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Wear resistance and friction behavior of thermoset matrix reinforced with *Musaceae* fiber bundles

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ABSTRACT

Fiber bundles from agricultural residues are promising sources of reinforcement for composite materials due to their technical and economic advantages. This work aims to compare the effect of variation of the fiber size, resin type and curing agent on friction and wear behavior of polymer matrix composites reinforced with fiber bundles obtained from *Musaceae* rachis. A pin on disc test equipment was used to study sliding of composites and steel as counter body with fixed test parameters. SEM images were used to identify the wear mechanisms. Results show that the wear resistance of composites is better than neat resin and increases when fiber size is reduced. The main wear mechanisms evidenced in all samples were adhesion, surface fatigue and crazing.

1. Introduction

Composite materials have been used to replace components traditionally fabricated with common materials with higher weight. The main reinforcements for polymeric composites are synthetic fibers like glass or carbon. Some researchers have shown [1,2] that an advantage of these composites is the improvement of the wear resistance behavior of the neat resin. It has also been shown that the fiber orientation is an important feature in the wear resistance [2–6]. These composites have the main wear mechanisms such as fracture, fibers debonding, as well as micro and macro cracking [1–6].

A strong trend in the world for environmental sustainability and the material availability issues is leading researchers all over the world to look for more environmentally friendly composites, using natural fibers in traditional synthetic resins or using biopolymers [7]. The use of these reinforcements may reduce the environmental influence of the polymers used [7]. These composites have low cost due to many of the fibers are obtained from crops residues. Their low density, flexibility during the processing, high specific strength and stiffness which is comparable with those of the composites reinforced with glass fibers [8].

The use of natural fibers has been studied in order to assess the suitability of these materials to replace the synthetic fibers in tribological applications. Early works with polyester resin and unidirectional cotton fibers were performed by Eleiche and Amin [9] who demonstrated that the use of the fibers decreased the wear rate as the fraction of reinforcement increased and depending on the orientation of the fiber respect to the sliding direction. In this case the fiber tips increased the diameter and spread out covering part of the resin preventing the composite from severe wear as is shown by the neat resin.

El-Tayeb has worked extensively with sugarcane fibers to reinforce polyester resin, studying dry sliding and abrasive wear [10–12]. Abrasive wear [10,11] shows that wear resistance of the composites is highly dependent on fiber size in the case of randomly distributed chopped fiber, and on the orientation of the fiber related to the sliding direction. However, in this abrasion case the best behaved composite was the glass fiber composite [11]. The sugarcane fiber reinforced composites have potential for tribological applications [10]. The wear mechanisms that El Tayeb found in these works include severe plastic deformation, microploughing, fibrillation, microcutting, and deterioration of the matrix among others. [10–12]

Similar dependence on the fiber orientation or size was observed in dry sliding of the sugarcane fiber reinforced composite. In this process, the polymer formed a layer that protected it from more damage giving it more wear resistance than the glass fibers reinforced composites. This lead El-Tayeb to conclude that the composites of polyester reinforced with sugarcane fibers can be competitive with those reinforced with glass fibers. [12].

These same results of wear resistance and wear mechanisms of natural fiber composites compared with the glass fiber reinforced ones

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have been found by other researchers such as Nirmal et al. [13–15]. He used chemically treated betelnut fibers as reinforcement, also appreciating the back transfer layer that reduced the wear previously mentioned by El-Tayeb [12].

Yousif using coir fibers [16] also found that this composite enhances the sliding wear performance of the neat resin. These results were also achieved by Yousif and El-Tayeb using treated and untreated oil palm fibers [17]. In this study they concluded that the treated fiber behaved better than the untreated one; both of them had better wear resistance than the neat resin.

All these successful studies with natural fiber for reinforcing polyester lead the authors to explore the *Musaceae* fiber bundles to reinforce polyester and vinyl ester resins for sliding wear applications such as gears or electrical isolation material, among others. *Musaceae* are a very important crop in the Uraba region in the Northern part of Antioquia Department – Colombia. The rachis of these plants are agroindustrial residues that have non-commercial use, but have a considerable amount of fibers in them that may be used as reinforcement for composite materials [18,19] such as those used in the present work. To the best of the authors' knowledge, no work has been found with the *Musaceae* fiber bundles to reinforce these resins for tribological purposes.

