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MODELLING AND OPTIMIZATION OF TRIBOLOGICAL BEHAVIOUR OF COCONUT AND JUTE FIBERS WITH NANO ZnO FILLERS ON POLYMER MATRIX COMPOSITES

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ABSTRACT

Polyamide 66 hybrid composites were prepared with surface-treated coconut fibers and jute fiberswith nano ZnO fillers using mechanical mixer and sonication techniques. Tests were conducted on a pin-on-disc test rig, for online measuring of wear rate and coefficient of frictionhas been developed. Experiments based on central composite rotatable design were carried out to study the effect of various input parameters, viz. normal force, sliding velocityand reinforcements. The mathematical models were developed between input and responses by using response surface methodology. Analysis of variance test had also been carried out to check the competence of the developed empirical models. It is found that the wear resistance of JF/ZnO/PA is greater than CF/ZnO/PA, whereas the frictional resistance of CF/ZnO/PA is found to be better than JF/ZnO/PA. Worn out surfaces were observed through scanning electron microscope.

Keywords: *Polymer composites,Wear rate, Co-efficient of friction, SEM, ANOVA, RSM, Coconut fiber and Jute fiber*

1. Introduction

In different sectors of national economy polymer materials are increasingly exploited. Thermoplastics composites have replaced the metals in light duty load bearing applications because of its superior mechanical properties, lightweight, economic fabrication, good chemical resistance, utmost hardness and stiffness.Nowadays, great attention is paid to polymer composites, because in this way one can obtain new materials with advanced or even new properties. Polyamide 66 (Nylon) were the first to be recognized as an engineering thermoplastic. Gears are the basic power and motion transfer elements used in every machine. The most important properties in gear design are cost, weight, silent operation and easy manufacturability[1].Another way of polymer modification, that has recently achieved a great interest, is development of hybrid polymer nanocomposites. In several studies it is reported about modification of broad range of thermoplastic polymers with various fibers and nanofillers. Considerably less effort, however, is devoted to the development of hybrid polymer nanocomposites. Therefore, in the current project hybrid thermoplastic polymer composites are investigated as potential candidates for developing of specific nanocomposites.Bearings are made of polymer-basedcompositesare often used when corrosive environments toavoid the rust. Polyetheretherketone (PEEK) matrix, reinforced with short carbon fibres used for sealing applications [2]. The wear performance of unfilled glass-epoxycomposite possesses strong relation with the applied load and temperature. Wear loss in both type of composites increases with an increase in temperature and normal force [3].The investigational results indicated that the wear rate and the friction coefficient of PA6 decreased with the addition of nano-MoS2[4].ZnO and Al2O3 nanoparticles aid in friction reduction. The friction reduction effect is obvious for smooth surfaces. Wear analyses for a polished surface shows that the nanoparticles act like abrasive wear particles and produce less wear on the surfaces, while harder particles produce more wear[5].Nanocaly is effective filler that contributes to the reduction in wear loss and specific wear rate. Wear resistance improved as increase in nanoclay content in the composites[6]. Utilizing SiO2 as filler in PTFE, it is possible to increase the wear resistance of the thin film[7].The arylboronic acid treatment can efficiently improve the interfacial adhesion of carbon fiber(CF)/PA66 composites. Tensile strength and tensile modulus values increased after arylboronic acid treatment [8].Polyamide 12 (PA12) composites, reinforced by Ti40.83Zr40.83Ni18.34 quasi crystal powder, possess

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significantly higher hardness and better wear resistance compared with neat PA12. The corresponding reduction in wear may be ascribed to the low coefficient of friction and high hardness of the quasi crystal powders [9].The abrasive wear rate increases with the increase in applied load value and with the increase in roughness of the abraded surface [10]. The main intention of this research is to make a comparative study on the friction and wear behaviour of two different hybrid composites. It is also expected that this work can be helpful to the use for bearings, pistons rings, impellers, etc., at elevated temperatures.

