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Experimental investigation on three-body abrasive wear behaviour of novel natural cellulosic pigeon pea stalk fibre reinforced epoxy biocomposites

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Abstract

The current research explores the possibility of reinforcing massively available, less utilised, low-cost agro-residue fibres in an epoxy matrix to create a new tribo-material. This study focuses on determining the three-body abrasive wear behaviour (volume loss and specific wear rate (SWR)) of natural cellulosic pigeon pea (PP) stalk fibre reinforced epoxy composites. Further, abrasive wear characteristics of untreated and treated E/PP20 (20 wt.% PP stalk fibre-reinforced epoxy) composites were analysed using Taguchi and ANOVA techniques. Untreated and treated biocomposite specimens were developed using the hand lay-up (open mould) technique. At 11.77 N, 23.54 N, and 47 N loads, the SWR of untreated E/PP20 composite was reduced by almost 5.03%, 3.68%, and 22.30% compared to epoxy specimens. Results of the untreated E/PP20 composite showed that the applied load was the main contributing parameter (54.72%), followed by sliding distance (21.82%) and sliding speed (15.31%). Results of the treated E/PP20 composite showed that the applied load was the main contributing parameter (48.96%), followed by sliding speed (26.24%) and sliding distance (20.78%). The regression model predicted the SWR with a pooled error ranging from 2.37% to −17.77% for untreated composite and 9.87% to −11.49% for treated composite, respectively. The alkali-treated E/PP20 composite exhibited better abrasive resistance than the untreated E/PP20 composite. Scanning electron microscopy images of the treated composites showed good fibre adhesion with the matrix. In addition, the surface of the treated composite showed no fibre pullout or ploughing compared to that of the untreated composite. Surface topography revealed the formation of more craters on the surfaces of the untreated composites and small-sized dispersed craters on the treated composites.

Introduction

There is a huge demand for natural and renewable resources (plant fibres, animal fibres, and mineral fibres) in developing eco-friendly and sustainable composites owing to their surplus availability, low cost [1], less weight, hazard-free, reuse, less abrasiveness, renewability [2], and excellent biodegradability [3]. These materials are essential for fulfilling the present and future infrastructure demands in many sectors like civil construction (decking, cladding, and fencing), automotive components (inner door panel, seatbacks, and headliners), home appliances, food packaging [4], eco-friendly concrete [5] and so on. The origin of natural plant fibres is from two sources. They are; purposefully grown plants and agricultural residues. Compared to the purposefully grown plants, agricultural plants can accomplish two goals at a time; production of food is the first concern, followed by composite development [6], electricity generation [7], pulp and paper production [8], building acoustics [9] and so on. India produces nearly 611 million tons [10] of agricultural residue. The use of this ample agro-material will not only fulfil the various human requirements but also contribute towards empowering the farmers of the nation and protecting the environment from the burning of its residues [11]. There are a lot of applications for

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agro-based biocomposites, including automobile interior parts, furniture, toys, and cascading. In accordance with Sustainable Development Goal SDG 12: Responsible Production and Consumption, a primary driver of the global economy relies on the use of natural resources and the environment. The use of natural resources contributes substantially to poverty alleviation and the transition towards low-carbon and green economies. Sustainable development goals include the sustainable management and efficient use of natural resources and reducing waste through prevention, reduction, recycling, and reuse [12].

Pigeon pea (Cajanus cajan), which belongs to the family of Fabaceae, is the sixth most important traditional food crop known to humanity. Each year, the PP crop is cultivated around 5.4 million hectares [13] globally. Within 6–8 months, the PP crop can reach up to 3 meters in height and produce an excellent woody residue along with its edible seeds. Nearly 55% cellulose content was noticed in the investigation of PP stalk material [14] which is comparable to other natural fibres [6].

Mechanical behaviours of polymeric composites have been studied for a long time, but their tribological properties are still being studied. This is because the tribological performance of polymers and their composites is complex and depends on many factors [15]. In the last few years, much research has been reported on the tribological behaviour of fibre-reinforced polymer composites [16–18]. An abrasive wear process occurs when hard asperities (abrasive particles) on one surface move or glide over a softer surface under load, removing the material and forming grooves on the softer surface. Three-body abrasion occurs when the material on a surface is removed by sliding and rolling the abrasive particles. Three-body abrasive wear is more prevalent in some engineering applications than two-body abrasive wear [19]. Abrasion is affected by many factors, including the surface properties of the material, the flow rate of hard asperities, the geometry and properties of abrasive particles, design parameters (generally load, distance, and speed), and environmental conditions [20]. Natural fibres like corn, palm, and sugarcane possess good frictional coefficients and are suitable for frictional materials [21]. The thorough literature review shows that the abrasive behaviour of polymer composites in abrasive testing depends mainly on fibre loading, normal load, sliding distance, and the size of the abrasive particles. Sugarcane polymer composites showed better wear resistance when compared to glass fibre composites in the study of sugarcane fibres/polyester composites [22]. The optimum fibre content and fibre length in composites can also influence the wear characteristics. In another study [23], fibre (Agave americana) of different lengths in the epoxy polymer was studied for wear characteristics. In this study, a length of 3 mm fibre exhibited excellent wear behaviour compared to a length of 5 mm and 7 mm fibres. In the study of palm fibre composites [24], an excellent improvement in wear characteristics has been observed by the inclusion of palm fibres in polymer compared to pristine polyester. The size of abrasive particles showed a direct influence on wear resistance in the study of betelnut fibre-reinforced composites [25]. The most common method of improving fibre properties and interfacial compatibility of fibre and polymer is the modification of lignocellulosic fibres through physical or chemical changes [26]. The inclusion of various chemical treatments for the natural fibres shows the reduction of wear rate in different studies [27–29]. The addition of natural eggshells as a filler in composites revealed a good resistance to erosion [30]. The Taguchi technique is a widely acceptable and highly powerful tool among researchers and engineers for optimising design problems whose response is influenced by multiple variables. This technique helps to get optimum combinations of variables with a minimum number of experiments (low-cost experimentations).

The literature survey cited above indicates that there is ample scope for understanding the abrasive wear mechanism of fibre loaded epoxy composites. Limited work has been done by the researchers on this abundantly available material compared to other agro-residue materials [31]. The ample availability of PP stalk material has encouraged the authors to investigate its potential use as reinforcement and study its composites for three-body abrasive wear characteristics. To the author's knowledge from the literature survey, no work has been reported on the experimental evaluation of three-body abrasive wear of PP/epoxy composites. In the present study, three compositions were considered, along with pristine epoxy, to study the influence of PP stalk fibres on abrasive wear behaviour. The Taguchi experimental design study was also carried out to understand the impact of applied load, sliding distance, and sliding speed on the SWR of the untreated and alkali-treated E/PP20 biocomposites. In this study, an attempt has been made for a new kind of material.

