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Morphological Characteristics and Wear Mechanism of Recycled Carbon Fibre Prepreg reinforced Polypropylene Composites

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ABSTRACT

The polypropylene (PP) reinforced with recycled carbon fibres (rCF) was successfully produced using a Haake internal mixer via melt compounding. The compounding was performed at 180°C, rotor speed of 50 rpm and compounding period of 10 minutes. The standard samples for the pin on disc testing were prepared using injection moulding. The effect of rCF filler loadings of 0.5, 1, 3, 5, 7, 10, 13, 15 and 20 wt% was studied for the tribological properties. The results were compared with 100% PP. The morphological behaviours for the effect of low and high fibre loadings were observed using scanning electron microscopy analyses. The composites with low carbon fibre loading of up to 3 wt% imposed higher resistance to dry sliding friction. In contrast, the increment of fibre loading at 5 wt% to 20 wt% decreased the wear rate of the composites due to patch film and transfer film formation. The wear mechanism of the composites for different fibre loading was graphically sketched from morphological observation. As the conclusions, the composites showed promising self-lubricating properties, capable of wear reduction with significant physical and mechanical properties.

Keywords:

Polymer composites, polypropylene, recycled carbon fibre, tribological properties, wear morphology

1. Introduction

Carbon fibre reinforced composites (CFRC) continue to play a critical role in the growth and development of various industries that require lightweight components. These engineered fibres and composites have been used in a variety of applications, including aerospace, automotive,

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construction, and energy, where their properties are most advantageous [1-2]. This is in response to the demand for weight reduction and increased stiffness [3]. However, their proliferation has raised concerns about the practices and strategies required to manage these costly engineered structures at the end of their valuable lives [4]. This situation highlights the critical need for more sustainable and efficient recycling methods for these products. This effort aligns with various industries' green and sustainability policies to enhance performance and preserve the environment. For this, a business must emphasise environmental stewardship and green generation as critical components of its social responsibility [5]. Additionally, it is to extend the product's life cycle and to pursue long-term business success. Similarly, the importance of managing dry carbon fibre scraps generated during the CFRC manufacturing process has grown in recent years.

Recently, the use of carbon fibre waste has been accepted as a prudent approach to enhancing carbon fibre's performance and as a green effort for waste management. There are three distinct methods for recovering CF: thermal, chemical, and mechanical [6-7]. Pyrolysis and fluidised bed pyrolysis are two thermal methods. Several methods, including solvolysis, acid digestion, and supercritical fluid solvolysis, are among the most widely used chemical methods for separating carbon fibre from composite materials. The mechanical method is primarily based on shredding, grinding, or milling the CFRCs into small pieces.

Polypropylene (PP) is a semicrystalline polymer that is widely used as a matrix material. It garners so much attention due to its one-of-a-kind combination of properties, affordability, and ease of fabrication [8]. Additionally, PP can be processed in various ways, including extrusion, multifilament, nonwoven, injection moulding, blow moulding, and profile extrusion [9]. PP has a high melting point of between 160 and 176 degrees Celsius and a density of approximately 0.946 g/cm³ (crystalline) and 0.855 g/cm³ (amorphous) [10]. Polypropylene (PP) is a highly versatile material used in automotive components, household appliances, and industrial applications. However, PP is rarely used in its pure form and is frequently combined with fillers or reinforcements to meet stringent engineering and structural specifications. According to Hanim et al. [11], adding fillers or reinforcements to PP frequently modifies its crystalline structure and morphology, resulting in property changes.

The purpose of this research is to determine the potential for using recycled carbon fibres as reinforcement in a polypropylene (PP) matrix, particularly for tribology applications. Additional enhancement of polymer composites' tribological performance can be accomplished by combining specific filler additions and fibre reinforcement, which demonstrated synergistic effects and governed the friction and wear of such hybrid composites. Nonetheless, recycled carbon fibres have been successfully used in a limited number of trials [4, 12]. However, research on the use of rCFs to prepare thermoplastic-based polymer-based tribomaterials is quite limited.

