



Effect of Treatment of Natural Fillers on the Tribological Behaviour of Polymeric Materials

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ABSTRACT

The study clarifies the mechanism of debris formation and adhesion on metal counterface. Polymeric materials filled with 30% sodium hydroxide 3, 4 and 5% treated and untreated coconut shell and bagasse fillers 100, 180 and 112 μm particle sizes, respectively, were produced. In the spectroscopic analysis, the increase in the transmittance level of a functional group indicates its reduction. 5% NaOH treated coconut shell filler and 4% NaOH treated bagasse filler had the highest reduced OH functional group in the treated fillers. Their carboxylic acid transmittances were 98.218% and 99.900%, respectively. The carboxylic acid transmittances of the untreated coconut shell and bagasse fillers were 95.218% and 95.954%, respectively. The unfilled polymers had a friction coefficient of 0.21, and their wear rates were in the order of $10^{-3}\text{mm}^3\text{N}^{-1}\text{mm}^{-1}$. The filled polymers had a friction coefficient lower than 0.21 with varied wear rates. Polypropylene with sieved 100, 180 and 112 μm coconut shell and bagasse particles had wear rates in this order 0.010, 0.008 and 0.011 $\text{mm}^3\text{N}^{-1}\text{mm}^{-1}$, and 0.009, 0.11 and 0.007 $\text{mm}^3\text{N}^{-1}\text{mm}^{-1}$, respectively. The wear rate of the polyethylene with sieved 100, 180 and 112 μm coconut shell and bagasse particles were in this order 0.012, 0.006 and 0.010 $\text{mm}^3\text{N}^{-1}\text{mm}^{-1}$, and 0.008, 0.009 and 0.008 $\text{mm}^3\text{N}^{-1}\text{mm}^{-1}$, respectively, in the same order with the particle sizes. The SEM micrograph of the wear track showed low adhesive and detached patchy shapes debris of the polymeric materials on the disc counterface. The larger filler particles caused agglomeration and low dispersion, especially in bagasse-filled polymeric materials. The suggestion, therefore, was the examination of the polymeric material on different metal counterfaces.

KEYWORDS: Alkalization, Coconut shell, Bagasse, Friction, Polymeric, Tribochemistry, Wear.

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1.0 INTRODUCTION

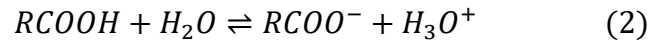
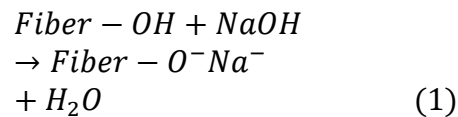
Thermoplastic-based, treated, natural filler composites have good mechanical properties and have gained prominence as a replacement for steel (Gunge *et al.*, 2019; Yaghoobi & Fereidoon, 2018; Friedrich, 2018). The treatment of natural fillers is one of the breakthroughs to increase the compatibility of the filler with the polymer matrix (Ye *et al.*, 2014; Sathishkumar *et al.*, 2017; Sullins *et al.*, 2017). Alkalization provides polymeric material with improved mechanical properties and low moisture affinity. Sodium hydroxide (NaOH) is the best candidate for natural filler treatment (Singh *et al.*, 2017; Hallad *et al.*, 2018; Bakri *et al.*, 2017; Preet *et al.*, 2017). Considering the tribological properties of polymeric materials, the nature and quantity of fillers in the polymer matrix play some role in the quality of the transfer film (Ye *et al.*, 2014). The reason for the chemical treatment of fibre is to eliminate the high OH coating, increase the surface roughness leading to an increase in mechanical interlocking with polymer matrices (Nirmal *et al.*, 2015; Al-Oqla *et al.*, 2015). This can be achieved when the chemical treatment time and the concentration of sodium hydroxide are increased (Chandramohan & John, 2017). Spectroscopic analysis has shown evidence of several important functional and molecular groups in natural fillers and polymers, and the increase in the transmittance level of a functional group indicates its reduction (Koay *et al.*, 2013; Hong *et al.*, 2019; Veerasingham *et al.*, 2020). Onodera *et al.* (2014), and Onodera *et al.* (2017) showed that polymers can form carboxylic acid (C=O) end groups under stress in a humid air environment. Ramesh (2019) added that based on the material properties, the change in the

