the interface.

The changes that occurred on the counterface roughness after tests are listed in Table 2. One can say that the counterface roughness was only slightly increased after the tests were conducted on T-OPRP. This could be another reason for the higher performance of the T-OPRP composite compared with others during the tests. The poor wear behaviour of NP could be because of the large increase in counterface roughness.
Fig. 6 $W_s$ versus sliding distance using the BOR technique at 50 N applied load

Fig. 7 Friction coefficient versus sliding distance using the BOR technique

Fig. 8 SEM micrographs of T-OPRP tested using BOR at 5.6 m/s sliding velocity for different sliding distances

Fig. 9 SEM micrographs of UT-OPRP tested using BOR at 5.6 m/s sliding velocity for different sliding distances
Wet adhesive wear characteristics of UT-OPRP and T-OPRP composites

3.2 Block-on-ring

The frictional and wear results obtained at 5.6 and 7.8 m/s sliding velocities are presented in Figs 6 and 7. In general, there is no remarkable effect of sliding distance on the $W_s$ for all the materials except the NP which shows an increase in $W_s$ with the increase of sliding distance at lower sliding velocity. T-OPRP shows lower $W_s$ when compared with others. This could be due to the same reasons as those mentioned for the POD technique, i.e. the interfacial adhesion characteristics determined the wear performance of the OPRP composite. The $W_s$ of T-OPRP is lower than that of NP by about 65–75 per cent. Meanwhile, untreated oil palm fibres give poor support to the polyester.

The friction coefficients of NP and T-OPRP composites exhibit the same trend, and the averages at 5.6 and 7.8 m/s sliding velocities are about 0.15 and 0.5, respectively. The UT-OPRP composite has lower friction coefficient compared with others. The lower friction coefficient for the untreated OPRP composite could be because of the ease of material removal (i.e. low resistance).

The sharp increase in the $W_s$ of NP (Fig. 6(a)) could be due to the transition of the adhesive wear to the three-body abrasive. Meanwhile, the results of the T-OPRP and UT-OPRP are consistent with other published works [3, 11], where there are no remarkable effects of sliding distance on the $W_s$. Further explanation is given with the assistance of SEM micrographs of the worn surfaces (Figs 8 to 10).

Figure 8 shows the micrographs of the T-OPRP composite after tests at different sliding distances at 5.6 m/s sliding velocity. It seems that after 14 km sliding distance the treated oil fibre is not highly damaged and still adheres well to in the matrix. Furthermore, at a longer sliding distance (28 km), it seems that the core of the bundle was partially filled with polyester. This means that, during the preparation process of the composite, the washing out of the outer layer of the oil palm fibres allowed some of the polyester to occupy the empty spaces in the bundle. This supports the idea that treating the oil palm fibres reduces the porosity of the bundles (i.e. enhances the wear properties [17]). In addition to that, it prevents the debonding of the fibres leading to reduced removal of the material at the surface.

On the other hand, for UT-OPRP (Fig. 9), at the very short sliding distances of 0.35 and 0.7 km, the untreated oil palm fibres are already debonded. Additionally, in Fig. 9(b), the cross-section of the untreated oil palm fibre indicates that the bundles of the untreated oil palm fibres are empty. This leads to damage of the fibre after increasing the sliding distance, as shown in Fig. 9(c) (i.e. the fine fibres in the bundle seem to be torn). Moreover, the resinous regions close to the fibres are damaged. This could explain the poor results of UT-OPRP compared with the high performance of T-OPRP.

Figure 10 shows the micrographs of the worn surface of NP. Scratches and abrasive wear nature can be observed on the worn surface. This could be because of the increase of the counterface roughness caused by the movement of debris in the interface (Table 3). This could be the reason for the high removal of NP compared with others (Fig. 6).