Experimental work

Materials

The PP crop is one of the main food crops which produces protein-rich pulses. In this work, abundantly available agro-residue (PP stalk fibres) was used as filler material, and commercially available epoxy LY 556 (Bisphenol-A Diglycidyl-Ether) with hardener HY951 was used as a matrix material to develop eco-friendly composites. Epoxy LY556 possesses excellent strength, adhesion, and water resistance properties. Epoxy resin with hardener was procured from 'Herenba Instruments and Engineers', Chennai, India, and sodium hydroxide

Table 1. Properties of untreated PP stalk fibre.

Properties	Equivalent value
Cellulose (%)	55.03
Hemicellulose (%)	18.33
Lignin (%)	18.32
Wax (%)	2.38
Ash content (%)	4.67
Moisture (%)	8.13
Density (g cc^{-1})	1.738
Crystallinity index	66
Organic matter (%)	94.63
Tensile strength (MPa)	131

(analytical grade) was procured from 'Veeresh Scientifics', Bangalore, India. PP plant is one of the woody-nature based plants, and hence stalk cannot be used as continuous fibre. Therefore, PP stalks need to be converted to flakes, particles, or pulp to develop biocomposites like wood-based composites. In the present study, only particles of PP stalks were used to develop biocomposites. The PP stalks were collected from farmers after harvesting their pulses. The small pieces (4 to 5 cm in length) of raw PP stalks were soaked in freshwater for removing dust and soluble contaminants for two days. These cut pieces were kept in open sunlight for moisture removal for about three days. The sun-dried pieces were kept in an air circulating oven at 100 °C for 24 h to remove the additional moisture content. These cut pieces of PP stalk were crushed into particles using a mechanical pulveriser, followed by ball milling. The PP stalk particles having less than 2 mm were isolated using the sewing process and used to develop PP/epoxy biocomposites. As a preliminary step, no chemical treatment was considered for PP stalk fibre to develop PP/epoxy composites. However, alkali treatment (5% sodium hydroxide) was used for PP stalk fibres to develop a treated E/PP20 composite to study the influence of chemical treatment on the abrasive wear behaviour. PP stalk fibres were mixed with 5% sodium hydroxide solution for 12 h at around 30 °C. The fibres were then washed in freshwater to remove the chemical content and defused with a 1% acetic acid solution. Further, the stalk fibres were rewashed in distilled water and dried using an oven drier. The various properties [14] of PP stalk fibre are given in table 1. The flow diagram of Pigeon pea stalk particle extraction process for composite preparation is shown in figure 1.

Development of PP/epoxy biocomposites

PP/epoxy composites were developed by the simple hand layup (open mould) procedure using a $200 \times 200 \times 5\ mm^3$ metallic mould followed by a light compression using a hydraulic press. Table 2 shows the designation and details of the weight fraction of PP stalk fibre and epoxy resin materials. In the present study, three compositions (E/PP15, E/PP20, and E/PP25) were considered, along with pristine epoxy for comparison purposes. Two polythene sheets and a mould releasing agent (silicone gel) were used to prevent the sticking of composites to the mould. The PP stalk fibre and epoxy with hardener as the per composition given in table 2 were mixed thoroughly using a centrifugal stirrer before pouring into the mould. After pouring the mixture, a light ramming process was used before closing the top plate of the mould. Then the entire mould assembly was shifted to a compression moulding machine. A pressure of 100 kPa was applied to the mould at around 30 °C and left for curing (24 h). After post-curing at 60 °C, specimens have been prepared for three-body abrasive wear testing as per ASTM G 65-04 [32] standards. The preparation of PP stalk particles and the development of PP/epoxy biocomposite is shown in figure 2.

Three-body abrasive wear test

A three-body abrasive wear test rig (MAGNUM) was used to determine the volume loss and specific wear rate (SWR) of epoxy and PP/epoxy composites at different conditions given in table 3. Chlorobutyl Rubber tyre wheel (diameter 200 mm) with shore 'A' hardness of 58–62 and silica sand (abrasive medium) of AFS 60 grade (morphology shown in figure 3) were used while testing the specimens.

The experiments were conducted at a constant speed of 200 rpm with varying forces for 600 m. A soft cloth soaked in acetone was used to clean the composite specimen surface, and a precision digital balance (Contech) with a 1 mg accuracy was used to measure their initial weight. The specimen was then carefully placed in the three-body abrasive test rig, and silica sand (abrasive) was directed to pass between the rubber wheel and the specimen (approximately 260 grams per minute). With the aid of the lever arm, the corresponding load is applied to the specimen at a constant speed of the rubber wheel for a given period. After the time interval, the specimen was removed from the test rig, cleaned thoroughly, and the final weight noted down. The three-body abrasive test rig setup is shown in figure 4. Each specimen's density was determined using METTLER TOLEDO

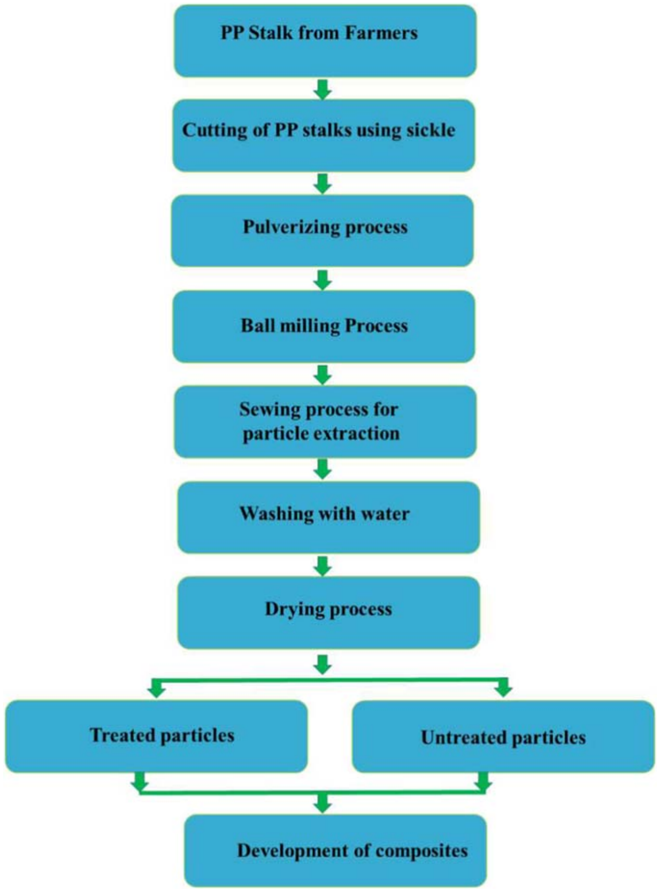

Figure 1. Flow diagram of Pigeon pea stalk particle extraction process for composite preparation.