Carbon fibres incorporated into thermoplastics are expected to improve the composites' physical, mechanical, and tribological properties. According to Lin et al. [12], solid lubricants can be used to enhance the friction-reducing ability of polymer materials. They investigated the tribological properties of a hybrid PEEK/recycled carbon fibre composite. The incorporation of fibres into polymer matrices may have two effects: (1) lowering the temperature at the interface by reducing the interaction between asperities, where the film transfer will not adhere well, and detachments may occur; or (2) another possibility is that a substantial film transfer made of polymer and fibre debris may form on the metal surface. According to Chang and Friedrich [13], friction reduction occurs due to the fibre collection and distribution process on counterpart surfaces.

In comparison, the increase in friction coefficient can be explained reasonably by the gradual removal of the trapped fibre from the transfer film. Lin et al. [12] emphasised the critical nature of analysing worn surfaces and tribo-films formed on steel counterfaces. It demonstrates that the dominant mechanisms for enhancing tribological performance in the presence of hard submicron-

particles are unrelated to the type of carbon fibres used to reinforce the composite. Additionally, the patch film on the composites served as a separator at the interface, with its low thermal conductivity reducing the effect of temperature and thus the friction coefficient [14].

In this study, we examined the wear behavior of polypropylene composites with recycled carbon fibre reinforcement at rCF loadings of 0, 0.5, 1.0, 3.0, 5.0, 7.0, 10.0, 13.0, 15.0, and 20.0 weight percent (wt%). Scanning electron microscopy analysis of the worn surfaces reveals the formation of significantly stable transfer films, which aided in reducing friction and increasing the wear resistance of the materials during dry sliding. The wear mechanism is deduced from the morphologies to distinguish between the tribological behaviour of low and high rCF filler loadings.

2. Methodology

2.1 Materials

Polypropylene (PP) homopolymer (TITANPRO 6531, isotactic type; Lotte Chemical Titan Holding Sdn. Bhd., Johor Bahru, Malaysia) with a specified melt flow index 6 g/min was used in this research. A local aero composite company supplied waste carbon fibre prepreg. Scissors were used to cut the rCF into 5–10 mm pieces. The rCF was crushed in a pulveriser and sieved in a sieve shaker to obtain finer particles. The rCF was then immersed in acetone for an hour to remove any remaining impurities and resin trapped within the fibre preregs. After washing with distilled water and drying for 24 hours at 60 °C, the rCF was crushed to produce finer particles (Figure 1).

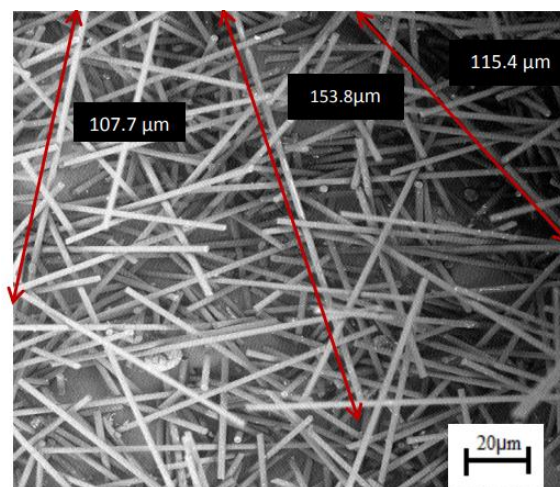


Fig. 1. SEM image of recycled carbon fibre

2.2 Sample preparation for tribological testing

Figure 2 depicts the sample preparation process up to the tribological test. The preparation of PP reinforced with rCF composites was carried out for 10 minutes in an internal mixer (Thermo Fisher Scientific [Waltham, Massachusetts], Haake Rheomix OS) set to 180 °C and 50 rpm. After allowing the PP/rCF compound to cool to room temperature, it was crushed into pellets using a crusher. Following that, the mixture was compression moulded for 15 minutes at 200 °C in a hot compression machine to produce a cylindrical shape for tribological testing following ASTM G99. Ten different compositions were created, containing 0.5, 1, 3, 5, 7, 10, 13, 15, and 20% wt rCF, as well as 100% PP as a control sample. Samples with a filler loading of less than 3% are considered low filler loading samples; samples with a filler loading greater than 10% are considered high filler loading samples.

The 3 wt% and 15 wt% loadings are used throughout this paper to represent samples with a low filler loading and a high filler loading.

