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RESEARCH

Analyzing the Tribological Behavior of Titanium Dioxide (TiO_2) Particulate Filled Jute Fiber Reinforced Interpenetrating Polymer Network (IPNs) Composite by using Taguchi Optimization Technique

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A B S T R A C T

The tribological behavior of titanium dioxide (TiO_2) filled with jute fiber reinforced interpenetrating polymer networks (IPNs) were studied by using a pin-on-disc wear test rig at dry slide conditions. During the study, 70 wt.% of epoxy and 30 wt.% of polyurethane have been chosen as the base matrix material. As well, various proportionate of titanium dioxide such as 0%, 3% and 5% have been utilized to fabricate the particulate reinforced IPN laminate. To do the wear analysis such as materials type (A), sliding speed (B) and applied loads (C) were kept as the influencing parameters whereas coefficient of friction (COF) and specific wear rate (SWR) have been seen as the outcome of the entire study. Taguchi technique was chosen to plan the entire experiments. Similarly, an orthogonal array and analysis of variance (ANOVA) was employed to examine the impact of process parameters on wear of the IPN laminate. Out of all, results show that, addition of particulate materials into the composite predominately increased the specific wear resistance of the IPN laminate significantly.

1. INTRODUCTION

Composites are just two or even more constituent materials together that have differing chemical or physical characteristics. They result in a substance with traits that are distinct from their initial characteristics once combined. The matrix and fibre are a composite's two primary constituents. Nowadays most of the researchers are started to increase the wear resistance of the composite such a way adding particulate matter into the matrix materials. Also, to improve the wear resistance of the composites frequently glass, carbon, aramid and natural fiber have been extensively explored by most of the investigators by using the above materials as reinforcement

[1-3]. Along with, organic and in organic additives also consequently utilized with different geometries. These additives could be different forms and molecular structures customarily; reinforcement of fibers increases the load carrying capacity of the laminate in impressive way by maintaining notable way of wear resistance. Usually, metals, metal powders and polymeric materials have been used as the reinforcement to increase the wear resistance of the composite. Conversely, these materials have low cost, fast production rate, lightness, design flexibility and non-corrosive nature; it is very extensively used as the particulate reinforcement in most of the industrial applications [4-6]. Moreover, polymer based tribo-materials are very widely accepted as the better replacement for heavy metal structures like sliding elements, bushes, bed ways and cages of bearings. In line of the above, nanoparticles have emerged as the best filler materials over the past two decades since it gives better filling effect and other benefits in engineering design. Further, usage of these nano materials in matrix compound reduces the porosity in turn increases the moisture absorption resistance and shows high thermal stability [7]. Additionally, nano fillers as well shows better synergistic effect with other constitute materials while forming the hybrid composites; also it arrests the crack propagation effectively than the other filler materials. Researchers gradually started to compare the effectiveness of various micro and nano particles in loading as the filler material in different matrix materials. Among the results, the nano particles indicated the effective specific wear rate (Ks) than the inclusion of micro particles into the composites. In recent years, trending of inclusion of various nano materials such as SiO_2, Al_2O_3, ZnO_2, nanoclay, carbon nano tubes, graphite and TiO_2 have been taken place in various research portfolios to obtain the better wear resistance into the composites [8]. Sunscreens typically contain titanium dioxide (TiO2) nanoparticles to block UV rays. Nanoscale TiO2, that cannot reflect optical light but absorbs UV light due in part to the particles' small size, creates a transparent barrier that protects the skin from damaging solar rays. The most significant white color pigment used in the polymer sector is titanium dioxide. It is frequently utilised because, when included into a plastic product, it effectively scatters light waves, adding whiteness, brightness, and opacity.