Table 2. Specimen designation and composition of specimens.

	Composition		
Sample Designation	PP stalk fibre (wt.%)	Epoxy (wt.%)	Chemical treatment
E/PP0	0	100	Untreated
E/PP15	15	85	Untreated
E/PP20	20	80	Untreated
E/PPT20	20	80	Alkali treated
E/PP25	25	75	Untreated

ME204 analytical balance equipment, and the results are listed in table 5. The volume loss and SWR are calculated from equations (1) and (2). The time required to complete the total sliding distance for each specimen is calculated from equation (3).

$$Volume\ Loss(VL) = \frac{(IW - FW)}{\rho} \tag{1}$$

$$Specific\ wear\ rate = \frac{(VL)}{(P^*d)} \tag{2}$$

$$Time = \frac{(d^*60)}{(\pi DN)} \tag{3}$$

Here, ρ is specimen density (kg m^{-3}), IW is the initial weight (kg), FW is the final weight (kg), P is applied load (N), d is wearing distance (m), D is the rubber wheel diameter (m), and N is wheel speed (rpm).

Figure 2. Preparation of PP stalk particles and the development of PP/epoxy biocomposites.

Table 3. Three-body abrasive test conditions for epoxy and PP/Epoxy composites.

Rubber wheel speed (rpm)	Distance (m)	Time (s)	Force (N)
200	600	287	11.77
—	—	—	23.54
—	—	—	47

Taguchi experimental design

The Taguchi design approach is used to determine the optimum combination of parameters (load, distance, and speed) and to evaluate the percentage contribution of each influencing parameter on SWR. Taguchi L9 orthogonal array is considered to analyse both untreated and treated E/PP20 composite (three parameters with three levels as given in table 4). Nine experiments were conducted on both untreated and treated E/PP20 composite specimens using the procedure described in the earlier section. Each experiment was repeated three

Figure 3. Morphology of silica sand used in three-body abrasive wear testing.

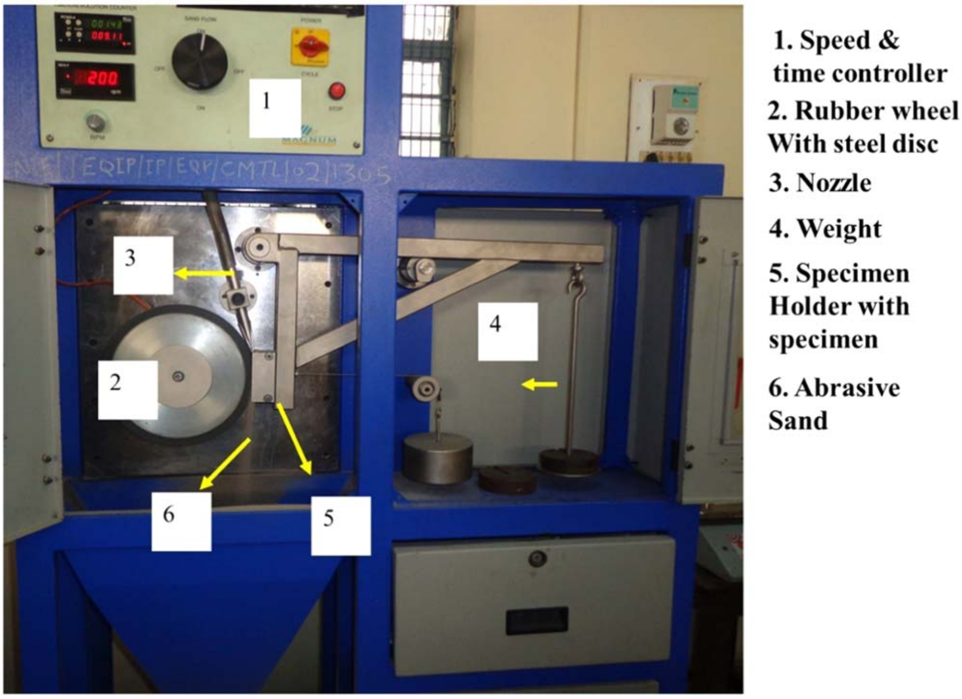

Figure 4. Three-body abrasive wear test rig used in wear testing of PP/epoxy composites.

Table 4. Factors and corresponding levels used in Taguchi L9 orthogonal array.

	Factors		
Level	Load (N)	Sliding distance (m)	Speed (m s^{-1})
1	11.77	300	1.0 (100 rpm)
2	23.54	600	2.1 (200 rpm)
3	47	900	3.1 (300 rpm)

times, and an average value was noted down for statistical calculations. All the results were analysed using the statistical software MINITAB 19. In the present study, the 'smaller the better' quality characteristic is taken, which is given in equation (4). A statistical analysis of variance is performed to predict the statistically significant parameters. The confirmation test was also considered by taking an arbitrary new set of levels to validate the

