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Mechanical and tribological properties of polypropylene filled with almond shells particles: Effect of particles size and proportion

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ABSTRACT

This work investigates the mechanical and tribological behavior of polypropylene (PP) filled with almond shell particles (ASPs) sustainable materials for green tribology, named PP-ASPs. The ASPs were obtained from the shells of almonds harvested in north of Algeria. The shells were dried then sieved and separated into two particle size ranges: D1 (600 µm ≤ D1 ≤ 800 µm) and D2 (400 µm ≤ D2 ≤ 600 µm). The PP-ASPs specimens were prepared with 5%, 10%, and 15% weight proportions (wt %) of ASPs for both size ranges. Shore D hardness tests, according to ASTM D 2240 standard, and tensile tests, according to ISO 37 Type II, were carried out on obtained PP-ASPs specimens. The tribological tests were conducted on a pin-on-disc RTEC MFT-5000 tribometer under fixed normal load, 10 N, and rotary speed, 180 rpm. The findings showed that the Shore D hardness and the Young modulus increase with ASPs proportion, particularly for D2 size. The tribological results reveal that the filling of PP with ASPs can lead to a beneficial effect on the friction coefficient, and wear mechanisms and can maintain the wear properties of the PP-ASPs composite relative to the neat PP.

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1. INTRODUCTION

Polymer based biocomposites have become an emergent field with a great variety of applications including many automotive parts (Roy et al., 2020), construction applications (Ahmad et al., 2022), and also packaging (Sharma et al., 2020). These materials are made by mixing polymers with natural particles producing ecofriendly composites. Polypropylene (PP) is one of the most polymeric materials widely used in fabricating of various industrial products due to its attractive characteristics including low density (Watt et al., 2020), acceptable rigidity (Jia et al., 2018), and chemical inertness (Feng et al., 2021). However, it is characterized by low hardness and poor mechanical properties and friction resistance (Grenadyorov et al., 2020) added to its biodegrade ability resistance (Alsabri et al., 2022). Consequently, these characteristics need improvement permitting to this polymer a good service performance and further usefully, gainfully, and sustainable ecofriendly material. One of the most studied technical solutions, for this issue, were its reinforcement with fibers, fillers of bio-sources. Based on these solutions, the effect of biofillers on the mechanical and tribological properties of PP constitutes a potential key to be investigated in the aim of involvement of its mechanical resistance and green tribological applications (Talib et al., 2018; Oladele et al., 2022; Mohamad Radzi et al., 2020; Zhang, 2013) as well as its implication for other industrial production.

In the literature survey, several studies have investigated the mechanical properties of polypropylene filled with particles of various bio-sources, such as almond shell particles (ASPs) (Essabir et al., 2013; McCaffrey et al., 2018; Lewis et al., 2023; Chiou et al., 2015; El Mechtali et al., 2015). These studies have shown that the incorporation of these particles into polypropylene can enhance its mechanical properties, especially, its flexural and Young modulus (Essabir et al., 2013; El Mechtali et al., 2015). However, they reduce the ultimate strain, tensile strength, and toughness (McCaffrey et al., 2018; Lewis et al., 2023). Elsewhere, the size and proportion effect of ASPs studies (Essabir et al., 2013; El Mechtali et al., 2015) have shown that the low size of fillers was more beneficial for the mechanical properties improvement of PP rather than the high size (McCaffrey et al., 2018). Besides the mechanical properties, several works have focused on the tribological behavior investigation of biofilled polymers. Barari et al (Barari et al., 2016) found that the Coefficient of Friction (*COF*) decreases with increasing volume fraction of nanocellulose fillers in the epoxy and only 1.4 wt% of nanocellulose enhances the tribological performance due to tribofilm effect of nanocellulose particles. Sharma et al (Sharma et al., 2020) exploited the Citrus limetta peels as a biofillers for developing an epoxy composite specimen. Three different size ranges, between 100 μm and 800 μm have been incorporated. They found that the *COF* decreases with decreasing the fillers size. Relatively to the neat epoxy, the lower, the fillers size, the lower, the wear loss was observed and the wear tracks were smoother for the low fillers size than to the high fillers size. The filling of PP with wood flours decreased the *COF* with a drop of 15% (Jan et al., 2023) at 40 wt% of these fillers occasioned by a wear resistance improvement. Furthermore, tribological properties such as low friction coefficients and low abrasion rates were obtained by Ibrahim et al (Ibrahim et al., 2019) with these same flours. Other studies involved that the addition of coir fiber (Liu et al., 2019) and cherry seed powder (Sydow et al., 2023) into PP have also led to a remarkable drop of the *COF* with 9% and a 40% enhancement of wear resistance (Liu et al., 2019). The coir fiber increases the composite crystallinity by 5 wt%, which plays a major role in wear reduction (Liu et al., 2019). The high content of cherry seed powder in PP shows also a positive effect on frictional behavior compared to the neat PP, especially with medium filler sizes ranging from 400 μm to 630 μm (Sydow et al., 2023). However, regarding the above-mentioned works, the tribological behavior of these polymer-based biocomposites was associated not only to the filler type but also to its proportion and size as well as. In addition, the particle size effect on the *COF* is not yet clear and it is linked to the used biofiller (Sydow et al., 2023). The abrasive and adhesive wear modes are the main damage mechanisms causing the deterioration of polymer based biocomposites under steel sliders. For low cherry seed filler contents, abrasion mode is the dominant wear mechanism, while for all cherry seed filler sizes of high content, the wear damage is the combination of abrasive and adhesive modes simultaneously (Sydow et al., 2023). In the case of wood powder content up to 20% in PP, the abrasion mode dominates the wear mechanism (Jan et al., 2023) and during the frictional tests, the wood flours existence in PP can lead to the formation of protective tribofilm (Ibrahim et al., 2019; Liu et al., 2019) resulting the improvement of the tribological behavior of the filled PP. On the other hand, there are few, if not non-existent, studies in the existing literature that investigate and exploit the almond shell fillers effect on tribological properties.