2. Experimental Study

2.1 Materials and manufacturing of hybrid composite specimen

In the present study thixotrophic orthopthalic polyamide resin was considered as the matrix and nano ZnO is a filler with density of 3g/cm3 with mean dia of 19 nm.It is an anti electromagnetic or conductive phase due to their current characteristics γ-aminopropyl triethoxnysilane (APS) is used to disperse/exfoliate the ZnO nano fillers in polyamide matrix. The jute fibers (JF) and coconut fibers (CF) are the reinforcements with the same dia of 6 µm. These fibers were autonomously oxidized by boiling in sodium hydroxide acid under reflux for 3 hrs (T=300C), then it was washed with distilled water to obtain neutral pH. This behavior was engaged just before the preparation of polyamide hybrid composites. These hybrid composites were prepared bymechanical mixer followed by sonication process,for better curing capability and mechanical properties thus make it particularly valuable for large component manufacture at relatively low cost. While curing, the polymerization reaction causes cross-linking among individual linear polymer chains with reinforcements which improves the tribological properties. The volume contents of jute and coconut fibers are constantly maintained at1.5%Vf, whereas the volume fraction of nano ZnO varied from 1.0 to 3.0% Vf. Table 1 shows the input parameters and their levels used in this study.

2.2 Friction and wear tests

The cylindrical hybrid composite pins of 10mm diameter and 20mm length were tested on pin-on-disc (POD) wear tester with data acquisition system, as per ASTM-G99 standard. It isversatile apparatus designed to study the wear behavior under different sliding conditions. Sliding normally occurs between a stationary pin and rotating disc. The disc rotates with a help of a D.C. motor having the speed range of 0-600 rev/min.

Table 1 Process parameters and their levels

Polyamide with CF hybrid composites					
Parameter	**-1.682**	**-1**	**0**	**+1**	**+1.682**
Normal force (N)	2	3	4	5	6
Sliding Velocity (m/sec)	0.2	0.4	0.6	0.8	1.0
Reinforcements (CF + nano ZnO)	(1.5 +1) =2.5	(1.5+1.5) =3.0	(1.5+2.0) =3.5	(1.5+2.5) =4.0	(1.5+3.0) =4.5

Polyamide with JF hybrid composites					
Normal force (N)	2	3	4	5	6
Sliding Velocity (m/sec)	0.2	0.4	0.6	0.8	1.0
Reinforcements (JF + nano ZnO)	(1.5 +1) =2.5	(1.5+1.5) =3.0	(1.5+2.0) =3.5	(1.5+2.5) =4.0	(1.5+3.0) =4.5

The smooth and hardened steel disc surface (SS 316 HSS, hardness 72 HRC) served as a counterpart and was finished by an abrasion against 1200-SiC grade sheet with provided a roughness of Ra 0.6-0.7µm. The pin was at right angles to the counterpart and parallel to the sliding direction. Prior to the test the pin was rubbed over a 400-SiC grade sheet to ensure proper contact between pin and disc. The surface of both pin and disc were cleaned with a soft paper soaked in acetone prior to actual testing. The material loss from the composite surface was measured using a precision electronic weighing balance with an accuracy of ±0.001 mg. The real time data has been collected from data acquisition system. COF was measured by a load cell sensor directly from the computer running friction-measuring software.

3. Response Surface Methodology

Response surface methodology (RSM) is the procedure for determining the relationship various between input parameters with the various output parameters criteria and exploring the effect of these process parameters on the coupled responses [11]. A second-order polynomial response surface empirical model can be developed as follows to evaluate the parametric effects on the various tribological criteria.

Table 2: Experimental matrix and responses

Sl. No	Normal Force (N)	Sliding velocity (m/sec)	RF	CF/ZnO/PA Hybrid Composites		JF/ZnO/PA Hybrid Composites	
				WR (mm^3/ Nm)	COF (μ)	WR (mm^3/ Nm)	COF (μ)
1	3	0.4	3.0	0.956	0.110	0.613	0.265
2	5	0.4	3.0	0.951	0.125	0.621	0.290
3	3	0.8	3.0	0.953	0.127	0.619	0.279
4	5	0.8	3.0	0.955	0.139	0.609	0.301
5	3	0.4	4.0	0.842	0.138	0.439	0.391
6	5	0.4	4.0	0.836	0.142	0.422	0.408
7	3	0.8	4.0	0.837	0.140	0.435	0.450
8	5	0.8	4.0	0.838	0.145	0.422	0.401
9	2	0.6	3.5	0.915	0.120	0.428	0.365
10	6	0.6	3.5	0.910	0.143	0.413	0.397
11	4	0.2	3.5	0.913	0.127	0.410	0.409
12	4	1.0	3.5	0.907	0.147	0.452	0.409
13	4	0.6	2.5	1.015	0.129	0.921	0.402
14	4	0.6	4.5	0.755	0.165	0.481	0.411
15	4	0.6	3.5	0.875	0.113	0.501	0.420
16	4	0.6	3.5	0.879	0.113	0.512	0.421
17	4	0.6	3.5	0.880	0.115	0.421	0.421
18	4	0.6	3.5	0.879	0.113	0.489	0.411
19	4	0.6	3.5	0.879	0.112	0.490	0.402
20	4	0.6	3.5	0.878	0.115	0.511	0.311