C. Testing and analysis

The dry sliding wear test was conducted on a TR20LE pin-on-disc tester machine from Bangalore, India. The ASTM G99 wear test was used to determine the dry sliding wear of the fabricated composites. The samples were 6 mm x 28 mm in size. Before testing, the samples were polished with SiC emery paper 2000 grade to achieve a surface roughness of Ra, 0.6 m for close contact with the counter surface. The tests were conducted at a sliding velocity of 200 rpm, with a constant load of 16 N and a constant sliding distance of 2.261 kilometres. We determined the wear behaviour as a function of wear rates (Ws) and coefficient of friction (COF). The Ws of the sample was calculated using the equation stated in Abd Latiff et al. [15]. Following the test, the worn surface was examined under a 500x magnification using an EVO 50 scanning electron microscopy (SEM) from Carl Zeiss SMT (Oberkochen, Germany).

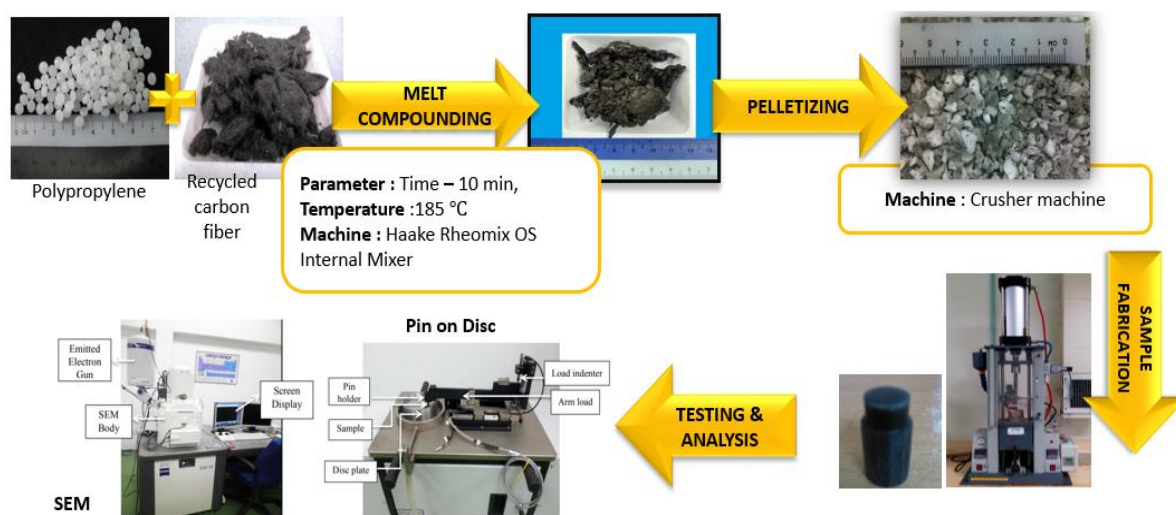


Fig. 2. The flow of the compounding, fabrication, testing and analysis.

3. Results

3.1 Wear Behaviour

Table 1 summarises the coefficients of friction (COF) and wear rates (Ws) associated with the effect of filler loadings and treatment. Meanwhile, the stability of the formed transfer film is demonstrated in Figure 3 by the graph of wear rates versus sliding time. In general, the COF increases as filler loadings increase from 0.5 to only 3 wt%. After that, it gradually decreases. However, the changes were negligible for composites treated with only nitric acid at low filler loading. COF exhibited a downward trend at filler loadings of 5 wt% and greater. The increase in shorter carbon fibres and almost particle-like carbon fibres distributed uniformly on the worn surface of the samples caused the COF to decrease with the percentage of fibre loading. The physical properties of the fibres enhanced the composites' lubricating mechanism and reduced friction between sliding components [16]. This observation is consistent with the SEM micrographs shown in Figure 4 in Subsection 3.1.1.

On the other hand, a significant number of fibres with a length greater than 10 m were observed at low fibre loading. This dimension increases the composites' resistance to friction stress. As a result, the COF increased at low fibre loading as the fibres imposed increased stress on the sliding action.

This is demonstrated by the presence of apparent cracks on the worn surface composites with 3% loadings.