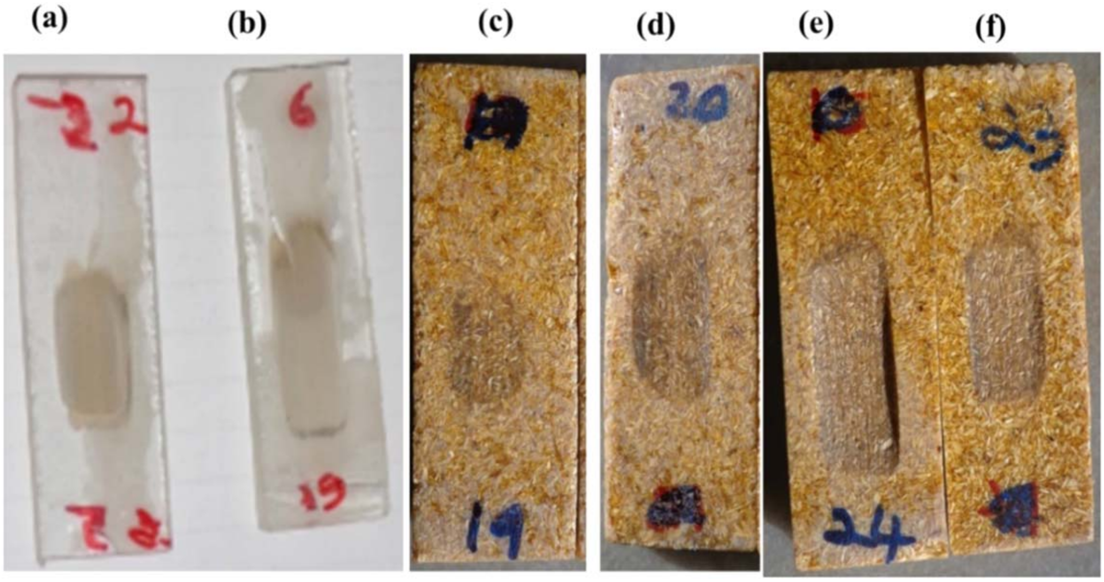

Figure 5. Tested specimens of epoxy and PP/epoxy composites: (a) & (b) Epoxy at 23.54 N & 47 N; (c) & (d) treated E/PP20 specimens at 11.77 N & 23.54 N; (e) & (f) untreated E/PP20 specimens at 47 N & 23.54 N.

experimental results with regression model results.

$$\frac{S}{N} = -10\ \log_{10}\left[\frac{(y^2)}{n}\right] \tag{4}$$

Here, $y_1, y_2 \ldots\ldots y_n$ denotes the response of three-body abrasive wear, and 'n' is the number of observations (experiments).

Morphological studies

The 10 mm × 10 mm portion was cut from worn-out specimens (both untreated and treated) and was examined using Zeiss (EVO 18) scanning electron microscope (SEM). Before capturing the micrographs of the abraded surfaces, a thin layer of gold film was vacuum evaporated onto the non-conductive fracture surfaces of the composite specimens (sputtering).

Results and discussion

Assessment of wear behaviour

The worn-out specimens of epoxy and PP/epoxy composites are shown in figure 5. Table 5 provides the results of volume loss and SWR of epoxy and PP/epoxy composite specimens at several loads with constant load and distance conditions. During testing, the abrasive particles penetrate the surface of the specimen and cause the breaking of fibres and matrix material. The volume loss of any natural fibre reinforced composites depends on the weight or volume fraction of the fibre in the matrix. The good adhesion between natural fibres and matrix can only be achieved when the right quantity of fibres is used to develop composites that have excellent mechanical and tribological properties.

Figures 6(a) and (b) depict the variation of volume loss and SWR of epoxy and PP/epoxy composites for three different loads (11.77 N, 23.54 N, and 47 N) with constant distance and speed. From figure 6 and its corresponding data, it is observed that the volume loss increases with an increasing load, but the SWR depends upon both load and the sliding distance. However, the volume loss and SWR of the E/PP20 composite are lower than its counterpart composites at a specified load. This shows that there is a good interfacial bonding between the fibre particles and the epoxy matrix, which causes an increase in wear resistance. Also, figure 6 demonstrates that the composites E/PP15 and E/PP20 have good wear-resisting capabilities compared to the E/PP25 composite, where a sudden rise in volume loss and wear rate was observed. This implies that there could be poor adhesion between PP stalk fibre and epoxy matrix material in the E/PP25 composite. A similar effect has been noticed at higher fibre content in the lantana camera fibre composites [33]. Increasing the percentage of fibres in the polymer matrix causes fibres to aggregate and form clusters, which means short particles are no longer distributed uniformly [34]. All composites, including epoxy, showed a small variation in volume loss up to

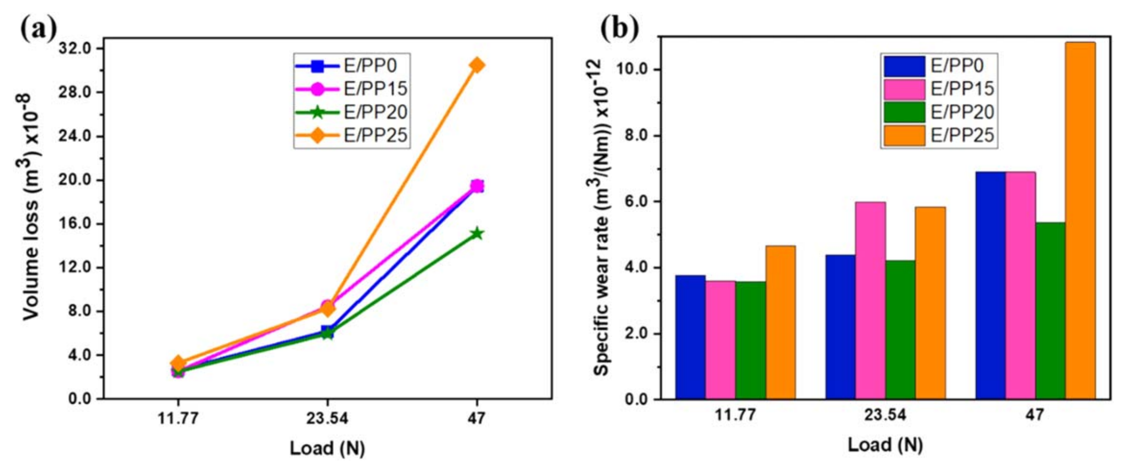

Figure 6. Three body abrasive characteristics: (a) Variation of volume loss; (b) Variation of specific wear rate.

Table 5. Three-body abrasive wear test results of epoxy and PP/epoxy biocomposites.

Exp. No.	Specimen	Load (N)	Density (grams/cc)	Initial Weight (grams)	Final Weight (grams)	Weight loss (grams)	Volume Loss (m^3) $\times 10^{-8}$	SWR (m^3/N.m) $\times 10^{-12}$
1	E/PP0	11.77	1.131	12.274	12.244	0.030	2.652	3.756
2	E/PP0	23.54	1.131	11.874	11.804	0.070	6.189	4.382
3	E/PP0	47	1.131	12.674	12.454	0.220	19.452	6.898
4	E/PP15	11.77	1.182	12.488	12.458	0.030	2.538	3.594
5	E/PP15	23.54	1.182	11.974	11.874	0.100	8.460	5.990
6	E/PP15	47	1.182	12.188	11.958	0.230	19.459	6.900
7	E/PP20	11.77	1.191	12.767	12.737	0.030	2.519	3.567
8	E/PP20	23.54	1.191	12.268	12.197	0.071	5.961	4.221
9	E/PP20	47	1.191	11.986	11.806	0.180	15.113	5.359
10	E/PP25	11.77	1.212	12.836	12.796	0.040	3.300	4.673
11	E/PP25	23.54	1.212	11.976	11.876	0.100	8.251	5.842
12	E/PP25	47	1.212	11.883	11.513	0.370	30.528	10.826