This work assesses the effect of the variation of the composite raw materials (resin type, hardener and fiber size) in the coefficient of friction and the wear behavior of these materials in sliding contact with carbon steel as counter-body. The behavior of the composites was compared with the one of the neat resin. The authors did not find any other work that has evaluated the effect of using different peroxides as hardeners in the wear behavior of the composites. This work is the first to do such comparison.

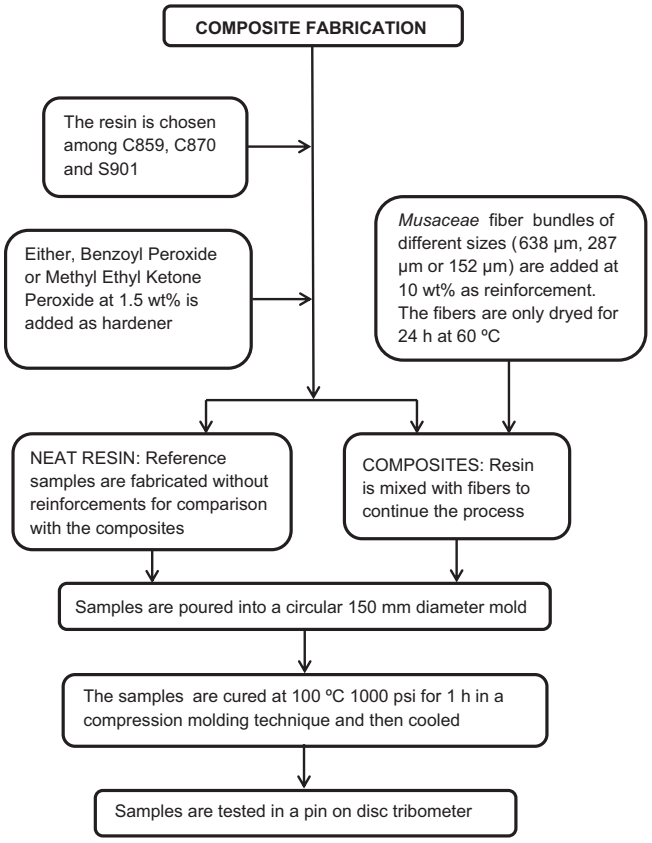

Fig. 1. Scheme of the composites fabrication process.

2. Experimental

2.1. Materials

Matrices: Resins correspond to: an orthophtalic polyester resin, reference Cristalan 859, denoted in this work as C859; one isophtalic polyester resin, reference Cristalan 870, denoted in this work as C870; and one vinyl ester resin, reference Swancor 901-3, denoted in this work as S901. These resins did not contain cobalt or any other promoter. Hardeners used in this work were methyl-ethyl-ketone peroxide (MEK) and benzoyl peroxide (BPO). Resins and hardeners used in this work were kindly supplied by ANDERCOL S.A.

Fiber bundles: The fiber bundless used in this work were extracted from the rachis of Colombian *Musaceae* plants. The plants are *Cavendish Valery* variety, and were this fiber bundles kindly supplied by BANACOL S.A.

The fiber bundles were delivered milled and a process of sieving was used to separate the fibers. Only those with an average fiber length of 638 μm, 287 μm and 152 μm were taken. These sizes were chosen because there were the 3 sizes of higher percentage on the sieving process in ANSI mesh size corresponding to those retaining in mesh 30, 50 and passing mesh 100. The fibers were not submitted to any chemical or physical treatment before being used in the composites. They were, however, dried for 24 h at 60 °C in order to eliminate any residual humidity that the fiber bundle may have held. The fiber bundles were then stored in sealed containers until their use in the composites.

2.2. Composites fabrication

The composites fabrication process is schematized in the Fig. 1 and is as follows.

Eighty grams of resin was poured into a pot with 1.5 wt% of hardener, and mechanically stirred with a NIPPO 5 speed handheld electric mixer in the second speed. Then, 10 wt% of fibers were added and the stirred for about 5 min. The mixture was then poured into a circular steel mold of 150 mm in diameter and 3.5 mm of thickness obtaining a uniform distribution of the blend. By using the BMC fabrication technique the mold was closed and compressed to 1000 psi with a hydraulic jack and the plates compressing the mold were heated to 100 °C. 1 h, then the heat was turned off and the mixture allowed to slowly cool for another hour. The pressure was then released and the plate unmolded.

Plates made of neat resin of each reference and each hardener, were also fabricated in order to have comparisons with the composites in the tribological behavior.

The Table 1 presents a summary of the Rockwell R hardness of all the composites and the neat resin plates fabricated in this work.