Table 1
 Wear characteristics for the effect of filler loading

wt.% of rCF	Coefficient of Friction (COF)	Wear Rates $K=1 \times 10^{-6}$ mm ³ /Nm
0	0.3216	10.17
0.5	0.322	9.82
1	0.323	9.53
3	0.325	9.35
5	0.3123	6.14
7	0.3001	6.05
10	0.2743	5.63
13	0.2399	4.48
15	0.2112	3.52
20	0.2052	3.18

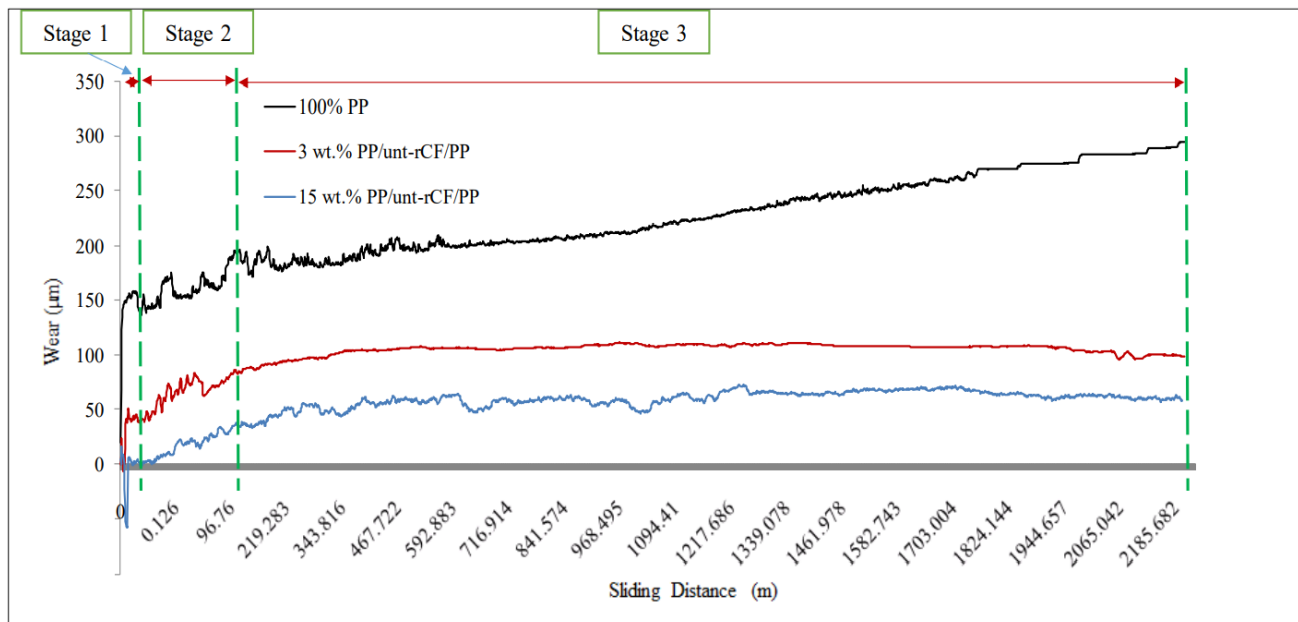


Fig. 3. Wear versus time for the effect of different fibre loading

Overall, the wear rates of all composites were less than those of pure PP. Carbon fibres were added to all samples to increase the wear resistance of the PP matrix. However, the observed differences in wear rates for low fibre loading composites at 0.5 wt% to 3.0 wt% were negligible. When 5% wt rCFs were added to the composites, a significant decrease in wear rates was observed, and the pattern gradually decreased thereafter. The transition from 3 to 5 wt% loadings could be considered the threshold at which the composites developed a self-lubricating mechanism [3]. The self-lubricating properties of this system are believed to be primarily due to its ability to deposit a thin protective layer of polymer onto the counterface referred to as transfer film [3, 16] and the formation of the polymer patch film on composites [3, 14]. Due to the weaker interaction between fibre and matrix, an early stable transfer film formed. The observed results were consistent with the morphological characteristics of the transfer films in composites with a fibre loading of 15% wt (refer Figure 6). The transfer film on the composite's counterpart was thin, uniform, and tenacious.