Regarding the above literature reviews and to lighten certain ambiguities of the effect of biofillers on mechanical and tribological properties of PP, in the present experimental work, Polypropylene-Almond shell particles (PP-ASPs) biocomposites were elaborated with two ASPs size ranges, D1 and D2, weight proportions of 5,10, and 15 wt%. Shore D hardness and mechanical properties via tensile tests were determined. Additionally using a pin-on-disk tribometer, tribological studies treating the effect of ASPs size and proportion on the PP composites were carried out. The Shore D hardness, tensile properties, friction coefficient, wear rate, penetration depth, and friction damage results were analyzed and discussed regarding the filler proportion and its size.

2.0 EXPERIMENTAL PROCEDURE

2.1 PP- ASPs Biocomposite Preparation

HE125MO grade is the polypropylene homopolymer (PP) from Borealis Polymers (Germany), characterized by a density of 0.905 g/cm^3, melt flow rate is 12 g/10 min at 230 °C/2.16 kg, which is good flow also has high stiffness. This makes it easy to process, particularly for pressing and high-speed injection. This PP was utilized as a matrix polymer for the PP-ASPs biocomposites specimen's preparation (Figure 1-a and b). The almond shells were extracted from almond fruits harvested in Ain Defla zone, Algeria during the summer season, of 2023. In its initial state, the shell inner is distinguished by its solidity and integrity of shape from cracks and openings during harvesting.

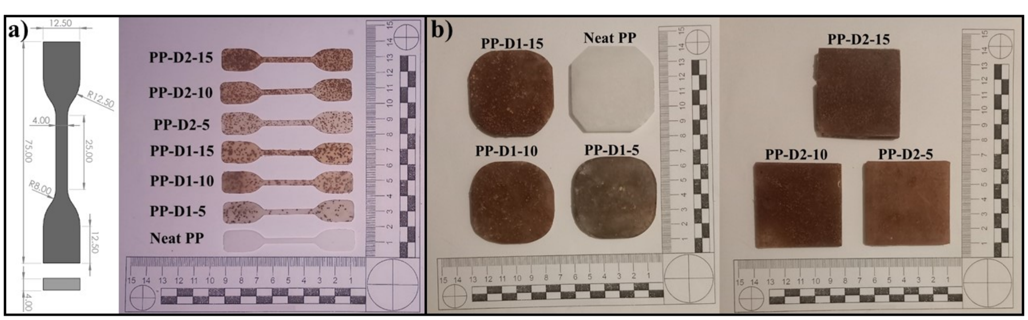

Figure 1: (a) Tensile tests specimen and (b) Shore D hardness and Pin-on-disk specimens for tribological testing.