2.3. Tribological testing:

Sample preparation: Five (5) samples, 9 mm each, were cut and affixed to a 10 mm long metallic pin with cyanoacrylate contact adhesive. Samples were machined to match the pin diameter of 6.3 mm (1/4 in.) and the front of the pin was face turned in order to have a parallel face with the counterbody. A pin scheme is shown in the Fig. 2.

Tribological test: The tribological test was performed using a pin on disc machine as schematized in Fig. 2. Parameters of the experiment were fixed with speed: 200 m min^{-1}, distance: 3 km, normal load: 4.9 N.

A 1040 steel disc was used as a counter-body. In Table 2 is presented the chemical composition of the counter-body. The disc was machined after each run of the test with exactly the same turning parameters to assure the same surface finish with a roughness of $Ra = 5.997 \pm 0.32$ μm and $Rq = 7.494 \pm 0.41$ μm. These roughness parameters were measured with a Mitutoyo Surt test SV 3000 roughness

Table 1
Summary of the Rockwell R hardness values of all, the composites and the neat resin, with their standard deviation.

Rockwell R hardness						
Hardener	**MEK**			**BPO**		
Resin	**C859**	**C870**	**S901**	**C859**	**C870**	**S901**
Neat resin	119.6 (± 4.67)	118.2 (± 3.42)	118.6 (± 3.21)	126.3 (± 0.58)	121.8 (± 3.86)	109.5 (± 4.20)
152 μm	95 (± 4.11)	119 (± 3.62)	110 (± 5.66)	122.7 (± 0.58)	120.7 (± 2.52)	119.3 (± 2.31)
287 μm	78.92 (± 19.83)	72.84 (± 5.48)	63.07 (± 16.06)	105.6 (± 6.52)	112.6 (± 3.21)	77.18 (± 6.14)
638 μm	98 (± 5.32)	90.14 (± 13.79)	94.28 (± 7.31)	107.5 (± 2.35)	97.6 (± 1.40)	75.2 (± 1.66)

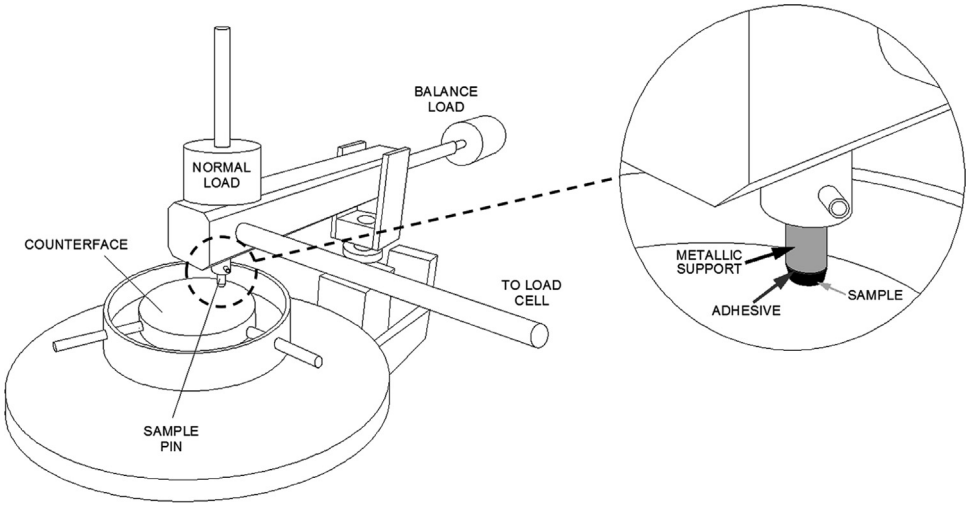

Fig. 2. Scheme of the Pin on disc machine used in this work.

Table 2
Chemical composition of the steel used as counter-body in the tribological test.

Fe	C	Si	P	S	Mn	Others
97.61% (± 0.018)	0.440% (± 0.0062)	0.344% (± 0.0063)	0.030% (± 0.00082)	0.028% (± 0.0039)	0.775% (± 0.0034)	0.773%

station with a 2 μm radius stylus tip according to the standard ISO1997 with sample length of 5 mm, a cut-off value of 2.5 mm and using 3 measurements in the disc.

The counterbody was set in motion and when it reached constant speed the pin was slowly put in contact with the disc and the clock was started for friction force measurements. The samples were weighed before and after each test to assess the mass loss with a balance KERN ABT 220-5DM with 0.01 mg of error.