$$Y_u = a_o + \sum_{i=1}^{n} a_i x_i + \sum_{i=1}^{n} a_{ii} x_i^2 + \sum_{i<j}^{n} a_{ij} x_i x_j + \varepsilon \quad (1)$$

Where *Yu* is the corresponding response, e.g. the WR and COF created by the various process variables of tribological parameters. Here$a_i$represents the linear effect of x_i, a_{ii}represents the quadratic effect of $x_i$and a_{ij}reveals the linear-by-linear interaction between $x_i$and x_j. The second term under the summation sign of the polynomial equation i.e. Eq. (1) attributes to linear effects, whereas the third term of the above equation represents the higher order effects and lastly the fourth term of the above equation includes the interactive effects of the process parameters. Due to wide range of factors, it was decided to set three factors, five levels. The present investigation studied the results of the effects of NF, SV and RF on WR and COF. The design matrix comprises, a full replication of 2^3 (=8) factorial design, plus six centre points and six star points at a distance of 1.682 units from the centre points. The first 8 rows correspond to the factorial portion, the rows from 9 to 14 correspond to the axial portion and last 6 rows correspond to the centre point portion. Therefore, the experimental design consists of 20 (8 + 6 + 6 = 20) experimental runs. The experimental matrix that was adopted in the present study in the coded form is shown in table.2.

3.1 Development of empirical models based on RSM

After knowing the values of the observed response, the values of the different regression coefficients of second order polynomial empirical equation i.e. Eq. (1) have been evaluated and the empirical models based on RSM have been developed by utilizing test results of different responses obtained through the entire set of experiments by using a computer software, MINITAB.16. Based on Eq. (1), the effects of various tribological process variables on WR and COFhas been evaluated by computing the values of different constants of Eq. [1]utilizing the relevant experimental data from table 2. The empirical relationship for correlating the WR and COF for PA/JF/ZnO hybrid composites as considered tribological process parameters shown in Eq.(2) and (3) is obtained as follows,

$$WR= 1.32703 - 0.11760A - 0.40523B - 0.17380 C + 0.01609A^2+0.04915B^2-0.00914C^2-0.10750AB+0.03550AC+0.21250BC \quad (2)$$

$$COF = 0.310468 - 0.023756A - 0.36506B - 0.068239C +0.000227A^2-0.1068018B^2+0.003409C^2+0.009375AB +0.004250AC+0.038750BC \quad (3)$$

The empirical relationship, obtained for analyzing the influences of the various dominant tribological parameters on the WR and COF for PA/CF/ZnO hybrid composites as considered tribological process parameters shown in Eq.(4) and (5) is obtained as follows,

$$WR=1.32703 - 0.11760A - 0.40523B - 0.17380C + 0.01609A^2 + 0.49150B^2 - 0.00914C^2- 10750AB+0.03550AC+0.21250BC \quad (4)$$

$$COF=0.310648 - 0.023756A - 0.36506B - 0.068239C + 0.000227A^2 - 0.106818B^2 + 0.003409C^2 + 0.009375AB +0.004250AC+0.03875BC \quad (5)$$

3.2 Analysis of the developed empirical models

The analysis of variance (ANOVA) and the F-ratio test have been performed to justify the goodness of fit of the empirical models. The calculated values of F-ratio for lack of fit have been compared to standard values of F-ratio corresponding to their degrees of freedom to find the adequacy of the different developed empirical models. The F-ratio has been calculated as a ratio of Mean sum of square of source to mean sum of experimental error.

The fit summary recommended that the empirical model is statistically significant for analysis of WR. The value of R^2over WR for CFand JF are 99.76% and 98.15 %,which means that the empirical model provides an excellent explanation of the relationship between the independent variables (factors) and the response (WR).The associated P-value for the model is lower than0.05(i.e., p= 0.05,or 95% confidence) indicates that the model is considered to be statistically significant.Similarly the value of R^2 over COF for CF andJF are 98.45% and97.12 %,which means that the empirical model provides an excellent explanation of the relationship between the independent variables (factors) and the response (COF).The associated P-value for the model is lower than 0.05(i.e., p= 0.05,or 95% confidence), which indicates that the model is considered statistically significant.