The almond shells drying process was carried out according to two steps. The first step was naturally during the summer season for 15 days with average temperature ranging from 42 to 46°C and poor humidity. In the second step, the residual moisture is removed by drying in a Memmert UN75pa air circulating oven at 80°C for 4 hours before grinding for 5 minutes with 7000 rpm of rotation speed. After drying, the obtained APS density was 0.35±0.05 g/cm^3. The resulting particles passed with a sieve grade allowing the separation of particle to two size ranges: D1 and D2 of 600 µm ≤ D1 ≤ 800 µm and 400 µm ≤ D2 ≤ 600 µm. PP granules were introduced and melted in Brbender plastograph, Plastograph EC & Mixer 50EHT – Brabender chamber with a volume of 50 cm^3 with a temperature of 180°C and a mixing rotation speed of 40 rpm. Thereafter, the ASPs are slowly added to the melted PP with weight proportions of 5%, 10%, and 15% wt% for each size range. The mixed components remained in the chamber for 10 minutes before they were removed and left to cool at room temperature of ~24 °C. Specimens of sizes 50×50×5 mm and 100×100×1 mm are molded under 180°C and 2 metric tons of pressure for 10 minutes in Platen Carver manual laboratory presses hydraulic unit (Model 3912, 12 metric tons). Finally, the obtained specimens were cooled under pressure for 10 minutes at the same room temperature (Table 1).

Table 1: PP-ASPs microstructure characteristics.

Specimen	Particle size µm	Particle wt%
Neat PP	-	0
PP-D1-5	600 ≤ D1≤ 800	5
PP-D1-10	600 ≤ D1≤ 800	10
PP-D1-15	600 ≤ D1≤ 800	15
PP-D2-5	400 ≤ D2≤ 600	5
PP-D2-10	400 ≤ D2≤ 600	10
PP-D2-15	400 ≤ D2≤ 600	15

2.2 Mechanical Tests

The Shore D hardness tests were conducted according to ASTM D 2240 standard on a Bareiss durometer (Germany) balance, on specimens as indicated in Figure 1-b, at room temperature of 23±2°C, five tests were realized for each specimen. The tensile tests were performed according to ISO 37 Type II specimens (Figure 1-b), on a universal testing machine Lloyd LS 1K Plus Tensile instrument (Lloyd Instruments, UK) with a displacement speed of 5 mm/min (strain rate of 0.0025 s^{-1}) using a 1 kN load cell. Each test was repeated three times and the average values of mechanical properties and their standard deviations were reported.

2.3 Tribological Tests

The tribological experiments, on specimens of 50×50×5 mm in size (Figure 1-b), were performed via RTEC MFT-5000 (San Jose, California, U.S.A.) pin-on-disc rotary tribometer under dry conditions and room temperature of ~23±2°C. The used slider, of high wear resistance steel, has a spherical shape of 6 mm diameter. This slider material was chosen to ensure that the wear occurs only on the specimens. The tests were under fixed normal load, Fn, of 10 N, constant rotational speed of 180 rpm following track radius, R, of 15 mm making a tangential speed of 0.28 m/s, for 90 minutes. The realized total wear distance, L, is thus ~1.5 km which ensures that the friction encompasses the running in and the steady state friction regimes. Both COF in the function of time and its average value accompanied by its standard deviation during the steady state were determined. In addition, the wear depths during the test were reported in function sliding distance.

The wear coefficient, W_c, (Equation 1) was determined using 3D topographic measurements via optical profilometer Leica DCM8 (Leica, Germany) on 3.51×2.64 mm^2 of area. The transversal wear tracks, S, were used in the loss volume, V_m, calculation (Equation 2). The two equations are as:

$$W_c = \frac{V_m}{F_n.L} \quad (1)$$

$$V_m = 2\pi RS \quad (2)$$

3.0 RESULTS AND DISCUSSION

3.1 Mechanical Properties

Figure 2 summarizes the Shore D hardness results of the studied specimens. The neat PP exhibits the lower hardness than the other specimens. This hardness increases with increasing the ASPs proportion for both D1 and D2. Comparing the two sizes, the D2 ASPs are harder than the D1 ASPs specimens. These results clearly demonstrate the significant effect of particle size on the biocomposite hardness, which is not observed in the case of cherry seed powder (Sydow et al., 2023). The enhancing of Shore D hardness is attributed better matrix-particles interface with small ASPs, which reduces the formation of voids and weak points within the composite material. The small particles enhance stress transfer between the ASPs and the polypropylene matrix, leading to improved mechanical properties.