2.4. SEM images

In order to assess the wear mechanisms of the composites, worn pin surfaces were observed with a scanning electron microscope JEOL JSM-5910LV. All the samples were covered with a thin layer of gold to ensure electrical conduction.

2.5. Estereo microscope images

To evaluate the counterbody conditions after the test, images were taken with a stereo microscope OLYMPUS SZ60-PT and with a camera CANON EOS REBEL T5i mounted in the photo tube.

3. Results and discussion

3.1. Behavior of the coefficient of friction in time

A typical curve of the coefficient of friction (COF) as function of the time for the composites and the neat resin is presented in Fig. 3. Shown are the curves of the C859 resin hardened with MEK (3a) and BPO (3b). The C870 and S901 resins showed similar behavior to the one presented.

According to the results, the COF values range between 0.25 and 0.45. These values are of the same order of magnitude for those presented in the literature for dry sliding wear in other composites of polyester resin reinforced with some other natural fibers [14,16,17] or glass fibers [2,4,6] using a steel counter-body. This may indicate that the resin is the main responsible for the coefficient of friction of the composite against steel, while fiber has a secondary role.

Results presented in Fig. 3 show that within the range of the tribological test, the behavior of the COF is stable for all the samples tested, with no severe changes in the curves. This may indicate that no severe stick and slip process is taking part in the system. Nevertheless, oscillating behavior present in the neat resin samples and to lesser extent the composites with the 152 μm fiber length has been found in the literature [2,14,17] and could be

considered typical. This oscillatory phenomenon may be due to adhesion in the asperities of the interacting surfaces as has been reported by Viáfara and Sinatora [20] using a pin on disc configuration where the pin and the disc were metallic, having the pin less hardness than the discs. Similar mechanisms take place in the system studied here since the polymeric pin has also lower hardness than the counter face disc. This behavior indicates that the use of fibers of sizes over 287 μm attenuates the oscillation of the coefficient of friction of polyesters and vinyl ester resins.

Composite samples hardened with BPO showed an evident reduction in the average value of COF of the composites compared with the neat resin. Their curves overlap showing that they have a very similar behavior among them no matter the size of the reinforcement throughout the testing period. Meanwhile, the composites hardened with MEK did not show the aforementioned behavior. However, according to the data and standard deviation presented in Table 3 there are no statistical differences among any of the coefficients of friction of these composites. This fact reinforces the conclusion that the resin holds the main role in the friction and the polyester and vinyl ester resins behave similarly, no matter if they are hardened with BPO or MEK.

3.2. Behavior of the wear resistance with the fiber size

Fig. 4 shows specific wear values for all the composites tested comparing the fiber size of the reinforcement. Fig. 4a shows the samples hardened with MEK and the b the samples hardened with BPO. Table 4 presents a summary of the percentages in the reduction of the mass loss comparing the composites with the

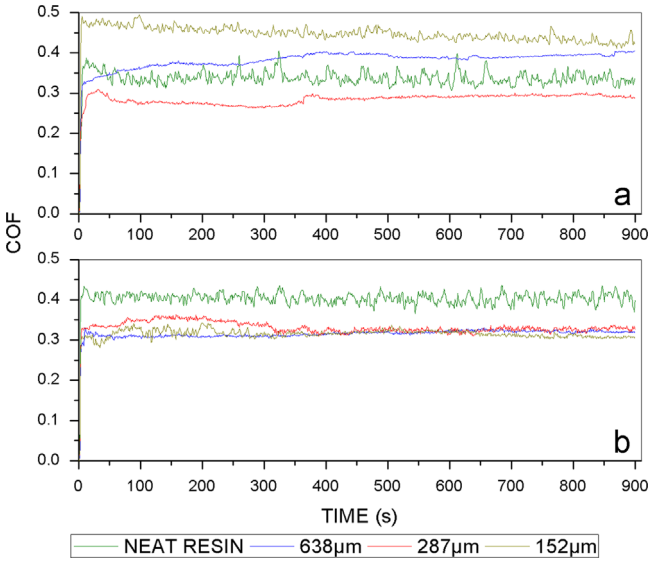

Fig. 3. COF vs Time of the composites fabricated with C859 resin. (a) Polyester hardened with MEK and (b) Polyester BPO.