Bazrgari et al. prepared the Nano composite with various proportionate of filler materials like 1% and 3% specimens and subjected the same for wear test, from the result, it was seen that, the 1% inclusion of nano Al_2O_3 particle shows greater wear resistance than the other combinations [9]. Chuang et al. fabricated the silane treated $nSiO_2$ loaded cyanate ester resin laminate, the results revealed that, wear resistance of the laminate kept increases upon loading of the particulates, and coefficient of friction (COF) shown lesser value, this was because of the uniform dispersion of particulate and better interface adhesion between the fillers and matrix. Furthermore, surface modifying technique also unveils better compatibility between the fiber and other constituents in the hybrid composites [10]. In modern industries, it is well-known fact that, friction and wear causes the maximum losses to all kind of functional parts, thus the way, induces maximum loss in all segment of industries. In order to optimize the various constituents of the laminates during fabrication, the critical parameters such as AL (Applied load), SS (Sliding speed), temperature, COF and wear have to be taken in to account profusely. Ramesha and Suresha, both of them in their research used the Taguchi design and ANOVA, in order to describe how filler loading and load impacts on the abrasion. Also they have analyzed the relationship between the abrasion wear and sliding velocity. Similarly optimization of 3BAW parameters like load, abrading gap and filler percentage of HNT was discussed by Muralidhara et al. by using the L16 array. Almost most of the researchers doing study in tribology have been investigating the effect of particulates on the tribological behavior of epoxy/vinyl ester based composites [11-12]. Taguchi method was considered as one of the most simple, systematic and effective methods to do optimization in most of the industrial problems as it remarkably reduce the tests, cost and time than the traditionally following methods [13].

By giving the standard variables into the Taguchi method, will be able to give the standard orthogonal array (O.A) with all necessary operating levels to do the experimental analyze and interaction. Consecutively, rows and columns in an orthogonal array clearly specify the maximum number of combinations as a test to be performed. As well, experimental results are arrived by analysis of variance to know the influence of each parameters response [14]. In this particular study, the jute fiber has been chosen as the basic reinforcement material. The main application for jute fibre is in the production of fabrics for the packaging of a variety of agricultural and industrial goods that demand for bags, sacks, packs, and wrappings. Jute is frequently employed because of its inexpensive cost whenever bulky, strong fabrics and twines resistance to straining are required. Furthermore, jute fiber is utilized in a variety of industries, such as textiles, vehicles, and even in certain load-bearing purposes. Along with that, various proportionate of titanium oxide (TiO2) like 0, 3 and 5% particulate is loaded into the matrix. The commonly available epoxy and polyurethane (70:30) have been selected as the base matrix material for this entire study as some weight percentage of amount in order to extract specific quality of the individual constituent [15-16]. A blend of various polymers in converged network, at least one of which is synthesised or crosslinked in the immediate proximity of the other, is known as an interpenetrating polymer network, or IPN (s). Two characteristics set an IPN apart from polymer blends, blocks, or grafts: (1) an IPN swells in solvent yet does not disintegrate in them; and (2) creep and movement are controlled. The tribological properties of the various proportionate of IPN composites were investigated for different AL, MT and SS for a constant speed of 1200 m at room temperature to avail the CoF and SWR. Taguchi L27 orthogonal array was chosen for conducting the entire analysis. The found experimental values are thoroughly analyzed with Taguchi method and their successive optimal process parameter like combination was determined in order to minimize the response values. Along with that, confirmation test also done to verify the quality characteristics for better improvement. However, impact of design parameters and their interactions like SWR and CoF of the IPN composites are also found by using the analysis of variance (ANOVA). Apart from that, to wear surface evaluation was also done with help of the scanning electron microscope to better understand the micro mechanism between the constituents[17].

2. EXPERIMENTAL

2.1 Materials

Titanium dioxide nano powder was utilized for the fabrication processes to make the particulate reinforced IPN composite, as such received from supplier which also used for the synthesis purpose. Due to its numerous distinctive properties, titanium dioxide is perfectly suited for a wide range of applications. It naturally exists as a solid and has extraordinarily high melting and boiling points of 1856°C and 2863°C, respectively. Regardless of its particle form, it is indistinguishable from water. Highly crystalline TiO_2 nanoparticles with a diameter of 5 nm were produced using a hydrothermal technique as high refractive index filler. To synthesize the nano powder, the necessary amount of solvent (heptane and propoxide) was taken. Finally to synthesize the nano particle, the particulate was mixed with the solvent and stirred thoroughly for the period of 2 hours. At last, the required amount of distilled water was gradually added to the mixture; finally it is kept at room temperature for the period of 70 hours. The formed mixture was filtered with normally available paper (filter). Ultra sonication procedure was used to uniformly disperse the TiO_2 particulate at ambient temperature for the period of 30 minutes. The necessary amount of polymers like epoxy and polyurethane (70:30) was taken and mixed thoroughly and kept separately with their respective hardeners. The respective hardeners also added along with the base matrix for curing purpose as shown in Figure 1 [18].