tribometer environment affect the friction and wear behaviour of tribo-pairs. Aghababaei *et al.* (2016) explained that on tribo-pairs, small asperities form smaller debris that can deform plastically and gradually smoothen out the roughness of tribo-pairs. Larger asperities form larger debris. Aghababaei *et al.* (2018) concluded that larger debris on tribo-pairs gradually deform in a closed system or are swept away in an open system. The present study clarifies experimentally the analytical works of Aghababaei *et al.* (2016), and Aghababaei *et al.* (2018). It focuses on the effect of the treatment of natural fillers (untreated and treated bagasse and coconut shell fillers) on the tribological behaviour of polymeric materials and their effect on debris retention on tribo-pairs.

2. MATERIALS AND METHODS

The materials used were bagasse (B), coconut shell (Cs), polyethylene (PE), polypropylene (PP), NaOH and distilled water. The apparatus used includes; a pulverizing machine, sieve with mesh sizes, 100, 180, and 112 μm , a two-roll milling mixer, model (HTR-300), a hydraulic press, Anton Paar Compact (600mm x 700mm, 550mm height) tribometer, Agilent Cary 630 Fourier transmission infrared spectroscopy (FTIR), and a scanning electronic spectroscopy (SEM). See Alibi *et al.* (2021) for a detailed description of the equipment. The bagasse and coconut shell fillers were treated separately with 3%, 4% and 5% NaOH solution in distilled water. The fillers were soaked in these solutions for 24hrs.

The aqueous NaOH degrades a certain amount of the natural fillers' constituents and exposed the reactive hydroxyl (OH) group on the fibre surface. The exposed hydroxyl (OH) group ionized to the alkoxides group and the number of reactive sites on the fibre surface increased (Sepe *et al.*, 2018; Sullins *et al.*, 2017). Equation (1) shows the reaction of the alkali treatment with the fibre. Also, the Carboxylic acid in the fillers ionizes slightly in water to form a moderately acidic solution Equation (2).



The particles were washed to remove traces of NaOH and others contaminants and dried in an ovum at a temperature of 80°C. This approach modifies fillers, increased their compatibility with the polymer by rendering their surface less hydrophilic. Both the treated and untreated filler particles were pulverized, scanned with FTIR, at a frequency range of 650–4000 cm^{-1} with a resolution of 8 cm^{-1} . The pulverized untreated and treated fillers were sieved into 100, 180 and 112 μm particle sizes. Further, 30%wt bagasse and coconut shell sieved 100, 180 and 112 μm treated particle sizes were formulated with 70%wt polymer making a composition of 100%wt, and 100%wt unfilled polymers as control (Table 1). The formulations were compounded in a two-roll milling mixer, compressed inside a mould in a hydraulic press, and were machined into polymeric pins.

The methods of Odi-Owei and Onuba (2017), and Odi-Owei and Alibi (2019), were used in the friction coefficient measurements. Equation (3) was used. Each polymeric sample was tested at 10N load 500 mm/sec speed, and a sliding distance of 150000 mm in the tribometer.

$$\mu = \frac{F}{L} \quad (3)$$

where μ is the friction coefficient, F is the frictional force, L is the applied load.

Table 1: Treated Filler Formulation

Particle sizes (μm)	PE	PP	B	Cs	Polymeric material (100%)
0	100		0	0	PP+B
0		100	0	0	PE+B
100		70	30		PP+B
180		70	30		PP+B
112		70	30		PP+B
100		70		30	PP+Cs

180	70	30	PP+Cs
112	70	30	PP+Cs
100	70	30	PE+B
180	70	30	PE+B
112	70	30	PE+B
100	70	30	PE+Cs
180	70	30	PE+Cs
112	70	30	PE+Cs

Wear is another phenomenon that occurs in tribo-pairs. One or both surfaces lose material. Equation (4) is the famous Archard equation.

$$V = k \frac{LS}{\sigma_0} \quad (4)$$

where V is the wear volume of the tribo-pairs, S is the sliding distance of the tribometer, σ_0 is hardness value of the materials, and, k is the wear rate of the tribo-pairs. The effect of other parameters is empirically merged into k , which does not stem from any theoretical development. This drawback limits the predictive capacity of the Archard model (Frérot *et al.*, 2016). Also, it is contradictory that the Archard equation that deals with adhesive wear have no adhesion parameter.