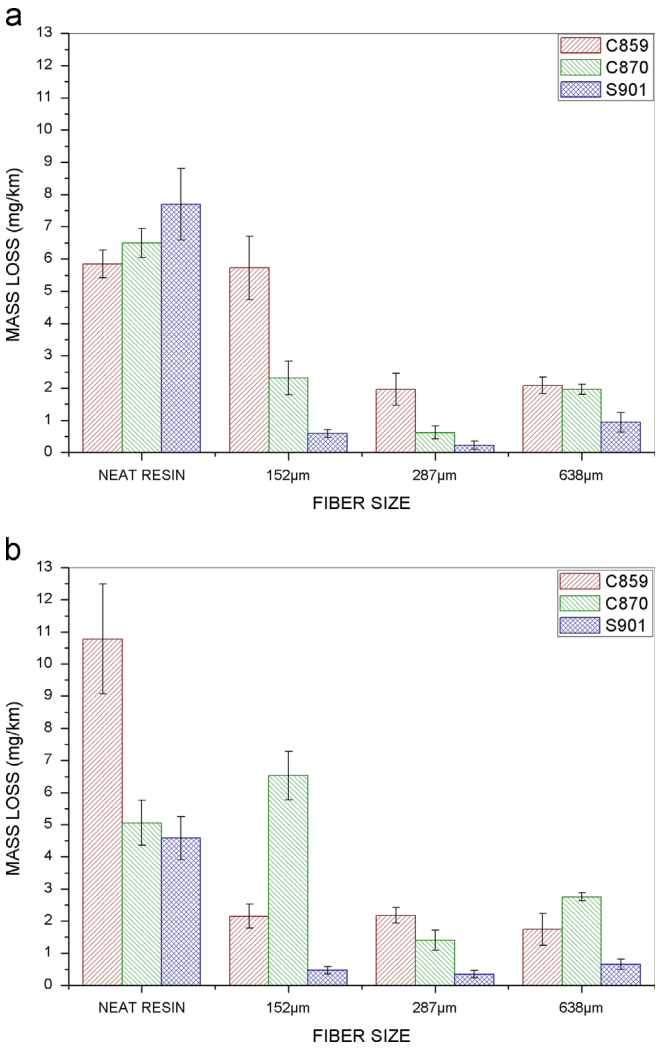

Fig. 4. Specific wear comparison of the composites tested in this work. (a) Resin hardened with MEK and (b) Resin hardened with BPO.

Table 4
Summary of the wear variation of the composites compared with the respective neat resin. Negative values in the table mean that the composite had lower wear resistance than the neat resin.

Mass loss						
Hardener	**MEK**			**BPO**		
Resin	**C859**	**C870**	**S901**	**C859**	**C870**	**S901**
152 μm	2.10%	64.34%	92.39%	80.04%	−29.15%	89.82%
287 μm	66.39%	90.46%	97.02%	79.83%	72.39%	92.57%
638 μm	64.40%	69.85%	87.79%	83.83%	45.57%	85.71%

Table 3
Summary of the average COF values of all, the composites and the neat resin, with their standard deviation (SD).

Coefficient of friction						
Hardener	**MEK**			**BPO**		
Resin	**C859**	**C870**	**S901**	**C859**	**C870**	**S901**
Neat resin	0.34 (± 0.06)	0.31 (± 0.06)	0.31 (± 0.10)	0.40 (± 0.11)	0.41 (± 0.04)	0.39 (± 0.10)
152 μm	0.45 (± 0.05)	0.40 (± 0.13)	0.37 (± 0.05)	0.31 (± 0.09)	0.32 (± 0.10)	0.32 (± 0.10)
287 μm	0.28 (± 0.06)	0.35 (± 0.13)	0.25 (± 0.05)	0.33 (± 0.04)	0.38 (± 0.08)	0.30 (± 0.09)
638 μm	0.38 (± 0.12)	0.29 (± 0.05)	0.31 (± 0.09)	0.31 (± 0.03)	0.36 (± 0.04)	0.29 (± 0.07)