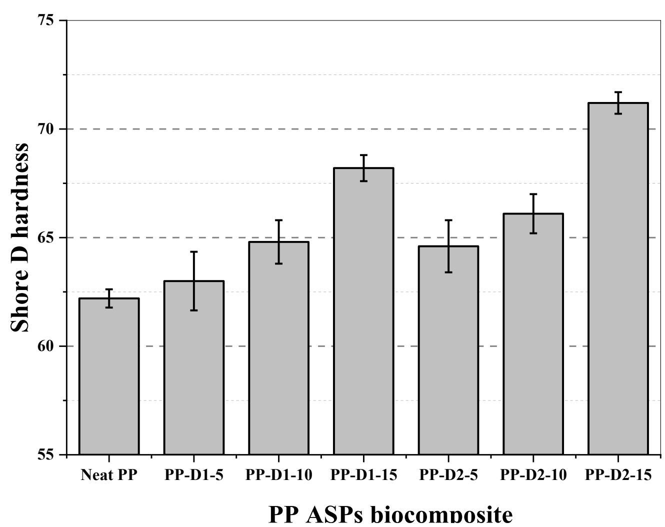

Figure 2: PP-ASPs specimens Shore D hardness.

Figure 3 presents the PP-ASPs properties obtained via tensile test. The Young modulus of neat PP was 1530 MPa lower than for all other specimens. Furthermore, this modulus increases with increasing ASPs proportion for both D1 and D2 sizes. Regardless of its sizes, the Young modulus was almost the same for 5 and 10 %, except for 15 % the Young moduli were 1766 MPa and 2013 MPa for D1 and D2 respectively slightly greater than of neat PP. Globally, the specimens with D2 size are characterized by Young moduli slightly higher than specimens with D1 size. There is a rise of Young modulus by 15 % s and by 32 % for D1 and D2 size respectively at 15 % of ASPs.

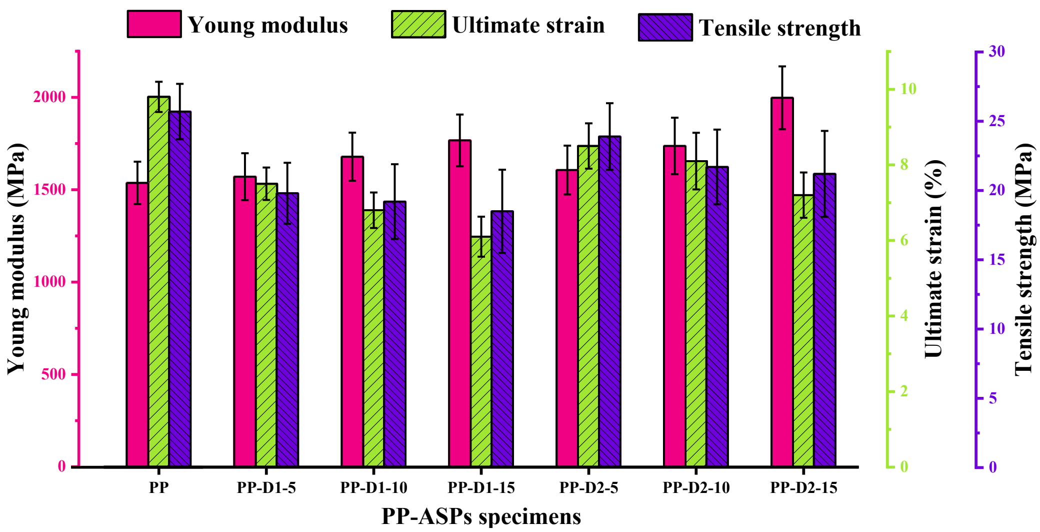

Figure 3: PP-ASPs specimen's tensile properties.

In addition, the neat PP exhibits the highest ultimate strain relative to the other specimens. The ultimate strain decreases with increasing the ASPs proportion for both used ASPs sizes. Comparatively, the D1 size exhibits lower ultimate strain values than the D2 size. Thus, the increasing ASPs content increases the biocomposite rigidity. However, the neat PP exhibits the highest tensile strength, while adding ASPs to the PP matrix contributes to the reduction of tensile strength. Furthermore, for both D1 and D2, the tensile strength decreased with increasing ASPs proportion, while the lowest values of tensile strength were obtained with ASPs size D1 of ~19MPa for 15 % of content.

There is an improvement of the tensile modulus by the addition of ASPs to the neat PP matrix. The tensile modulus increased by 15% with the addition of 15% ASPs D1 and by 32% with the same proportion of ASPs D2. This latter notable enhancement is more pronounced for the smaller ASPs size D2 compared to the larger ASPs size D1. This indicates that the smaller ASPs are more efficient in the improvement of the tensile modulus of the PP-ASPs composite. However, the addition of ASPs to the PP leads to a reduction of both tensile strain and strength. The tensile modulus decreased by 38% with the addition of ASPs D1 and by 15% with ASPs D2. Similarly, tensile strength decreased by 28.8% with the addition of 15% high-size ASPs (D1) and by 20% with the same proportion of low-size ASPs (D2). This behavior is attributed to the adhesion between the ASPs and the PP matrix, where the ASPs-PP interface plays a crucial role in determining the tensile properties of the PP-ASPs biocomposite.