According to Figure 3, the wear behaviour of composites can be classified into three distinct stages: 1) initial wear due to static friction, 2) transition wear for the formation of the patch and transfer films, and 3) steady-state wear. At low fibre loading, strong adhesion between the fibre and matrix resulted in the formation of a later and more stable transfer film than composites with weaker interaction at high fibre loading [10].

3.1.1 Morphological Analysis of Worn Surfaces and Transfer Film

Pins on disk test observations of a worn surface provide direct evidence for studying the wear and tear mechanism. Figure 4 depicts the worn surface of the control sample (virgin PP without fibre loading). The worn surface can provide direct evidence for studying the wear and tear mechanism. The virgin PP generally exhibited a ductile failure in the micrograph, as the contact surface appears rougher after the test was conducted. It resulted from plastic deformation caused by shear and compression stresses during the sliding contact [10, 13]. Additionally, as illustrated in Figure 4 (a), there was the adherence of wear debris formed on the worn surface. As a result of the abrasive wear on the virgin PP, a significant amount of material was lost from the surface, gradually increasing with sliding time, as illustrated in Figure 3.

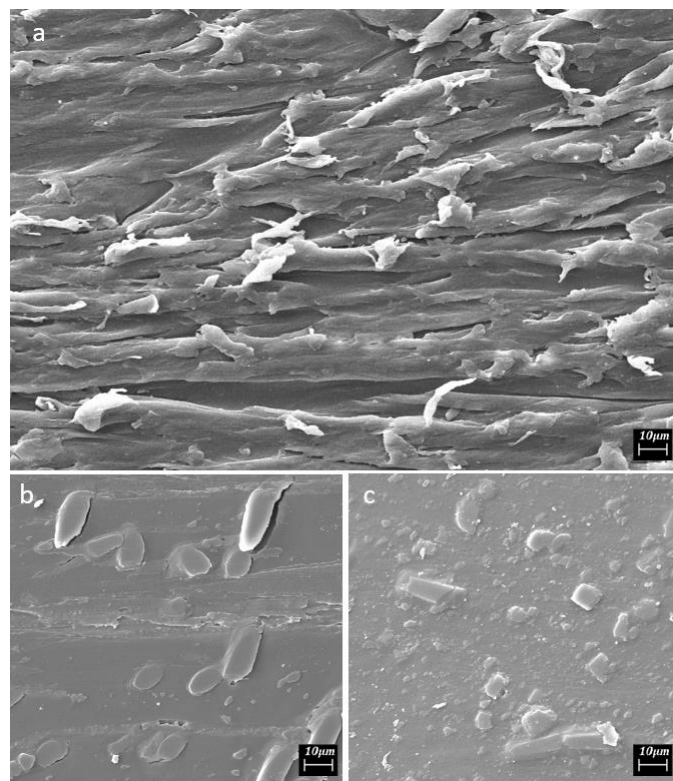


Fig. 4. SEM micrographs of worn surfaces of (a) virgin PP (b) PP/rCF composites at 3 wt. % of fibre loading and (c) PP/rCF composites at 15 wt. % of fibre loading

The nature of the interfacial bonding between the fibre and matrix dictates the significant properties of a composite under the wear and friction actions. The worn surface micrographs of various samples are shown in Figure 4 (a) and (b) at magnifications of 500x for two different fibre loadings of 3 wt% and 15 wt%. In general, the worn surfaces of composites with high filler loadings appeared to be smoother than those with a filler loading of 3 wt%, indicating a higher degree of brittle-like failure during abrasive wear. As the rCFs increased, the shearing stress on the composites

increased, resulting in patch films with shorter smeared CFs for a more efficient critical length under friction. On the worn surface, a significant number of rCFs appeared to be particle-like.

In comparison, longer rCFs of more than 10 μm were observed in composites with low fibre loading. Although the carbon fibres protruded from the rubbing surface in the composites, they appear to be tightly embedded in the PP matrix by the patch film at higher rCF loadings. This indicates that the fibres could support the normal load, which benefits the wear rate reduction [17]. In composites with low fibre loading, shearing microcracks were observed on the surface, either at the fibre-matrix boundary or at weak points at the fibre-matrix interface. Microcracks caused by carbon fibre detachment and shearing of the polymer matrix resulting from external mechanical forces can eventually result in wear. Due to the material's weakening, the removal of fibres from the matrix increases matrix wear. It should be noted that the more particle-like fibres performed better at reducing stress concentrations during rubbing, thereby reducing abrasive wear in the composites. The foregoing evidence indicates that fibres play a significant role in this process [18].