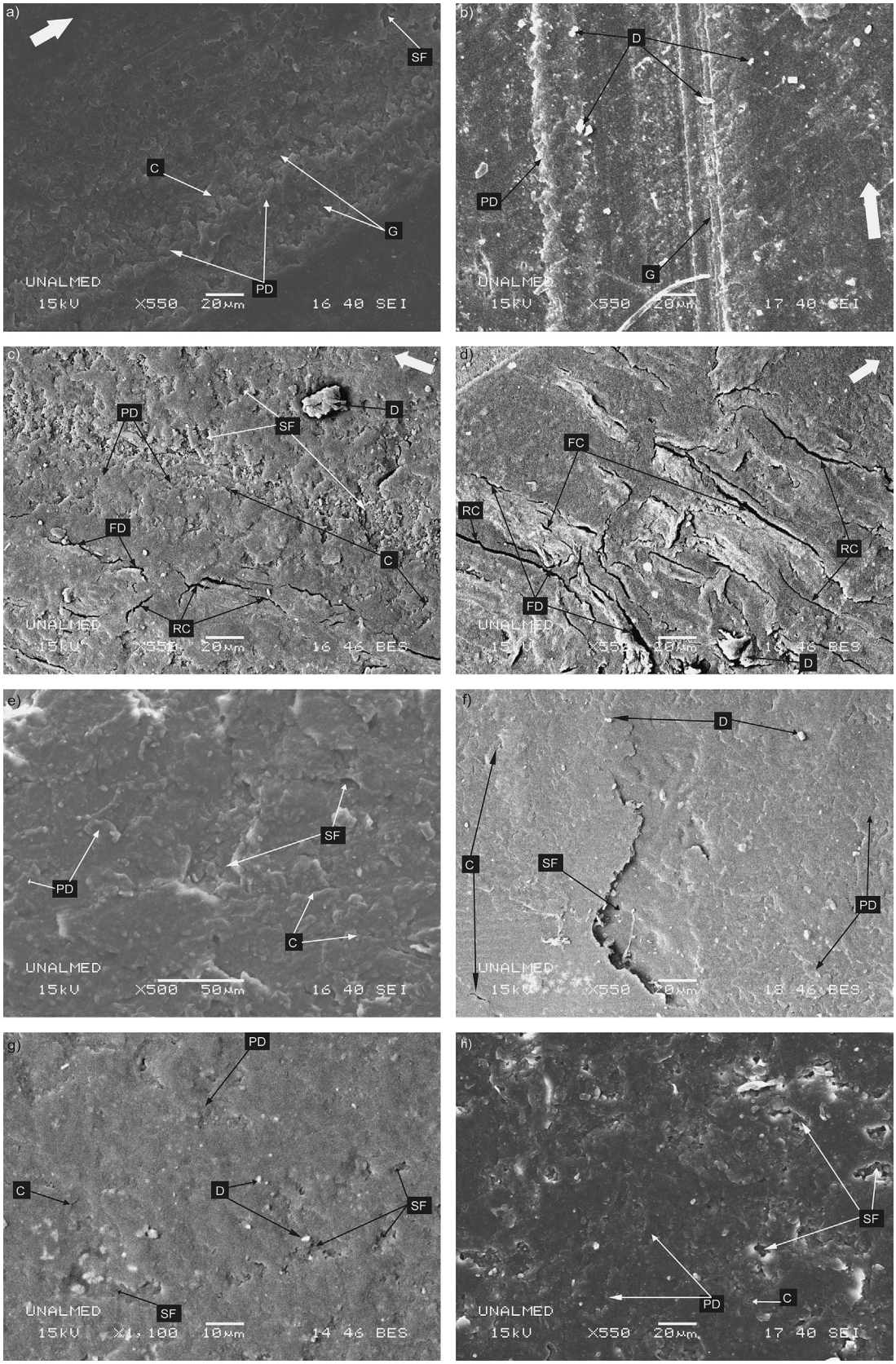

Fig. 5. SEM micrographs. Thick arrow shows the sliding direction. (a) S901+MEK+638 μm fiber length. (b) S901+BPO+287 μm fiber length. (c) S901+MEK+638 μm fiber length. (d) S901+MEK+638 μm fiber length. (e) S901+BPO+638 μm fiber length. (f) S901+MEK+287 μm fiber length. (g) S901+MEK+Neat Resin. (h) S901+BPO+287 μm fiber length. The meanings of the letters are listed in the Table 5.

neat resin, obtained using the expression:

$$\%\text{Mass loss reduction} = \left(1 - \frac{\text{mass loss}_{\text{neat resin}}}{\text{mass loss}_{\text{composite}}}\right) * 100$$

Results showed an increment of specific wear resistance in all the composites compared to the neat resin. The only exception is the composite of C870 resin, which hardened with BPO and reinforced with 152 μm of fiber length. The mentioned composite lost about 30% more mass compared to the neat resin. The composite of C859 resin hardened with MEK and reinforced with 152 μm of fiber length presented the least weight loss reduction with just 2.10%.

For all the composites, those with the reinforcement size of 287 μm presented the best specific wear resistance.

The specific wear resistance of the composites fabricated with the resin C859 hardened with BPO was the same no matter the size of the reinforcement but is about 80% higher than that of the neat resin.