The mechanical properties of polymer composites strongly depend on the filler's size, degree of dispersion, interfacial adhesion, and filler proportions. The increase in Young's modulus with increasing ASPs is attributed to the higher stiffness of ASPs compared to PP, as previously reported (Essabir et al., 2013; El Mechtali et al., 2015). However, the ultimate strain and tensile strength decrease with increasing ASPs proportion due to stress concentration and decohesion between the PP matrix and the fillers, leading to accelerated specimen breaking and reduced ultimate strain. PP-ASPs with D2 ASPs exhibits higher values of ultimate strain and tensile strength compared with D1 ASPs due to less pronounced decohesion propagation.

3.1 Tribological Behavior

Figure 4-a presents the friction coefficient (*COF*) evolution as a function of sliding distance. The average friction coefficient (COF_m) and its standard deviation are presented in Figure 4-b relative to the steady stage period. All tested specimens go through the running in process to the steady state regimes before about 200 meters of sliding (Figure 4-a) which corresponds to about 10 minutes of sliding. During the steady state period and relative to the *COF* evolution of neat PP, the obtained results permit to distinction two *COF* groups. The group of PP-ASPs filled with D2 size exhibits almost the highest *COF*, and the other one with D1 size exhibits the lowest *COF*. This ascertainment shows the effect of particle size on the evolution of friction coefficient.

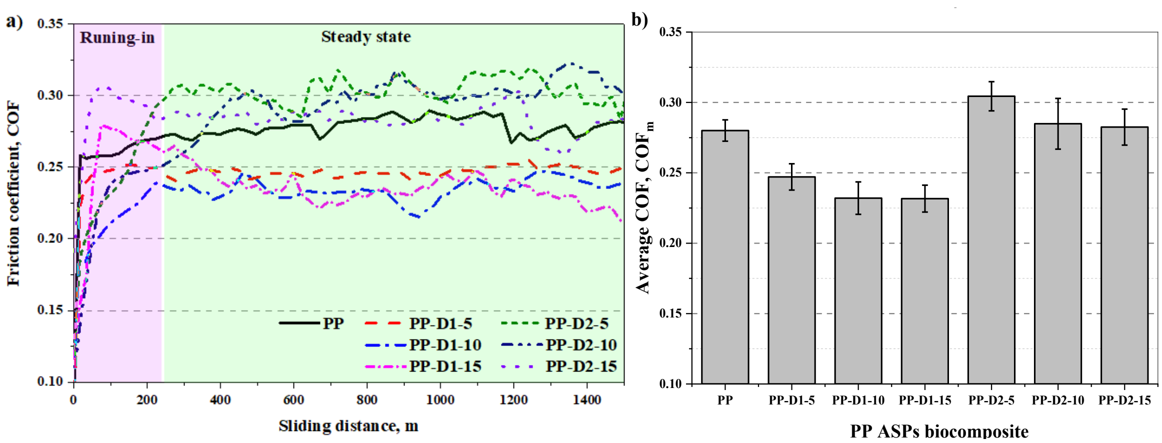

Figure 4: a) Friction coefficient, *COF* vs. test duration and b) steady state average COF_m.

During the steady state, the COF values for all tested specimens ranged between 0.2 and 0.3 (Figure 4-b). The COF values obtained with D1 size particles were close, ranging between 0.23 and 0.25, which are lower than those obtained with neat PP and D2 size particles. Furthermore, the COF decreases with increasing ASPs proportion for both D1 and D2 sizes. Similar findings were previously reported for wood filler and cherry seed filler in the PP matrix (Jan et al., 2023; Ibrahim et al., 2019; Sydow et al., 2023). Notably, the lowest COF was observed with D1 size particles at 10% and 15% concentrations, resulting in a 20% reduction compared to neat PP. This COF reduction achieved with 10% ASPs, was more significant than the reductions obtained with 40% and 55% wood powder, which resulted in COF reductions of 15% and 20%, respectively (Jan et al., 2023; Ibrahim et al., 2019). The reduction of COF observed in this study can be attributed to the presence of high-stiffness ASPs particles. However, the COF is not directly related to the particle size but it is related the used biofiller (Sydow et al., 2023). It was previously observed that large cherry seed fillers were more effective in the COF reducing compared to the small fillers (Sydow et al., 2023). In contrast, for Citrus limetta peel fillers in epoxy, the smaller fillers, the lower COF.