Carbon fibres can be separated from the matrix by steel asperities during aggressive micro-plowing and micro-cutting attacks—an increase in rCFs increases abrasive wear. Additional polymer matrices were transferred and peeled away from the composites as more weak points opened due to the protruding carbon fibre.

3.1.2 Wear Mechanism of Worn Surfaces and Transfer Film

Figure 5 illustrates the wear mechanism by comparing the worn surface at low and high fibre loading. At the low filler loadings, the worn surfaces were relatively smooth compared to when loading was high. Due to the reattachment of softening wear debris, the patch films obscured the rCFs on the worn surfaces. The peeling matrix and carbon fibres were then constrained because carbon fibres, which are particle-like, effectively support the load from the counterface, as evidenced by significant adherence between the carbon fibres and matrix [3]. According to Li and Xia [19], this result demonstrates that the excellent combination of carbon fibre and matrix reduces the rate of wear. As reported in our previous work [15], four product removal mechanisms predominate in these composites during dry sliding: (i) wear of the polymer matrix, (ii) abrasive wear of fibres, (iii) cracking, and (iv) spalling of fibres and separation of fibres from the matrix.

It is noted that the ability of a polymer to form a transfer film on the counterface has a significant effect on the friction and wear behaviours of a polymer sliding against metal [19]. Thus, the transfer films formed on the steel counterface by PP reinforced with rCFs may be used to explain the composites' friction and wear mechanisms [20]. Figure 6 illustrates the transfer films observed on the counterface for high (15 wt%) and low (3 wt%) fibre loadings following dry sliding testing. The composites had a less uniform, belt-like, and thick transfer film with relatively larger patchy flakes at low fibre loadings [3]. The absence of patch film at low fibre loadings increased the likelihood of rCFs fibre being ploughed out from the composite surfaces. This condition occurred due to the longer fibre's increased resistance to friction when it came into contact with the asperities of the steel counterface. The preceding events then increased matrix removal in the weak points created by removing the fibres. As a result, these composites exhibited increased wear rates.

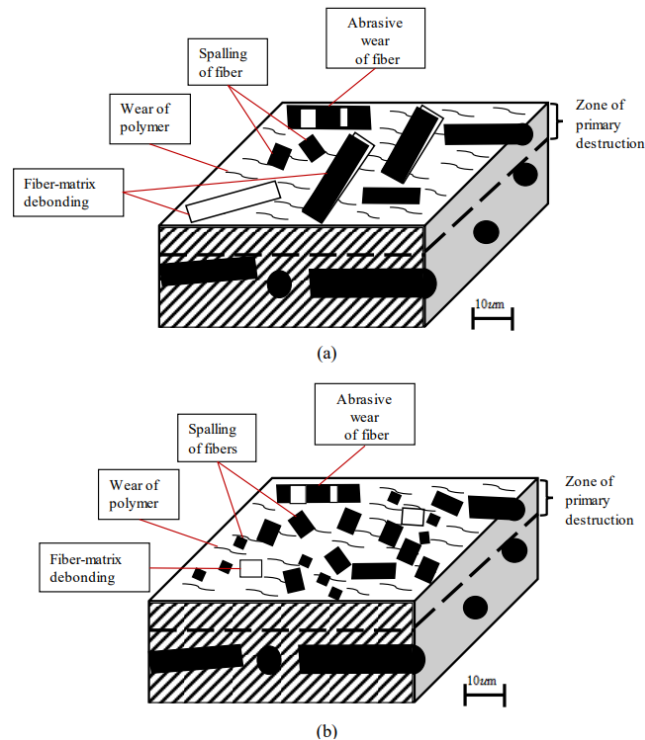


Fig. 5. Schematic model of wear mechanism for (a) low fibre loading composites and (b) high fibre loading composites [10]