The standard percentage point of distribution for 95%confidence limit is 5.05. The F- valuesof WR and COF for CF and JF of the hybrid composites follows(2.16, 1.65) and(2.14, 1.20) for lack of fit are smaller than the standard value. Thus both the models are adequate.

4. Results & Discussion

The estimated response surface plot for WR is presented in Fig.1.WR increases with theincrease of normal force. It is attributed to the excessive frictional heat originated for theincreasing of normal force. The frictional heat can result in excessive deterioration of thecomposites transfer from materials to the disc counterpart. Furthermore, increase ofnormal force will lead to excessive damage of the fibers and thus result in adisproportionate increase in wear loss[12]. On the other hand by the addition ofreinforcement, WR decreases. The reason may be that the oxidation improved the nano particles-matrix interfacial adhesion; only a few particles are debonded and the broken particles were contained in the matrix [13]. Further increasing of nano particles WR also increases.This is because of the resistance of the transfer film on the slide of the sample was small and WR also small. A line shaped transfer film also covered the surface of the steel disc, it was protuberant. During sliding, this kind of transfer film could puncture the surface of composites, plough some grooves and form wear debris simultaneously, and resulted in larger sliding resistance than that of smooth one, and increased WR [14].

Surface plot of WR with respect tonormal force and sliding velocity is shown in Fig.2. It is clear from the graph that slidingvelocity and normal force increases, WR increases. This could be due to during slidingthe two surfaces are in contact and move relative to each other under high slidingconditions, hence the friction that exists between these two surfaces converts kineticenergy into heat energy because of brittleness behavior of polyamide resin which causesdecrease the shear strength and thermal softening of the composites which results in anincrease in the WR.

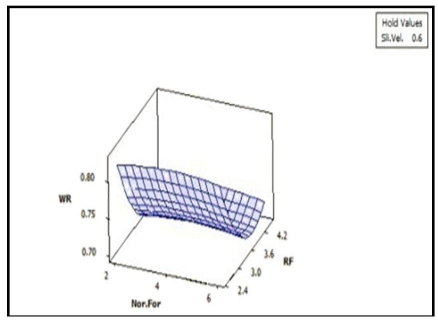


Fig.1 Influence of Nor For and RF On WR

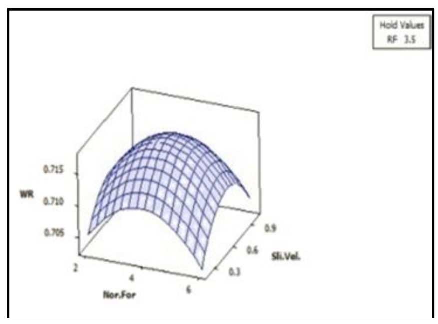


Fig.2 Influence of Nor For and Sli Vel On WR

Fig.1&2 CF/ZnO/PA Hybrid Composites (WR)

Further increase in normal force and sliding velocity WR decreases.It is owing to the mechanism of heat energy barrier by third body particles at the counterfaceis not sufficient, as a result, particles cannot penetrate deeper into the matrix, whichacts as a solid lubricant and form wear scar, plucked and ploughed marks andmicrofracture on the composites[15].

The estimated response surface plot for WR is observed in Fig.3. WR increases monotonously with normal force. It is partially attributed to the excessive frictional heat originated at the counter surface. This results in excessive deterioration of the composites and more debris transfer to disc. Furthermore increase of normal force will lead to excessive damage of the fibers and thus result in a disproportionate increase in wear loss [16]. On the other hand by the addition of reinforcements WR linearly increases, it shows that the aggregation of reinforcements uniformly distributed on the sub surface of polyamide composites which reduces the distruction of polymer composites during wear process[17].