The tribological properties of thermoplastic composites have a considerable link with their mechanical properties (Kneissl et al., 2023), especially their viscoelastic behavior. The wear coefficient, W_c, results are presented in Figure 5. The neat PP and PP-D2-15 exhibit the lowest W_c of ~2.10-4 mm³/Nm, while for both 5 and 10 % proportions, the obtained Wc values are almost closer to ~3.5.10-4 mm³/Nm for both D1 and D2 sizes. The wear of PP-D1-15 was occasioned by the highest W_c value. This result implies that for both 5 and 10 % low proportions, the particle size has a minor effect on the wear coefficient, while for high particle proportion, the size is a decisive factor in determining the wear coefficient. The high W_c values for D1 can be attributed to its high size causing the rupture of a brittleness ASPs and made more material removal than D2 size specimens. The observed ASPs filler size effect on the W_c was similar to the Citrus limetta peels fillers size effect, where the high fillers size produces voids leading to a poor composite miscibility (Sharma et al., 2020). It should be noted that these results were obtained at the end of the friction test.

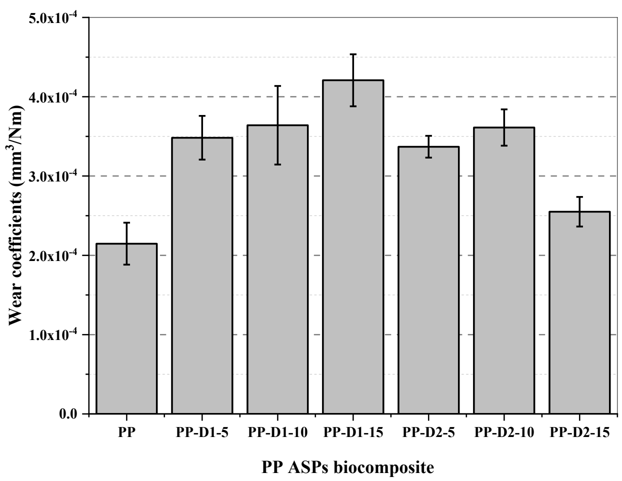

Figure 5: Wear coefficient, $W_c$vs the size and proportion of particle at the end of wear tests.

The wear coefficient (W_c) of PP-ASPs specimens at the end of testing does not provide a comprehensive overview of the wear behavior. To achieve a thorough analysis of W_c, the wear depth (*WD*) was evaluated throughout the test (Figure 6). The wear depth, as a function of sliding distance, evolved through three distinct stages. The first stage represents the running-in process, where the depth slightly increases due to the removal of surface asperities during the initial 200 meters of sliding distance. Following this, the *WD* exhibits a pseudo-stability during the second stage, corresponding to regular wear, where the material demonstrates relative wear resistance. In the third stage, the *WD* continues to evolve similarly to the second stage but then rapidly drops. There is a similarity in the *WD* evolution of neat PP and PP-D2-15 during the entire sliding distance, showing shallower depths compared to the other specimens. However, PP-D1-15 exhibits the deepest track wear over the overall distance traveled. At the end of the test, neat PP and PP-D2-15 biocomposites display the lowest *WD*, while PP-D1-15 shows the highest *WD*. These results are in good concordance with the findings of the wear coefficient (*Wc*) (Figure 5).

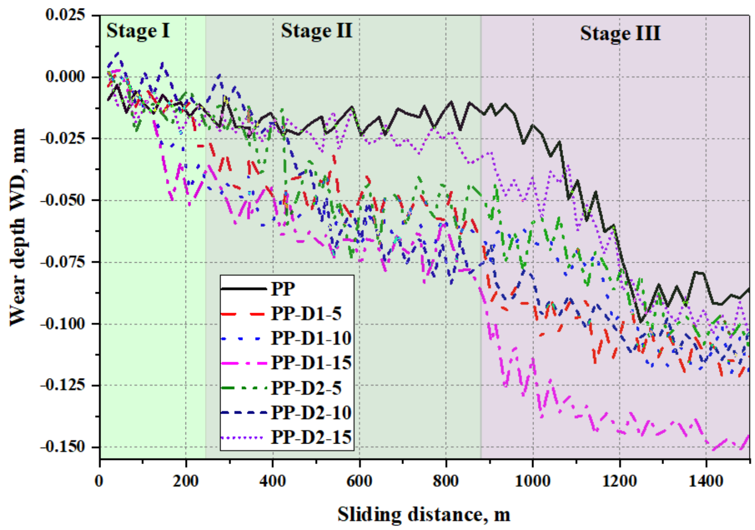

Figure 6: Wear depth vs. sliding distance.