Therefore, Popov (2018) merged the Archard model and Rabinowicz criteria. The maximum stress in the model was considered in the order of the material hardness σ_0 . The stored elastic energy U_{el} was estimated as the product of energy density $\sigma_0^2/(2G)$ and stressed volume D^3 , Equation (5).

$$U_{el} \approx \frac{\sigma_0^2}{2G} D^3 \quad (5)$$

Equation (6) is the adhesive energy that is needed to detach two asperities to create a new free surface. The energy would relax if a worn particle (transfer film) is formed. The process of wear particle detachment occurs when the stored elastic energy of the contacting asperities exceeds the energy needed to create a new free surface. Thus:

$$U_{adh} \approx \Delta w \cdot D^3 \quad (6)$$

where Δw is the work of adhesion that is associated with the newly created free surface per

unit area of crack growth in each of the asperity tips. It follows that only particles larger than some critical length scale D_{crit} , Equation (7), can be detached (Aghababaei *et al.*, 2016).

$$D > D_{crit} = \frac{2G \cdot \Delta w}{\sigma_0^2} \quad (7)$$

Also, the presence of a lower bound of wear particle size is predicted from Equation (6), when both surfaces contributed equally to debris formation. To further clarify this, Popov (2018) showed that tribo-pairs of materials that are not the same, a hard and soft material, and the softer material will form more debris. Based on this idea, Popov (2019) developed a power-law wear model under the assumption that for a purely elastic material with tangential stress in contact characterised by friction coefficient, the condition of wear particle detachment can still be satisfied. In this case, the material hardness is not part of the functional parameter. Also, with the assumption that $V \propto S$, $P = F/L^2$ and assuming that the wear process occurs homogeneously in the whole contact area, Equation (8) was developed.

$$V \propto F^\alpha G^\beta L^\gamma W^\delta S \quad (8)$$

Dimensional analysis gives:

$$\begin{aligned} \beta &= \alpha + \gamma - 2 \\ \delta &= 2 - \gamma - 2\alpha \end{aligned}$$

And

$$V = k F^\alpha G^{\alpha+\gamma-2} L^\gamma W^{2-\gamma-2\alpha} S \quad (9)$$

3. RESULTS AND DISCUSSION

The spectroscopic analysis for the untreated and treated coconut shell filler is shown in Figure 1. The higher the value of the transmittance the lower the intensity of the functional groups in the fillers.