Vinyl ester resin composites (S901) reduced over 85% the specific mass loss in all the samples compared with the neat resin. They were the samples with the largest increase in the specific wear resistance compared with the polyester samples. When the samples with the same size of fiber are compared, within the vinyl ester resin; samples hardened with MEK had larger reduction in mass loss than the composites hardened with BPO. Thereby, the composite with the best specific wear resistance is the one fabricated with the vinyl ester resin, hardened with MEK and reinforced with 287 μm fiber length.

The main wear mechanism in the neat resin is the surface fatigue evidenced by the crack growing, as will be discussed in the next section; however the presence of the fibers in the composites may be the responsible to slow down this crack growing. This appears to prevent or restrict wear of almost all the composite materials.

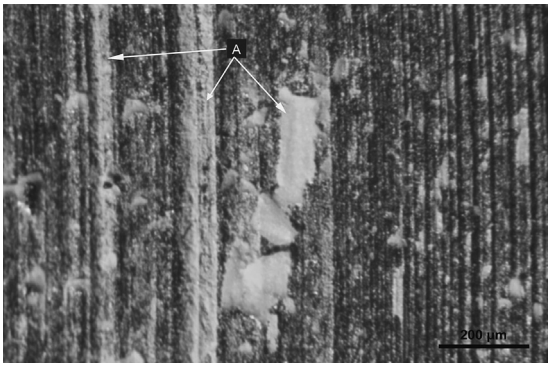

Fig. 6. Stereo micrograph of the counterbody used in the wear tests.

3.3. Composite surfaces microscopy analysis

The Figs. 5 and 6, show the evidences of wear mechanisms and surface characteristics that are present in the samples and in the counter-body. These are described in Table 5, where the first column stands for the abbreviations shown in the micrographs.

The surface fatigue, as evidenced by the plastic deformation, crazing and the scars shown in the micrographs of the Fig. 5a, c, e–h. this is the main wear mechanism of the composites tested. The presence of surface fatigue in this kind of tests may be due, as mentioned by Terheci [21], to a compressive and tensile stresses suffered by the surface of the material in the test. This wear mechanism has also been reported by Betancourt et al. [22] in a composite that used the same fibers used here but treated with pyrolysis and reinforcing a different thermoset polymer.

The phenomenon of crazing is shown in the micrographs of the Fig. 5a, c, e–h. Crazing has been reported to be an important surface damage phenomenon linked to the plastic deformation for brittle polymers [23,24] and has also been reported to be a wear mechanism by Betancourt et al. [22].

Grooving is also present as a surface damage phenomenon associated to the surface fatigue. This may be due to hard particles formed from wear debris of the resin that have been trapped in the interface. The grooves may be seen in the micrographs of Fig. 5b–d. These marks may also be due to the asperities of the counterface, who may be too large judging by the value of the roughness previously mentioned.

Important features could be appreciated in stereo microscope scale as is shown in Fig. 6. Micrograph exhibits a part of polymer added to valleys of the steel counter-body surface, filling them and reducing the size of steel asperities. However, image shows roughness and surface features of counter-body including pathways of turning process. The debris of the polymer does not fill completely the canyon and the higher hills could cause the grooving damage appreciated in the composite surfaces shown in the micrographs on Fig. 5. In addition, a transfer layer of the polymeric matrix to the counter body can be appreciated. This layer has been described by Hutchings [25] and Bhushan [26], and has been evidenced in the works of El-Tayeb [12], Nirmal et al. [13] and Yousif [16].

El-Tayeb [12], Nirmal et al. [13], Yousif [16] mentioned in their work with natural fibers the formation of the back transfer layers of the resin to the composite reinforced with natural fibers preventing the wear of the samples. This back transfer layer is not evidenced in the micrographs presented in the Fig. 5.

Fig. 5c,d shows that other important evidence of surface fatigue are the growing of cracks. This crack may have grown due to the break of the crazes of the crazing, becoming micro cracks as those

Table 5
Summary of the wear mechanisms and surface damage phenomena identified in Fig. 5.