To analyze the wear mechanisms of the studied biocomposites, the Optical 3D micrographs of wear tracks at the end of wear tests, were exposed in Figure 7. The neat PP exhibits a wear track profile of the smallest width and containing voids (Figure 7-a). The addition of ASPs has contributed to the formation of pile up on the track sides more pronounced for D2 size than for D1 size. For the filled PP, the wear tracks in the case of D1 are occasioned by rough and deep surfaces more noticeable (Figure 7-b, c and d) than in the case of D2 size of smooth grooves (Figure 7-e, f and g), for all ASPs proportion. Sharma et al (Sharma et al., 2020) found the same above phenomenon where the lower, the fillers size, the smother, the wear track surfaces. The wear of both sizes occurs with the formation of microscratches, these microscratches resulted from the crushing and displacement by the slider of ASPs during the continued sliding, which indicates the presence of the wear with the formation of third body, which leads to the formation of tribofilm between contact surfaces.

The observed wear mechanisms are primarily influenced by sliding history, environmental conditions, changes in surface topography, and subsurface material damage. These factors exacerbate the wear damage, leading to delamination phenomena and the formation of wear debris. Notably, neat PP filled with D2 size particles exhibits better wear resistance compared to PP-ASPs filled with D1 size particles. As shown in Figure 7, the wear mechanism of neat PP is occasioned by the formation of voids caused by removed wear debris which adhere on the slider surface and tear off from the track surface (Figure 7-a). These observed voids in neat PP can be attributed to the stick-slip phenomenon (Jiang et al., 2015). In contrast, these voids were absent in the case of filled PP. When comparing the damage forms observed for both particle sizes, it can be deduced that the formation of tribofilm is more noticeable for D2 than for D1, which affects the wear coefficient (Figs. 5 and 6). This wear behavior is similar to the cases of incorporating wood flour (Ibrahim et al., 2019) and coir fiber in PP (Liu et al., 2019). The presence of ASPs on the wear surfaces increased the abrasion by third-body debris formation (Sharma et al., 2020; Jan et al., 2023; Ibrahim et al., 2019; Sydow et al., 2023). The filling of PP with ASPs and cherry seed powder results in a similar effect on the wear mechanism (Sydow et al., 2023).