### Table 2 Counterface roughness (μm Ra) after the tests using POD

<table>
<thead>
<tr>
<th>Materials</th>
<th>Before test</th>
<th>After test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neat polyester</td>
<td>0.09–0.12 μm Ra</td>
<td>0.50–0.57 μm Ra</td>
</tr>
<tr>
<td>Untreated oil palm fibre-reinforced polyester</td>
<td>0.09–0.12 μm Ra</td>
<td>0.22–0.24 μm Ra</td>
</tr>
<tr>
<td>Treated oil palm fibre-reinforced polyester</td>
<td>0.09–0.12 μm Ra</td>
<td>0.15–0.19 μm Ra</td>
</tr>
</tbody>
</table>

### Table 3 Counterface roughness (μm Ra) after the tests using BOR

<table>
<thead>
<tr>
<th>Materials</th>
<th>Before test</th>
<th>After test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neat polyester</td>
<td>0.09–0.12 μm Ra</td>
<td>0.50–0.57 μm Ra</td>
</tr>
<tr>
<td>Untreated oil palm fibre-reinforced polyester</td>
<td>0.09–0.12 μm Ra</td>
<td>0.18–0.21 μm Ra</td>
</tr>
<tr>
<td>Treated oil palm fibre-reinforced polyester</td>
<td>0.09–0.12 μm Ra</td>
<td>0.14–0.18 μm Ra</td>
</tr>
</tbody>
</table>

The counterface roughness, after and before the tests, are listed in Table 3. It seems that NP greatly increased the roughness of the counterface than other composites. The T-OPRP composite has low effects on the counterface roughness. The high increase in the counterface roughness when NP was tested could be because of the removed debris acting on the interface, which damaged both the composite surface and the counterface. Meanwhile, reinforcing the polyester with treated oil palm fibres strengthens the surface at the sliding and enhances the surface characteristics. This results in less damages to the counterface compared with the NP.

It has been mentioned in a previous work by the present authors [3] that the mechanical properties of the polyester composite are highly enhanced with the addition of oil palm fibres, especially the treated fibres. In particular, the hardness of polyester composites, based on the treated fibres, was significantly higher than that of NP. For the current tribological results, it can be seen that there is a correlation between the hardness of the materials and their tribological characteristics. The high wear performance of the polyester composite, based on treated oil palm fibres, is because of the high hardness of the composite surface. In other words, the higher the hardness of the composites, the better the wear performance. On the other hand, the low hardness of NP could be the reason for its low wear performance.

In comparison with the published works, two points could be raised.

1. Previously, the UT-OPRP composite was tested under dry contact conditions [3]. The effects of thermo-mechanical loading highly damaged the composite surface. The wear mechanism was dominated by pullout, bending and tearing of fibres. However, for the present work under wet contact conditions, the cooling process via water prevented the pullout of fibres, i.e. the damages were lower compared with dry contact conditions. Besides, UT-OPRP under dry contact conditions showed higher $W_s$ values when compared with the present work (under wet contact conditions).

2. In comparison with the previous published works on synthetic fibres reinforced thermosets [9], oil palm fibres offered less attack on the counterface roughness (increased from 0.09 to 0.15 μm Ra). At the same operating parameters, glass fibres in polyester composites increased the counterface roughness from 0.09 to 1.25 μm Ra [9]. Additionally, in terms of wear performance, the chopped strand mat glass-fibre-reinforced polyester (CGRP) composite showed higher $W_s$ when compared with the T-OPRP (Table 4) where the removed broken glass fibres highly damaged the rubbing surfaces [9]. For the friction coefficient, there are not much differences in the values of all the materials.

3. There is a correlation between the composite hardness and the wear properties. The high hardness of the material leads to high wear performance.

4. Treated oil palm fibres have a potential to replace glass fibres/polyester composite for tribological applications.

4 CONCLUSIONS

From the experiments carried out, the following conclusions can be drawn:

1. The wear characteristics of the polyester composites are significantly controlled by the interfacial adhesion characteristic of the oil palm fibres and the test technique. Wear properties of the polyester composites are highly enhanced with the addition of treated oil palm fibres, i.e. from 35–52 per cent to 65–75 per cent, when the materials were tested using the POD and BOR techniques, respectively.

2. The modifications that occurred on the stainless steel counterface roughness played an important role in controlling the wear characteristics of the material, especially the NP. Testing the NP and the UT-OPRP composites caused higher counterface roughness compared with the T-OPRP composite.

3. Treated oil palm fibres/polyester composite for tribological applications.

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