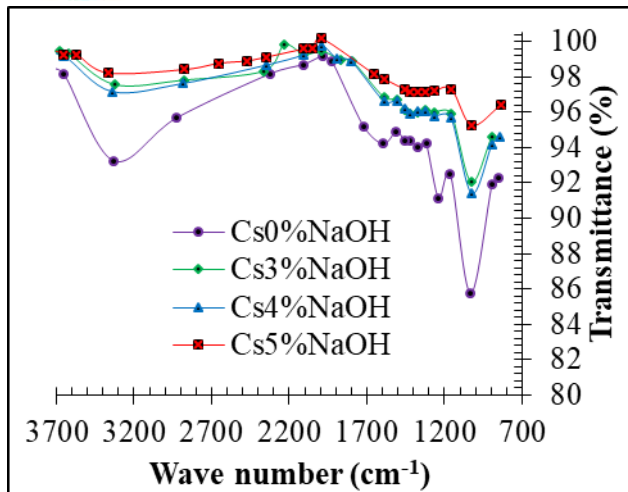


Figure 1: Spectra of Untreated and Treated Coconut Shell Filler

The broadband $3700\text{-}3000\text{cm}^{-1}$, at a transmittance of 93.256% was assigned to the hydroxyl (OH) group of the untreated filler (Cs0%NaOH). Compare to the untreated filler, the intensity of the OH spectra was reduced in the treated fillers Cs3%NaOH, Cs4%NaOH, and Cs5%NaOH, their transmittances were 97.561%, 97.17% and 98.218% respectively. Thus, it could be inferred that Cs5%NaOH has the highest reduced OH functional group in the treated fillers. Further, the spectrum of the carboxyl functional group of the fillers is found at 1722cm^{-1} , at this point, the transmittance of the untreated filler (Cs0%NaOH) was 95.218. For the treated filler Cs3%NaOH, Cs4%NaOH, and Cs5%NaOH the values of the transmittance are 97.154%, 96.637% and 97.304%, respectively, which indicates a reduction of carboxylic acid by the NaOH (Equation 10).



In Figure 1, the presence of the C-O functional group was found at the band $1596\text{-}1506\text{cm}^{-1}$ at the transmittance of 94.207% for the untreated fillers and 97.892% for the treated fillers. This further confirmed the presence of carboxylic acid in the natural fillers and its reduction. The NaOH also decomposed C-H at the 1375 band, C-O-C, at the 1162 band, and C-O-C at the 1241 band, in the treated filler. These are evident in Koay *et al.* (2013) and Hong *et al.* (2019).

Several research groups have shown that the formation of carboxylic acid end groups promote the adhesion of wear debris to a metal counterface and is effective in transfer film formation (Onodera *et al.*, 2014; Ye *et al.*, 2014; Onodera *et al.*, 2017). Therefore, the treatment of the coconut shell filler with NaOH should change the coconut shell filler from its highly hydrophilic nature to a less hydrophilic filler, and expose the carboxylic acid layer of the coconut shell filler. This would enable the filler in conjunction with polymers to produce a film that would adhere tenaciously to the metal counterface of tribo-pairs.

In Figure 2 spectra of the untreated (B0%NaOH) and treated, (B3%NaOH, B4%NaOH and B5%NaOH) bagasse filler is presented. The band at $3000\text{-}3500\text{cm}^{-1}$ at the transmittance of 93.113% was assigned to the OH group of the bagasse filler. The carboxylic acid content was very low in the untreated bagasse filler, it occurs at wavenumber 1722cm^{-1} at a transmittance of 95.954%, which was absent in the treated bagasse filler (see Figure 2). However, the best-performed filler that has the least OH band in the treatment was the filler with 4% NaOH treatment, which has an absorption spectrum of 3291.2cm^{-1} at the transmittance of 97.035% lower than that of the other specimens (B3%NaOH, and B5%NaOH).

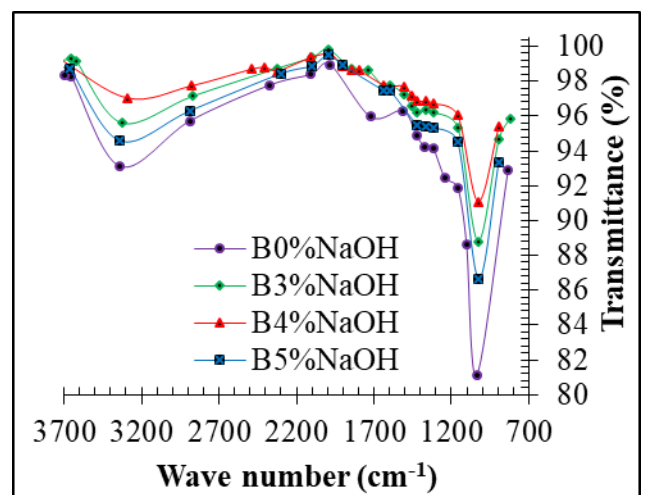


Figure 2: Spectra of Untreated and Treated Bagasse Filler

In Figure 3, the friction coefficient of polypropylene filled with varied particle sizes of bagasse, coconut shell, and the unfilled polymer are presented. The unfilled polymer has a friction coefficient of 0.21, as evidenced in the literature (Odi-Owei & Alibi, 2019).