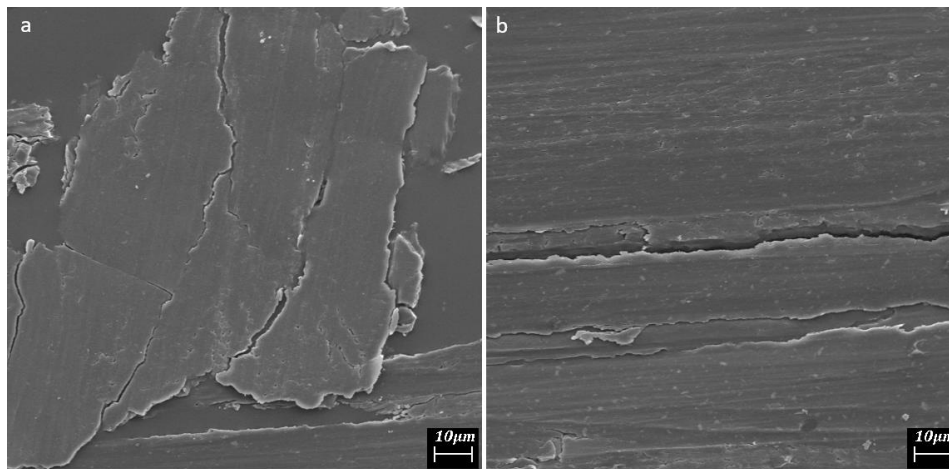


Fig. 6. Comparison between transfer films morphologies at low fibre loading (3 wt %) and high fibre loading (15 wt %)

In comparison, at a 15% fibre loading, thinner, more complete, and adherent transfer films were observed. The rubbing action resulted in the formation of smeared shorter fibres and more particle-like carbon fibres, which resulted in a reduction in bulk material removal for this composite due to self-lubrication increment [3]. In these composites, the weaker interaction between the fibre and matrix resulted in the premature removal of the PP matrix and spalled rCFs that served as a lubricant for the sliding materials. With increasing rCF content, the amount of smeared rCFs and patch film layer increases, resulting in the formation of a stable transfer film. As a result, these composites formed a transfer film that was more uniform, thin, continuous, and tenacious. Ye et al. discussed the strong correlation between transfer film characteristics and wear rate [16]. They concluded that

the high-quality transfer films associated with the low-wear bond primarily through the mechanical interlocking of small debris and high-quality films might contribute to low material wear.

4. Conclusions

This work aimed to investigate the friction and wear properties of polypropylene reinforced with recycled carbon fibre composites when subjected to a dry sliding action. This tribological investigation was expanded to include fibre loadings of 0, 0.5, 1.0, 3.0, 7.0, 10.0, 13.0, 15.0, and 20.0 wt%. Friction resistance increases as filler loadings increase from 0.5 to only 3 wt% and then gradually decreases. The highest coefficient of friction (COF) of 0.38 was observed in the range of 0.5 to 3.0 wt% fibre loading. On the other hand, all composites had a lower wear rate than pure PP. A transition between 3 wt% and 5 wt% loadings could be considered the tipping point at which the composites began to exhibit self-lubricating behaviour. The properties were attributed to the material's ability to deposit a thin and protective layer of polymer onto the counterface as a transfer film and patch film on the surfaces of the composites reattached during the sliding. The composites exhibited significantly stable transfer films, which aided in reducing friction and increasing the materials' wear resistance during dry sliding.

Additionally, the patch film on the composites acted as a separator at the interface, lowering the coefficient of friction. Carbon fibres were added to all samples to increase the wear resistance of the PP matrix. Due to the high quality of patch film and transfer film, direct contact between composites and counterfaces is effectively avoided; thus, wear is significantly reduced. SEM was used to observe the image of patch film and transfer film. The composites were found to wear due to the following mechanisms: 1) wear of the polymers, 2) abrasive wear of the fibres, 3) spalling of fibres, and 4) debonding of fibre and matrix. The patch film, transfer film, and shortening of the fibres due to the spalling mechanism contribute to the composites' increased self-lubricating behaviour. Currently, efforts are being made to produce sustainable materials made from various polymer composites to reduce mechanical component failure caused by tribological loading conditions.

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