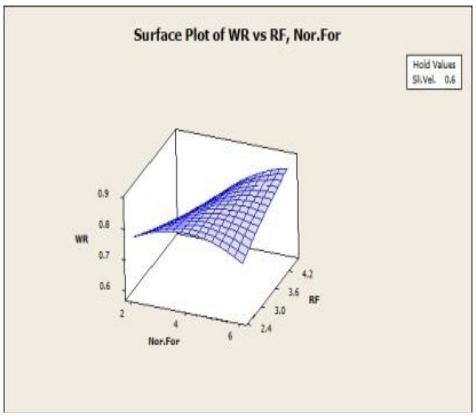


Fig.3 Influence of Nor For and RF On WR

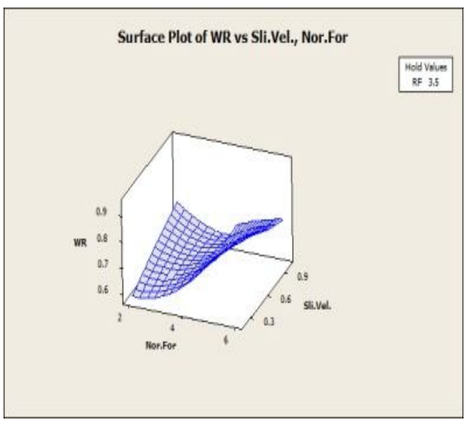


Fig.4 Influence of Nor For and Sli Vel On WR

Fig. 4&5 JF/ZnO/PA Hybrid Composites (WR)

Plots of sliding velocity and normal force on the influence on WR are shown in Fig.4. WR increases with normal force. It shows while sliding, plastic flow traces and plowed marks appeared on the worn surfaces of composites, which are parallel to sliding direction, it suggests that the wear process was governed by the plastics deformation. At the same time, the uniform and continuous transfer film formed on the surface of the disc. As for as the role of transfer film, it is widely believed that the transfer film can prevent the polymer composites from directly contacting the hard metal surface and intensely decrease plowing damage and improve the wear resistance [18]. WR linearly decrease with increase of normal force, this would be attributed that the real contact area and the decrease in the shear strength of composites [19].

Fig.5 highlights the COF on normal force and reinforcement. It can be found thatCOF decreased almost linearly with increasing the normal force. While sliding, thefriction-induced heat increased rapidly with the increasing load, which resulted in twocontrary effects on the COF. On the one hand, the real contact area increased, increasingthe COF.

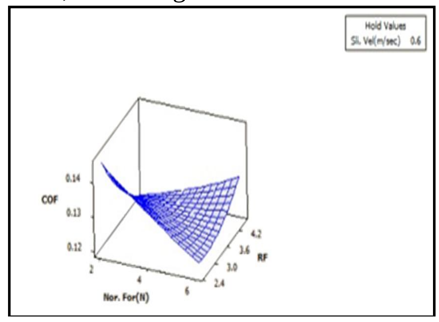


Fig.5 Influence on Nor For and RF On COF

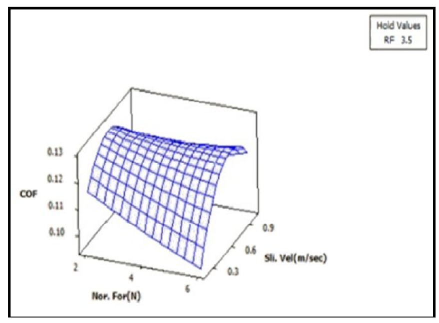


Fig.6 Influence on Nor For and Sli Vel On COF

Fig. 5&6 CF/ZnO/PA Hybrid Composites (COF)

On the other hand, the worn surface became smoother with increasing load, theshear strength decreased, and thus the COF decreased. With an increase of reinforcementCOF increases for all levels of normal force. This is attributed that reinforcement particlesare generally embedded in the polyamide matrix at the frictional surfaces, and easilymove to the counterpart surface of the matrix and it leads to plough some grooves andminute puncture occurs on the transfer-film during sliding. Further addition ofreinforcement, it is impossible to rub against the counterpart surface of the composites[20]. The effect of normal force and sliding velocity on COF is shown in Fig.6. Initially,surface of the composite and the steel counterparts were rough and thus stronginterlocking took place, resulting in a high COF. As the wear process continued, therough profiles of the steel counterpart that protected the materials, and then the COFdecreases. Therefore, when the sliding velocity was higher, which caused a rise of frictiontemperature, the viscosity of the surface layer would decrease, hence the COF was lower[21]. The normal force has the same effect of sliding velocity.

Fig.7 and Fig.8 represents the COF on normal force, sliding velocity and reinforcement. Initially COF increases after attain the maximum limit it decreases for all the cases. It is known that polymers are visco elastic materials and that their deformation under the load is viscoelastic. Moreover the critical surface energy of polymer leads to frictional heat temperature on the contact area, which leads to relaxation of polymer molecule chains; it causes increase in COF increases[22]. After attain the maximum limit it tends to decreases for both, it is due to formation of transfer film on worn surfaces, it can polish to a certain extent owing to softening of the adhesive resin that explains the decreasing of COF[23].