23.54 N. But the sudden increase in volume loss was observed at a higher load (47 N). Here, the fibre particles and matrix material might have broken because of the increased friction on the specimen surface caused by the rubbing of the abrasive particles. Also, abrasive particles gain higher energy from the rotating disc and cause more wear to the composites. This shows that the higher load has a more positive impact on the volume loss of all the composites. At loads of 11.77 N, 23.54 N, and 47 N, the SWR of untreated E/PP20 composite was reduced by almost 5.03%, 3.68%, and 22.30% compared to epoxy specimens. This indicates that the inclusion of PP stalk fibres in epoxy resin has a positive impact on the reduction of SWR. This could be due to the presence of the cellular structure of PP stalk fibres. Normally epoxy matrix exhibits brittle behaviour, whereas natural fibre shows a flexible behaviour. The combination of fibre and matrix reduces the brittle nature and thereby offers higher shearing resistance. This results in better wear characteristics of natural cellulosic fibre filled composites. Among all developed composites, the E/PP20 composite displayed excellent wear resistance characteristics (lower volume loss and SWR) at all the test conditions compared to its counterparts. This shows that 20 wt.% (PP stalk particles) is the optimum percentage for the epoxy matrix for tribological applications. Similar results have been noticed in the study of abrasive wear characteristics of orange peel particulate epoxy composites [17]. In this study, 20 wt.% of peel particles showed optimum wear behaviours.

Analysis of untreated and treated E/PP20 composite using the Taguchi method

In the previous section, the untreated E/PP20 composite exhibited better wear characteristics under all testing conditions than its counterparts. Therefore, the study was extended to determine how the influencing parameters (load, sliding distance, and speed) affect the SWR of untreated and treated E/PP20 composites. The experimental results of untreated and treated E/PP20 composites (at three parameters and three levels as per Taguchi L9 orthogonal design) and their corresponding signal-to-noise (S/N) ratio are tabulated in table 6. The ranking of influencing parameters using the S/N ratio for both untreated and treated composites is tabulated in

Table 6. Results of three-body abrasive wear of untreated and treated E/PP20 biocomposite.

	Control parameters			Untreated		Treated	
Exp. No.	Load (N)	Sliding distance (m)	Speed ($m\ s^{-1}$)	Exp. results SWR (m^3/N.m) $\times 10^{-12}$	Statistical results Signal to noise ratio (dB)	Exp. Results SWR (m^3/N.m) $\times 10^{-12}$	Statistical results Signal to noise ratio (dB)
1	11.77	300	1	3.091	230.19	2.605	231.685
2	11.77	600	2.1	3.567	228.95	2.960	230.574
3	11.77	900	3.1	3.329	229.55	2.842	230.929
4	23.54	300	2.1	5.350	225.43	4.973	226.068
5	23.54	600	3.1	4.518	226.90	3.907	228.163
6	23.54	900	1	3.662	228.72	3.236	229.799
7	47	300	3.1	8.158	221.76	6.345	223.951
8	47	600	1	4.853	226.27	3.202	229.891
9	47	900	2.1	4.248	227.43	4.072	227.804

Table 7. Response table for signal-to-noise ratio for untreated and treated E/PP20 composites.

Untreated E/PP20 composite				Treated E/PP20 composite			
Level	Load	Sliding distance	Speed	Level	Load	Sliding distance	Speed
1	229.6	225.8	228.4	1	231.1	227.2	230.5
2	227.0	227.4	227.3	2	228.0	229.5	228.1
3	225.2	228.6	226.1	3	227.2	229.5	227.7
Delta	4.4	2.8	2.3	Delta	3.8	2.3	2.8
Rank	1	2	3	Rank	1	3	2

table 7. For untreated composite, the applied load is the main influencing parameter, followed by sliding distance and speed. However, in treated composite, the sliding distance is the third contributing parameter. This shows good adhesion of the fibres with matrix material in the treated composite. In this scenario, high loads lead to the removal of more material from either the fibre region or the matrix region. It also causes more heat to be produced, thus causing an increase in temperature. Furthermore, the high temperature softens the matrix (epoxy), which in turn leads to poor adhesion between PP stalk particles and matrix material, resulting in increased material removal. From the results, the chemically-treated composite exhibited lower abrasive characteristics than the untreated composite under all conditions. The effect of control parameters for mean values of the SWR of both untreated and treated composites is shown in figures 7(a) and (b). In response graphs, all the parameters with the highest values yield a minimum SWR. From the response graphs, the average of the S/N ratios is found to be 227.24 and 228.76 for untreated and treated composites, respectively. Also, it is clear that the wear rate of both untreated and treated composite increases linearly with an increase in applied load and speed. However, a linear decrease in wear rate is observed with an increase in the sliding distance. The optimum condition for minimum SWR for both composites is 11.77 N (load), 900 m (sliding distance), and 1 $m\ s^{-1}$ (speed).

Analysis of variance (ANOVA) and the influencing parameters

ANOVA is used to analyse the experimental values and to investigate the percentage contribution of each influencing parameter on the output (SWR). The results of the analysis of variance of both untreated and treated composites for SWR are given in tables 8 and 9. For untreated composite, the applied load is the strongest and most important factor (54.86%) on SWR, followed by sliding distance and speed. The sliding distance (21.82%) and sliding speed (15.31%) are the second and third contributing parameters on SWR, respectively. For treated composite, the applied load is the first contributing parameter (48.96%), followed by speed (26.24%) and sliding distance (20.78%).

Multiple linear regression model

The regression model representing the relation between the SWR and the influencing parameters of the E/PP20 composite is obtained using MINITAB 19 statistical software. Regression equations for both untreated and treated E/PP20 composite are expressed in equations (5) and (6). The model provides the relationship between independent and response variables.