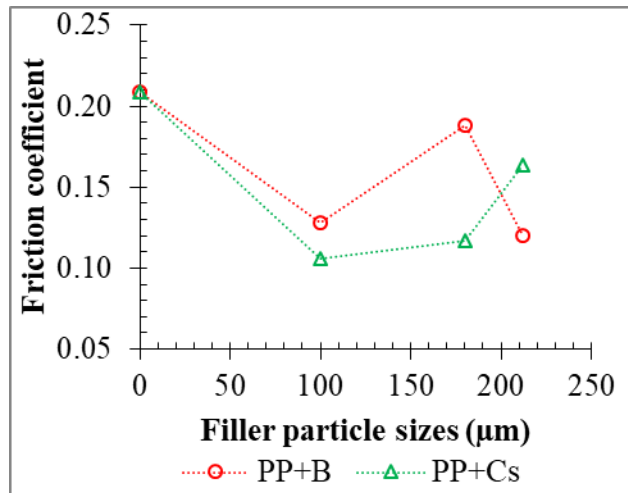


Figure 3: Polypropylene-based Bagasse and Coconut Shell Friction Coefficient

The polymer-based bagasse composites had a friction coefficient lower than the unfilled polymer. The highest friction coefficient was observed in the polymer-based 180µm bagasse-filled particle sizes. Besides, the polymer-based coconut shell-filled composite had a low friction coefficient than those of the unfilled and bagasse-filled polymeric materials. Their friction coefficient increased as the coconut particle sizes were increased. However, the inconsistencies in the friction coefficient of the bagasse-filled polymeric material as the particle sizes increased could probably have occurred due to the low dispersion of the fillers in the polymer matrix, which may have resulted in filler agglomeration in the polymer matrix.

In Figure 4, the wear rate of the polymer-based bagasse-filled composite decrease as the filler particle sizes were increased. The wear rates were in the order of $10^{-3} \text{mm}^3 \text{N}^{-1} \text{m}^{-1}$. The Polymer-based bagasse particle size 212µm has the least wear rate and the polymer-based coconut shell-

filled composite particle size 212µm has the highest wear rate. The deviations in the wear rate of the 100 and 180µm coconut shell and bagasse filled polymer-based composites were not much compared to the unfilled polypropylene. This is an indication that the effect of the filler particles was not felt in the tribo-pairs due to the decrease in the C=O content of the treated coconut shell and bagasse filler by the sodium hydroxide.

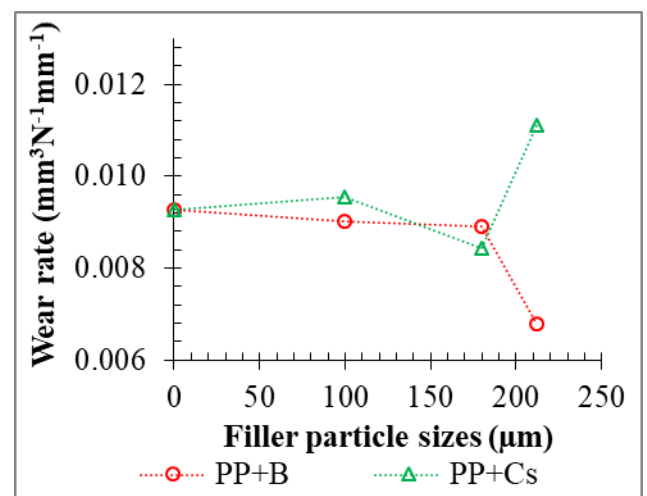


Figure 4: Polypropylene-based Bagasse and Coconut Shell Wear Rate

Also, due to the loss of wet adhesion of the debris to the metal counter-face, (see plate 1) it is reasonable to suggest that the debris were swept away as the disc continued to rotate. Thus, retention of the debris on the counter-face is very important for a wear rate reduction which is in agreement with Aghababaei *et al.* (2016), and Aghababaei *et al.* (2018).