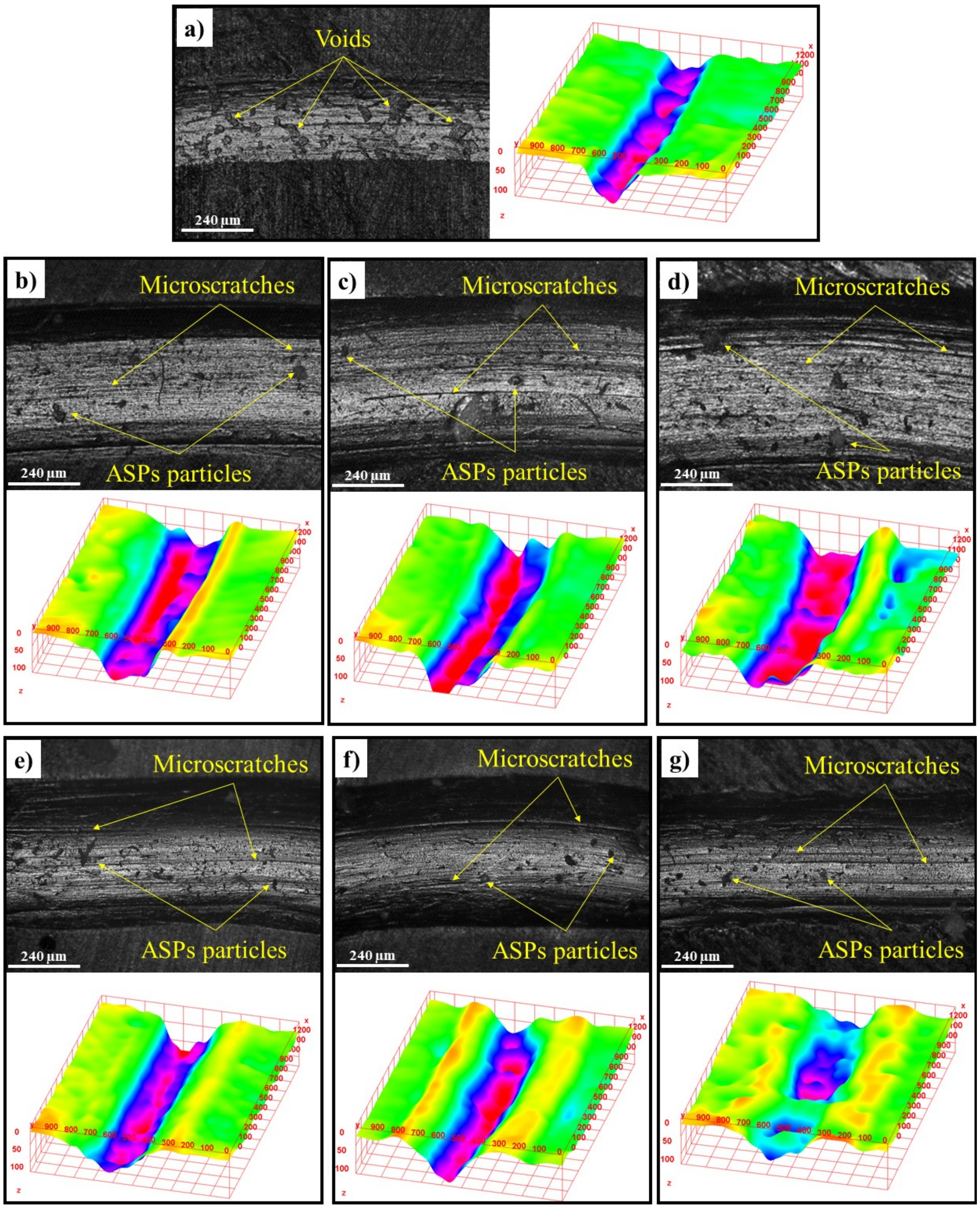

Figure 7: PP-ASPs wear tracks micrographs and Optical 3D, (a) PP, (b) PP-D1-5, (c) PP-D1-10, (d) PP-D1-15, (e) PP-D2-5, (f) PP-D2-10 and (g) PP-D2-15.

The enhanced wear resistance observed in neat PP filled with D2 size particles can be attributed to the better distribution and adhesion of these small particles within the PP matrix. This improved particle distribution reduces the formation of voids, leading to a more homogeneous wear pattern. Additionally, the formation of a tribofilm is more important with D2 particles providing a protective layer and enhancing wear resistance.

In summary, the wear mechanisms in PP composites are significantly influenced by the size and distribution of filler particles. Small particles, such as D2, provide better wear resistance due to their ability to form a protective tribofilm. The presence of ASPs as for cherry seed powder in the PP matrix leads to increased abrasion, but the overall wear resistance is improved with small particle sizes.

Based on the presented findings, our study contributes to the comprehension of the effect of ASPs on the mechanical and tribological properties of filled PP. Both properties are connected and evaluated differently with the filling parameters such as the particle size and their proportions. The filling of PP with ASPs gives better hardness and Young modulus and reduces the *COF*. Nevertheless, the wear can be reduced with an adequate choice of size and proportion of the ASPs. The adequate choice of ASPs parameters can enhance the mechanical and tribological behavior of the obtained composite compared to the neat PP. This research show the requirements and objectives of sustainable materials for industrial applications have proved that the filling of PP with ASPs, of low cost, can be a suitable and sustainable choice for green tribology reducing the environmental impact of polymers.

CONCLUSIONS

Based on these obtained experimental results, the conducted analyses, and the observations presented in this study, the following conclusions can be extricated as:

The hardness of PP-ASPs composites increases as the proportion of ASPs increases. The smallest ASPs fillers are more effective in the hardness enhancement of PP. The addition of ASPs to PP also increases the Young's modulus. A high proportion of ASPs (15%) and a low particle size led to higher Young's modulus than the other cases. A significant increase of 32% in Young's modulus was obtained with D2 size and 15% ASPs.

Tribological findings indicate that the addition of high ASPs size, D1, with high proportion (15%) is more effective in reducing the coefficient of friction (COF). The wear coefficient increases with the addition of ASPs regardless of their size, except for PP filled with D2 size at a proportion of 15% showing a minor effect on the wear coefficient relative to neat PP. The adhesive wear mode dominates the wear mechanism for neat PP and low ASPs proportions. However, this wear mode transits to the abrasion mode with high ASPs proportions.

The presence of ASPs on the wear tracks causes wear by third-body abrasion mode. PP filled with small ASPs size, D2, exhibits better wear resistance compared to high ASPs size, D1.

The requirements and objectives of sustainable materials for industrial applications have provided that the filling of PP with ASPs can be suitable and sustainable choice for green tribology benefits reducing the environmental impact of polymers.

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