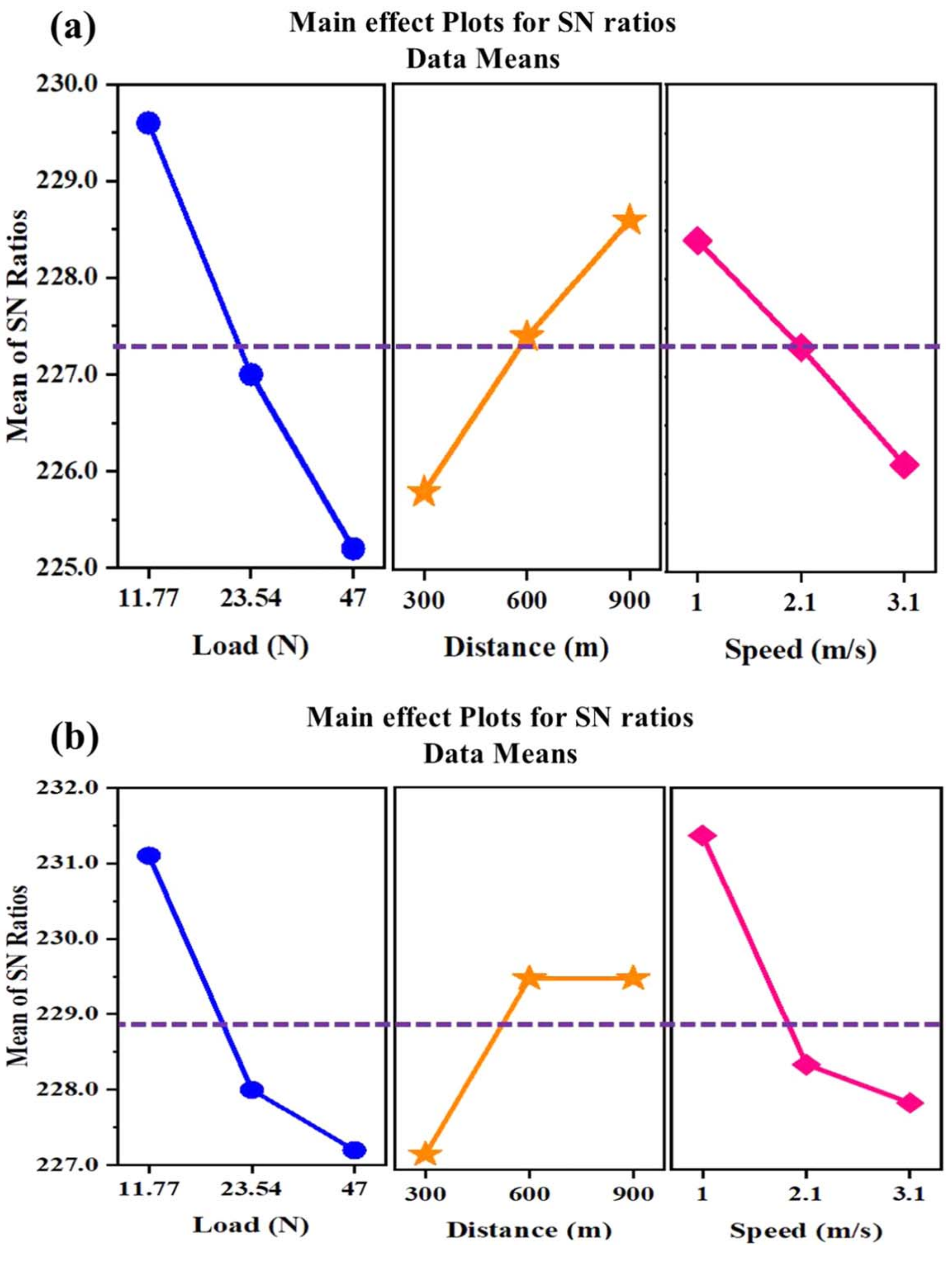

Figure 7. Main effects plot for SN ratio: (a) Untreated composite; (b) Treated composite.

Table 8. Analysis of variance for SN ratios (untreated E/PP20 composite).

Source	DF	Seq SS	Adj SS	Adj MS	F	Percentage of contribution (%)
Load	2	29.353	29.353	14.677	6.71	54.72
Sliding distance	2	11.706	11.706	5.853	2.68	21.82
Speed	2	8.209	8.209	4.104	1.88	15.31
Residual Error	2	4.372	4.372	2.186		8.15
Total	8	53.640				100

$$SWR = (3.054 + 0.0666 \times L - 0.002987 \times SD + 0.697 \times S) \times 10^{-12} \tag{5}$$

Table 9. Analysis of variance for SN ratios (treated E/PP20 composite).

Source	DF	Seq SS	Adj SS	Adj MS	F	Percentage of contribution (%)
Load	2	24.750	24.750	12.375	12.23	48.96
Sliding distance	2	10.508	10.508	5.254	5.19	20.78
Speed	2	13.266	13.266	6.633	6.55	26.24
Residual Error	2	2.024	2.024	1.012		4.0
Total	8	50.549				100

Table 10. Results of confirmation tests for both untreated and treated composites.

	Control parameters			Untreated E/PP20 composite			Treated E/PP20 composite		
				Specific wear rate (m^3/N.m) $\times 10^{-12}$			Specific wear rate (m^3/N.m) $\times 10^{-12}$		
Exp. No.	Load (N)	Sliding distance (m)	Speed (m/s)	Experiment	Regression model	Error (%)	Experiment	Regression model	Error (%)
1	9.81	250	1.25	3.257	3.836	−17.77	2.872	3.202	−11.49
2	19.62	500	2.30	4.581	4.472	2.37	4.216	3.799	9.87
3	33.35	750	3.34	4.750	5.369	−13.04	4.325	4.575	−5.77

$$SWR = (2.469 + 0.0453 \times L - 0.002096 \times SD + 0.647 \times S) \times 10^{-12} \tag{6}$$

Here, L is the load (N), SD is the sliding distance (m), and S is the rubber wheel speed(m/s). The coefficients for load and speed are positive in equations (5) and (6), but the coefficient for sliding distance is negative. This demonstrates that the rate of wear increases as the load and speed increase. Conversely, when the sliding distance increased, the rate of wear reduced.

Confirmation test

The confirmation test plays a vital role in validating the conclusions made during the analysis phase. The confirmation test was performed by taking a new set of levels for each factor (table 10) to compare the SWR of both the experimental model and regression model. A total of three experiments were conducted for each untreated and treated composite, and their experimental results were noted down using a similar procedure stated in the earlier sections. Also, the SWR for the new set of parameter levels was determined by the developed regression equations, and the results are tabulated in table 10. The error ranging from 2.37% to −17.77% and 9.87% to −11.49% was noticed for untreated and treated E/PP20 composites, respectively. This demonstrates that the developed regression equations (5) and (6) are adequate and feasible under three-body abrasive wear circumstances.