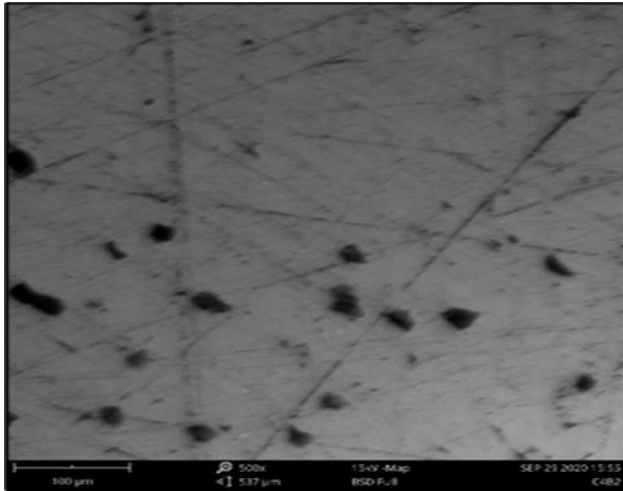


Plate 1 Polypropylene-base Bagasse and Coconut Shell Wear Debris

In Figure 5 the friction coefficient of the unfilled polyethylene was within the range of 0.2 as evidence in the literature for unfilled polyethylene (Odi-Owei & Alibi, 2019). The polymer-based bagasse particle sizes 100, and 180µm composite had friction coefficient lower than the unfilled polyethylene. The polymer-based bagasse particle size 212µm had a friction coefficient similar to the unfilled polymer.

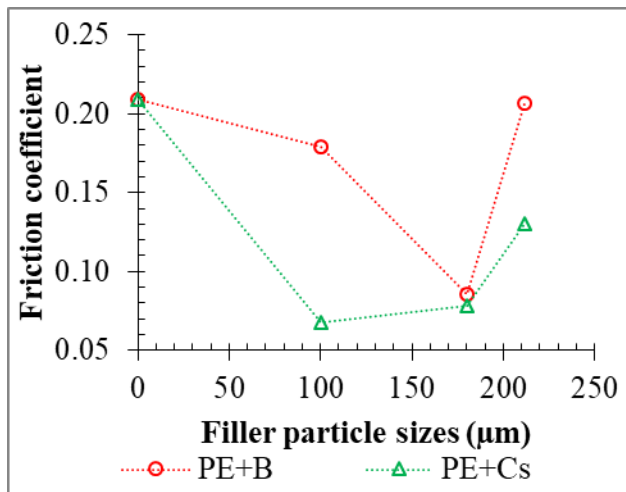


Figure 5: Polyethylene-based Bagasse and Coconut Shell Friction Coefficient

The friction coefficient of the polymer-based coconut shell-filled composite is lower than those of the unfilled polymer and polymer-based

bagasse-filled composite. The coconut shell filler particle sizes affected the friction coefficient of the composites. The friction coefficient increased as the particle sizes were increased although, lower than that of the unfilled polymer. This attribute of the fillers in the friction coefficient measurement suggests a lack of wet adhesion, which was highest in the polymer-based bagasse-filled polymeric materials.

In Figure 6, the polymer-based bagasse-filler composites had higher wear than the unfilled polymer, but lower than those of polymer-based coconut shells with 100 and 212 µm filler particle sizes. Only the 180 µm polymer-based coconut shell-filled composite had a slightly lower wear rate than those of the unfilled polymer and polymer-based bagasse-filled composites.