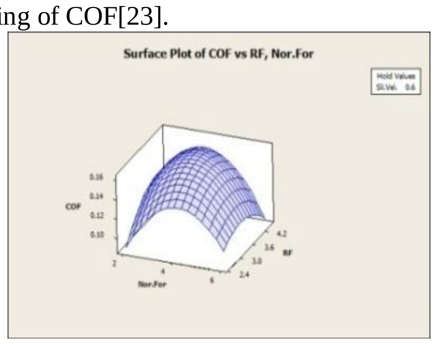


Fig.7 Influence of Nor For and RF On COF

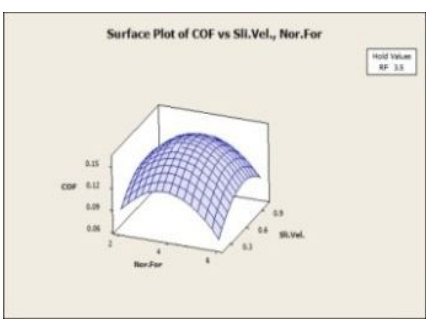


Fig.8 Influence of Nor For and Sli Vel On COF

Fig. 7&8 JF/ZnO/PA Hybrid Composites (COF)

5. Multi-Objective Optimization Using Response Surface Methodology

As analysis for the optimization of the process parameter has been carried out using RSM optimization technique. The desirability of the optimization has also been calculated to show the feasibility of the optimization parameter.

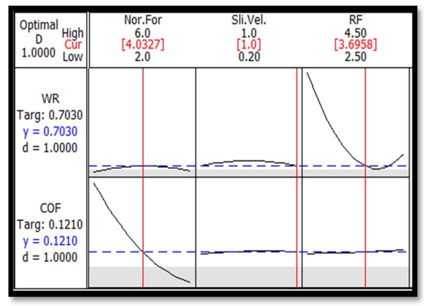


Fig.9 Optimal chart through RSM CF/ZnO/PA)

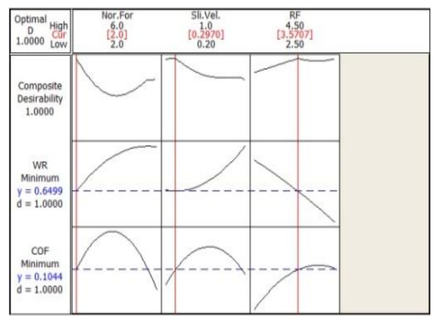


Fig.10 Optimal chart through RSM (JF/ZnO/PA)

Fig. 9&10 JF/ZnO/PA Hybrid Composites (COF)

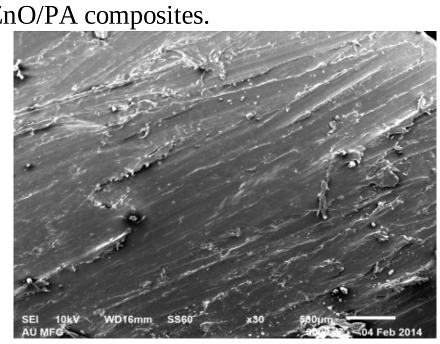
After that both the responses have been optimized with respect to the target value, thus getting the parameter setting for the whole process optimization. Desirability for the whole process optimization has been calculated to show the feasibility of optimization, i.e., to explore whether all the parameters are within their working range or not. The goal was to minimize WR and COF while both are considered at a time. The CF/ZnO/PA hybrid composite desirability is close to one. Fig.9. and10 exhibits the optimization plot for the both responses for both hybrid composites. The optimum values obtained for WR is 0.7030 (mm^3/Nm) and for COF is 0.1210 (μ) and the relevantparameters such as normal force, sliding velocity and reinforcement are 4.0327 N, 1.0 (m/sec) and 3.6958 (%Vf) respectively.The JF/ZnO/PA hybrid composite desirability is close to one. Figure 10 exhibits optimization plot for the both responses. The optimum values from the plot are WR=0.6499 (mm^3/Nm) and COF=0.1044(μ) and the relevant parameters like normal force, sliding velocity and reinforcements are 2.0 N, 0.2970(m/sec) and 3.5707(%V_f) respectively.