Comparison of specific wear rate of developed composites with other agri-based biocomposites

A significant improvement in tribological properties was observed with the addition of natural fibres to several polymers at different tribological conditions. To develop machine parts and devices with increased reliability for various applications, it is necessary to have a thorough understanding of the tribology of materials. Selecting optimum fibre loading in the matrix, fibre length, fibre treatment, sliding orientation, and combining with other fillers to form hybrid systems is key to achieving good tribological performance. In table 11, the complete details about the type of wear testing, the method used to develop the biocomposite, additives or chemical treatments used for fibres, the wear characteristics observed, and the major findings observed for various biocomposites made of agri-based materials have been outlined. Comparing the results of the study to those of other agri-based biocomposites, the PP/epoxy biocomposites are found to have similar wear resistance characteristics.

Morphological studies

The morphology of the abraded surfaces of both untreated and treated E/PP20 composites was studied using the micrographs captured by scanning electron microscope. Figure 8 shows worn out surfaces of untreated E/PP20 composite at different loads. A lower load (11.77 N) produced a smooth abrasive surface (minimal matrix and fibre breakage) on specimens owing to lower induced temperature. In addition, the lower load failed to shear off the particles from the matrix. This is supported by the lower weight loss of the composite specimens during

Table 11. Comparison of wear characteristics of PP/epoxy composites with other agri-based composites.

Sl. No.	Agri-based biocomposite	Type of tribological testing	Composite development method	Additives/ concentration	SWR/Friction/mass loss	Major findings	References
1	White/brown coir epoxy composites	Three-body abrasion resistance	Vacuum infusion	Alkali treatment	Mass loss 0.066 to 0.092 grams	Best wear resistance observed in brown coconut fibres/epoxy composites	[35]
2	Musa Acuminata/Corchorus Capsularis Reinforce Hybrid Composites	Two body abrasive wear (pin on disc)	Compression molding technique	Alkali treatment	SWR 0.3 to 0.55 $\times$ 10^{-4} mm^3/N-m	Tribological properties of the composites were influenced more by the fibre weight fraction	[36]
3	Rice husk/phenolic composites	Two body abrasive wear (pin on disc)	Hot compression technique	—	Coefficient of friction 0.4 to 0.6	Increase in rice husk content showed better wear performance	[37]
4	Jute/Coir Polyester Composites	Two body abrasive wear (pin on disc)	Compression molding method	Alkali treatment	Wear 13-131 micro-metre	Incorporation of ESP and NC particles in jute/coir hybrid composites improved the wear resistance of hybrid composites	[38]
5	Banana-Reinforced Composites	Two body abrasive wear (pin-on-disk tribometer)	Hand layup process	Water retting process	Wear 2.19 to 81.99 micro-metre	Wear is more in 0° orientation and less in 90° orientations	[39]
6	PP/epoxy	Three- body abrasive wear	Hand layup process followed by compression	Treated and untreated	SWR 2.6 to 4.07 m^3/N.m $\times 10^{-12}$	Treated PP/epoxy composites showed better wear resistance characteristics	Present study

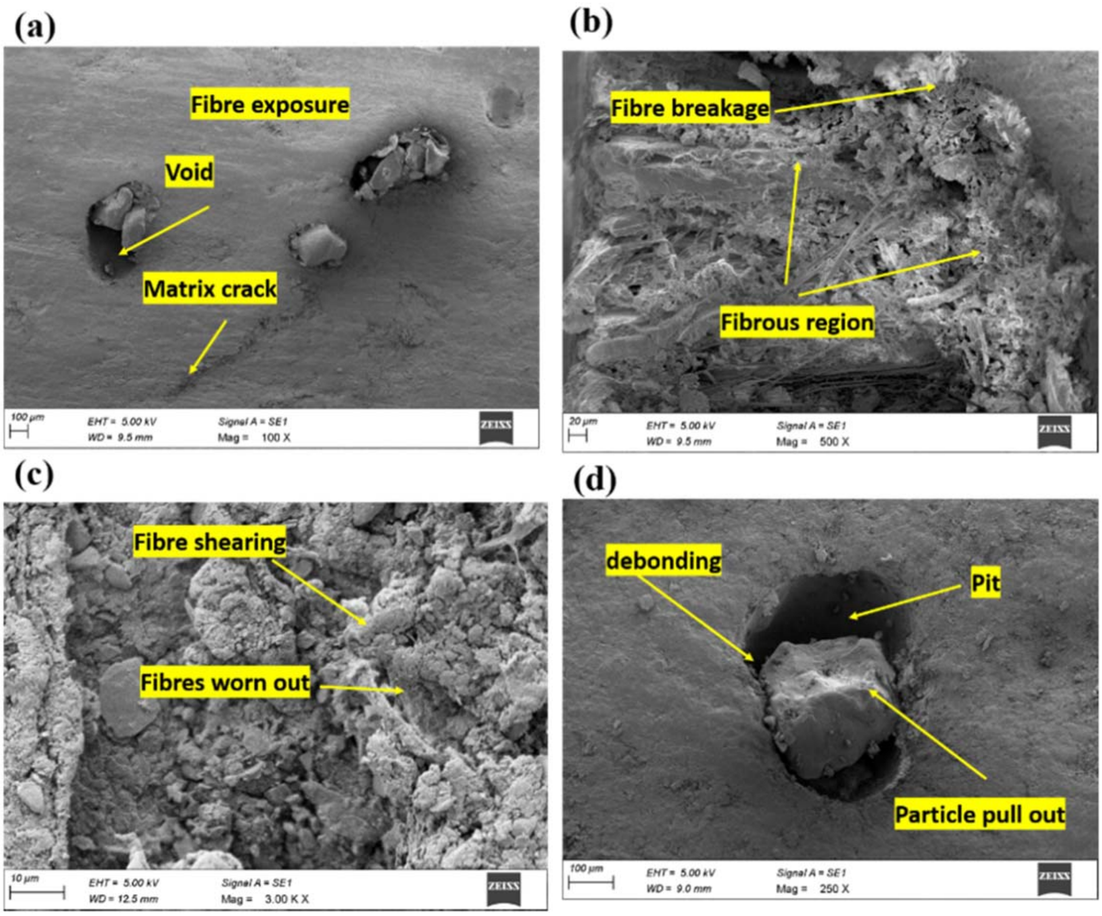

Figure 8. Morphology of abrasive wear surface of untreated E/PP20 composite: (a) at 11.77 N; (b) at 23.54 N; (c) & (d) at 47 N.