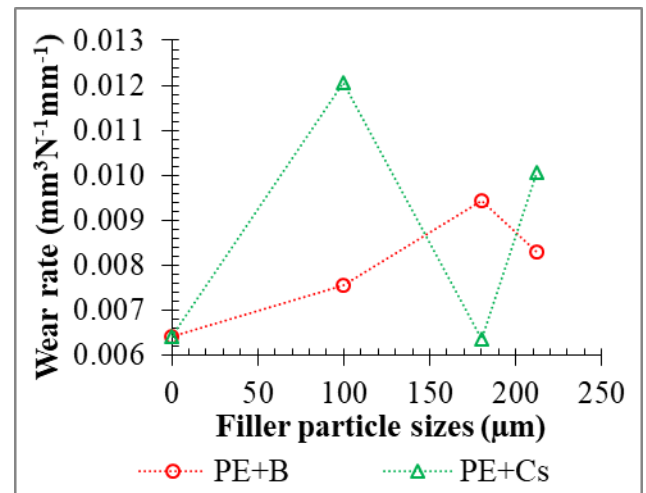


Figure 6: Polyethylene-based Bagasse and Coconut Shell Wear Rate

In Plate 2, the SEM micrograph of the polymeric materials wear debris showed the patchy nature of the debris. This is evident that the lack of wet adhesion on the counterface probably caused the removal of the wear debris from the steel counterface.

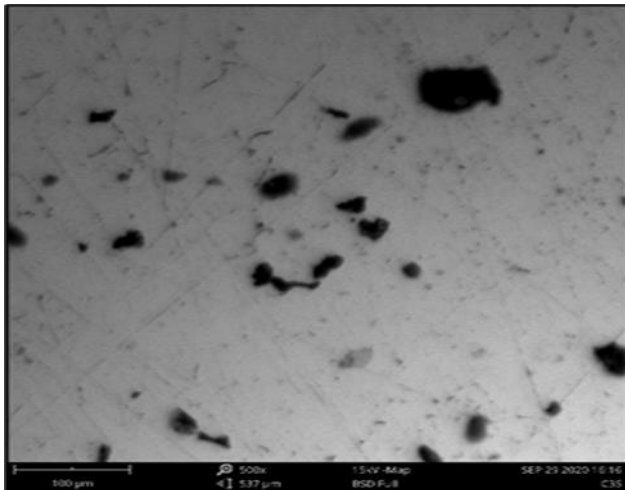


Plate 2: Polyethylene-based Bagasse and Coconut Shell Wear Debris

Thus, it could be inferred that the treatment of the fillers with sodium hydroxide greatly affected the friction and wear behaviour of the polymeric materials. The neutralization reaction between the sodium hydroxide and the carboxylic acid in the fillers caused the low adhesion of the wear debris on the metal counterface. This led to the detachment of the wear debris, increase in the wear rate and friction coefficient of the polymeric materials due to the starvation of transfer film on the disc surface.

4. CONCLUSION

The outcome of this research work showed that the retention of debris on the counterface of steel is a function of many contributing factors. It is therefore concluded that

- i. the NaOH used for the treatment of the natural filler neutralized the carboxylic acid of the filler, which led to a loss of wet adhesion and detachment of wear debris from the disc surface.
- ii. plastic deformation of the debris to smoothen out the roughness of tribo-pairs depend on wet adhesion, the presence of carboxyl acid, and the merging and retention of the deformed debris by the carboxylic acid on the counter-face to form transfer film.
- iii. transfer film forms during sliding contact
- iv. transfer film control both friction and wear of counter-face.

It is recommended, therefore, that Different metal counterfaces should be used to ascertain changes that may occur on their surfaces, acid treatment of the fillers, and quantification and confirmatory test of the carboxylic acid of the polymeric material using Gas chromatography-mass spectrometry (GC-MS).

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Nomenclature & Units

σ_0	Hardness
B	Bagasse
C=O	Carboxylic acid
Cs	Coconut shell
D	Stress volume (Nm^{-1})
D_{crit}	Critical length scale
F	Frictional force (N)
FTIR	Fourier transmission infrared
G	Energy density (J)
GC-MS	Gas chromatography-mass spectrometry
K	Wear rate ($\text{mm}^3\text{N}^{-1}\text{m}^{-1}$)
L	Load (N)
PE	polyethylene
PP	polypropylene
S	Sliding distance (m)
SEM	Scanning Electronic Spectroscopy
U_{adh}	Adhesive energy (J)
U_{el}	Store elastic energy (J)
V	Wear volume (mm^3)
W	Work of adhesion (J)
μ	Friction coefficient