6. SEM Investigations

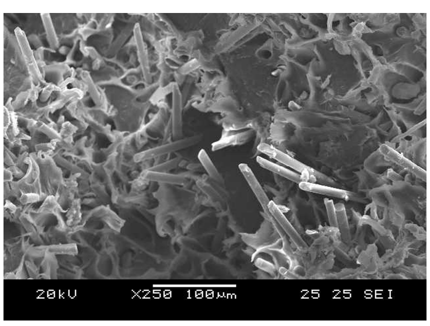
6.1 Worn surfaces

To find out further information about the effect of fiber fillers on the COF and WR behavior of the ZnO/ PA composites, the worn surfaces and transfer films of ZnO/PA composites filled with different fibers were studied by SEM, as shown in Fig.11and Fig.12.

Fig.11. (a)-(b) shows the SEM images of worn surfaces of JF/ZnO/PA and CF/ZnO/PA composites respectively. Fig.11. (a) indicates that the smooth worn surfaces of JF/ZnO/PA composites could reduce the adhesive wear of ZnO/PA composites. Meanwhile, many small ZnO fillers exist on the worn surfaces of PA composite. It can be seen from Fig.11. (b) that the worn surface of the CF/ZnO/PA composites is relatively rough. Also, it is evident that CF are peeled off from the composites, which well corresponds to the higher WR of CF/ZnO/PA composites.



(a) JF/ZnO/PA

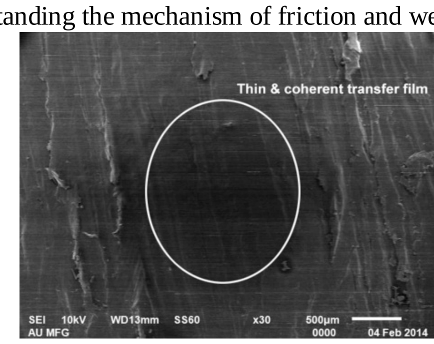


(b) CF/ZnO/PA

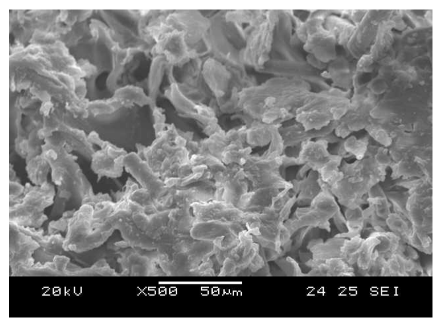
Fig.11 SEM Images of Worn Surfaces

6.2 Transfer films

As is well known, that the wear studies of polymers and their composites sliding against a steel disc under dry friction conditions are strongly influenced by their ability to form transfer films on the counterface. Once the transfer film is formed, COF between the matrix and the polymer transfer film, irrespective of the composition of the substrate [24]. Hence, it is worthwhile to explore the transfer film for understanding the mechanism of friction and wear.



(a) JF/ZnO/PA



(b) CF/ZnO/PA

Fig.12 SEM Micrographs for Transfer films

The SEM micrographs of the transfer film of ZnO/PA composites are shown in Fig.12. It can be seen that the transfer film of CF/ZnO/PA composites is rough, which can be explained by the larger size of CF is shown in Fig.12 (b). On the contrary, a smooth transfer film is gained for JF/ZNO/PA composites, which provides an excellent anti wear property for the jute based composites is shown in Fig.12 (a).

7. Conclusion

1. The experiments were conducted on a pin on disc wear testing machine for coconut based and jute based hybrid composites. The responses were WR and COF. The second order polynomial model developed for WR and COF were used for optimization.

2. The optimum values of WRand COF obtained through RSM for coconut based hybrid composites were 0.7030 mm^3/Nm and 0.1210 µ respectively and the relevant parameters like normal force, sliding velocity and reinforcements are 4.0327 N, 1.0 m/sec and 3.6958 Vf respectively.

3. The optimum values of WR and COF obtained through RSM for jute based hybrid composites were is 0.6499mm^3/Nm and 0.1044µ and the relevant parameters normal force, sliding velocity and reinforcements are 2.0 N, 0.2970 m/sec and 3.5707V_f respectively.

4. The SEM studies indicate that JF/ZnO/PA composites reduce the adhesive wear of ZnO/PA composites obviously and be favorable for forming a smoother transfer film.

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