testing. But at a higher load (47 N), wear debris, micro-cracks, pits, broken particles, and wear scars (severe wear) were observed over the surface of the composite specimen. As a result of induced heat due to higher load, a complete fibre breakage and plastic deformation (softening) of epoxy resin was seen. However, PP/epoxy composite showed low matrix breakage due to the soft and flexible nature of the PP stalk fibre. In general, an increase in applied load (more frictional force) causes fracture initiation and the formation of ridges on the surfaces of the specimen (thermo-mechanical loading). When PP stalk particles were exposed to an abrasive media (silica sand) at a load of 47 N, the wear mechanism was dominated by fibre breaking and fibre pull out, resulting in void or pit formation. Figure 9 reveals the abraded surfaces of treated E/PP20 composite specimens at different loads. At lower load, a noticeable matrix and fibre breakage was not seen. This shows that chemically treated fibres have a better bonding with the epoxy matrix compared to untreated fibres. At higher load, fibre fracture, as well as matrix crack, was observed. But, a noticeable debonding was not observed. In addition, surface topography revealed the formation of more craters on the surfaces of the untreated composites and small-sized dispersed craters on the treated composites. This indicates that chemically treated PP stalk fibre-filled composites have better abrasive wear characteristics than untreated composites.

Conclusions

PP stalk agro-residue has been successfully used to develop eco-friendly composites. Based on the experiments and analysis on the three-body abrasive wear behaviours of the untreated and treated PP stalk fibre epoxy biocomposites, the following conclusions can be summarised.

- Volume loss of all developed biocomposites, including pristine epoxy, increased with increased applied load with constant sliding distance and speed. The drastic increase in volume loss and wear rate was noticed at a load of 47 N, causing deeper grooving on the specimen surface. The untreated composite with 20 wt.% PP stalk fibres (E/PP20) exhibited the lowest volume loss and SWR compared to its untreated counterparts (E/PP15 and E/PP25) and pristine epoxy at different loads.

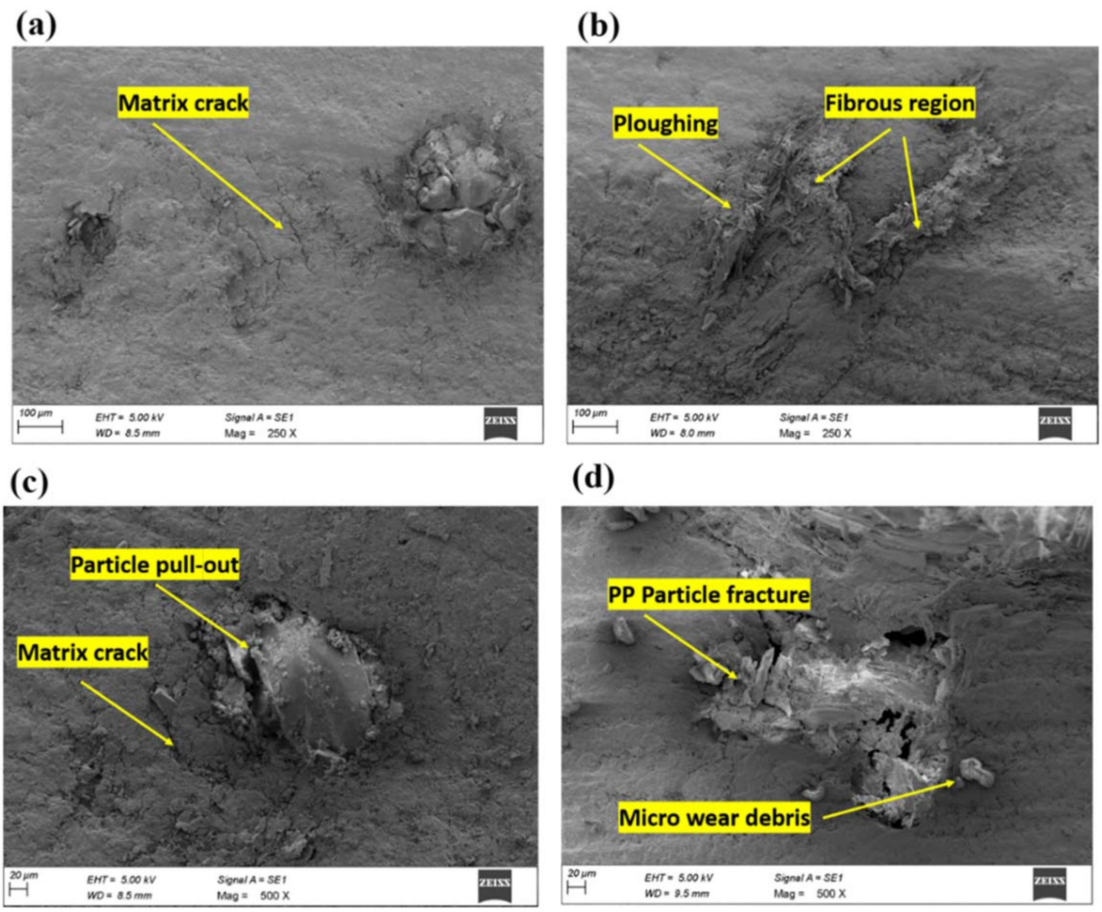

Figure 9. Morphology of abrasive wear surface of treated E/PP20 composite: (a) at 11.77 N; (b) at 23.54 N; (c) & (d) at 47 N.

- Alkali-treated E/PP20 composite exhibited a lower specific wear rate compared to untreated E/PP20 composite. From Taguchi analysis, the combination of a load of 11.77 N, a sliding distance of 900 m, and a speed of 1 m s^{-1} demonstrated the lowest SWR.
- ANOVA Results of the untreated E/PP20 composite showed that the applied load was the main contributing parameter (54.72%), followed by sliding distance (21.82%) and sliding speed (15.31%). Results of the treated E/PP20 composite showed that the applied load was the main contributing parameter (48.96%), followed by sliding speed (26.24%) and sliding distance (20.78%). The Regression model predicted the SWR with the pooled error ranging from 2.37% to −17.77% for untreated composites and 9.87% to −11.49% for treated composites, respectively.
- SEM images of the abraded surfaces of both untreated and treated E/PP20 composites revealed the presence of wear debris, fibre cracks, matrix crack, and pits. Also, microscopic observations showed that chemically treated PP stalk fibres have better adhesion with epoxy matrix compared to untreated fibres. Surface topography revealed the formation of more craters on the surfaces of the untreated composites and small-sized dispersed craters on the treated composites. The results of the newly developed composites showed comparable results with the existing biomaterials. Therefore, these materials can be used to develop new kinds of friction materials in future.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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