Convention	Meaning	Description	References
A	Adhesion	(Wear mechanism) When two surfaces are in sliding one to each other, asperities are bonded then detached due to the shear stresses of the sliding	[22,26,36,37]
SF	Surface fatigue	(Wear mechanism) the cycles of load and unload induce the formation of cracks that leads to breaking up of the surface removing fragments of the material leaving scars, also known as pits, in the surface.	[21–23,26]
C	Crazing	Is a form of surface fatigue localized that occurs in brittle polymers chraracterized by crazes who are small cracks where the two edges are bridged by nanometric sized fibrils. When it leads to mass removal from the surface can be considered a particular wear mechanism in polymers.	[22–24]
G	Grooving (plowing)	Material is removed from the surface by indentation and relative movement of asperities of the counter body	[6,7,14,25,26,31,38]
RC	Resin cracks	The fibrils of crazes break due to high stress levels in them and the crack grows	[3,24,26,27,32,39]
FC	Fiber cracks	Cracks within the fibers bulk. Also called micro fracture	[11,39,40]
FD	Fiber debonding	The adhesion of the untreated natural fibers to the polyester resin is poor due to the hydrophobicity of the later and the hydrophilia of the earlier	[28–35]
PD	Plastic deformation	Due to the pressure of the asperities of the counter-body there is a local plastic flow and the material is displaced and piled up to the sides of the grooves	[4,5,11–14,17,25–27,39]
D	Debris	Particles removed from the sample due to the grow and intersection of the cracks	[7,10–13,16,17,27,32,39,40]

Table 6
Comparative summary of the COF, wear resistance and wear mechanisms identified with natural fibers in thermoset resins in the literature, including the present work.

Author	System used	Tribometer	COF	Wear	Wear mechanism
Eleiche and Amin [9]	Unidirectional cotton/polyester	Pin on disc	~0.5–0.75	~0 to > 170 (E−8) g/cm	Not mentioned
El Tayeb [12]	Sugar cane/Polyester	Pin on disc	0.01–0.025	2 to 10 (E+07) Nm/m^3	Not mentioned
Yousif [16]	Coir/polyester	Block on disc	0.5–1	10 to > 30 (E−5) mm^3/Nm	Deformation microploughing fiber debonding
Yousif and El Tayeb [17]	Untreated and untreated oil Palm/polyester	Block on disc	0.2–1	< 1 to > 2.5 (E−5) mm^3/Nm	Fiber debonding deformation
Nirmal et al. [14]	Betelnut/polyester	Block on disc	0.4–0.7	~1 to > 20 (E−5) mm^3/Nm	Microcracks plastic deformation debonding and detachment of fibers
Present work	*Musaceae*/polyester or vinyl ester	Pin on disc	0.25–0.45	0.23 to 10.72 mg/km	Adhesion surface fatigue

reported in the literature [13,27]; and its subsequent growth having the cracks that are evident in the Fig. 5c–d.

A crack also may grow as the consequence of debonding of the fiber from the resin as observed in the micrographs of Fig. 5c–d. This result has also been reported by other researchers [11,12,14,16,17]. The interfacial adhesion of the fibers and the resin that can be appreciated on the micrographs, and mentioned earlier, may be due to the poor wettability of untreated natural fibers by polyester resins, which is a common borderline broadly reported in natural fibers composite systems [28–35].

The cracks present in the composites may have also come from the cracking due to a lack of cohesion within the fibers, which has also been reported in natural fibers in sliding conditions by other authors [11,32,33]. This cracks on the fiber, are shown in the micrograph of Fig. 5d.

The Table 6 presents a summary of a review of some of the authors that has worked with natural fibers and thermoset resins in wear studies. In the table the approximated coefficient of friction and wear rates are presented as well as the wear mechanisms reported by the authors and the tribometer used in their research. In this summary the results of the present work are included. According to this, the results of the present work show: a COF narrow range, wear behavior similar than reported by other authors and different wear mechanisms.

4. Conclusions

- The coefficient of friction of all the composites and resins tested was in the range of 0.25–0.5 which is within the typical range for these kinds of composites sliding against a steel counterface.
- Resin holds the main role in the friction behavior and all resins tested here behave similarly, no matter if they are hardened with BPO or MEK.
- The composites, in general terms, have lower specific mass loss compared with the neat resin showing that the reinforcement is suitable to enhance the polyester or vinyl ester resins for tribological applications because the fibers may be obstacles for the growing of cracks in the matrix.
- The vinyl ester resin (S901) had the biggest enhancement on specific wear resistance among all the composites; the hardener that best behaved comparatively was MEK, except in the case of the C859 resin that was otherwise. As for the fiber size, the samples with the bigger increment on the specific wear resistance were those reinforced with the fibers of 287 μm.
- The main wear mechanisms identified in the samples tested in this work were adhesion and surface fatigue, while crazing was also observed and related to localized mass loss from the surfaces.

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