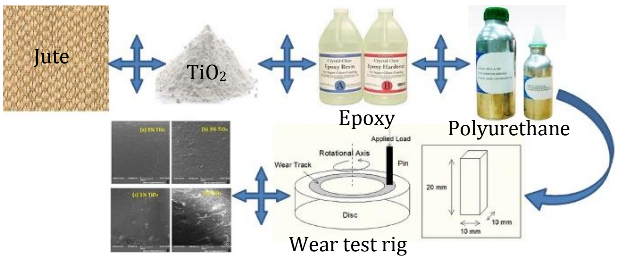


Fig. 1. Schematic representation of fabrication of IPN composite.

2.2 IPN laminate preparation

The exact proportionate of titanium dioxie about 0%, 3% and 5% were taken initially. After which, the required amount of interpenetrating polymer blend (IPN) was taken and both the particulate and blend was mixed thoroughly. Secondly, the already cut jute fiber was placed over the flattened surface, jute fiber was wet with blend and particulate combination, this sequence of procedure was continued till the five layers of fiber stacking [19].

Table 1. IPN composites code and concentrations of different constituents of composites.

IPN composite	Fiber wt.%	Matrix wt.%	Particulate wt.%
IPN0	60	40	0
IPN3	60	40	3
IPN5	60	40	5

3. MATERIAL CHARACTERIZATION

3.1 Wear test

The entire wear test was followed as per the ASTM G99 standard, in which pin on disc wear test rig was used. The Figure 2. Shows the schematic view of pin on disc and the rotating counter face. Also, the test specimen glued into the cylindrical pin to do the experimental processes [20].

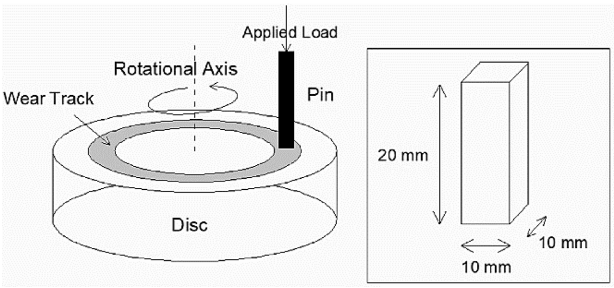


Fig. 2. Pin on disc wear test rig.

The test specimens were thoroughly cleaned with acetone in order to remove the unwanted foreign micro matters from the surface. Once the test was completed, the cotton cloth was used to remove the debris from the worn out surface. Before start of the experiment and after the end of the experiment, the samples wear volume was calculated with help of the equation 1 [21].

$$Wv = (Wi - Wf)/\rho c \quad (1)$$

Where, Wv – wear volume (m3), Wi – Initial weight of the specimen (g), Wf – final weight of the specimen (g) and ρc – Density of the composite (g/m^3).

Once the wear volume was found, by using the equation 2, Ks of the IPN composite was found.

$$Ks = Wv / L \times D \quad (2)$$

Where, Ks – Specific wear rate, Wv – wear volume (m3), L – load (N), and D – Sliding distance (m).

All set of experiments were performed on the IPN composite by using the Taguchi method, in order to minimize or avoid the time duration to get the optimum wear conditions. The common factors like sliding velocity, filler loading and applied load normally would influence on Ks and COF at ambient temperature, as well as these factors are taken for present research of study with the sliding distance of 3000 m for all set of experimental runs. The control factors of various levels (FOUR) are taken into account at different levels as shown in the Table 2 [22].

Table 2. Wear analysis factors with various levels.

Control factors	Unit	Levels			
		1	2	3	4
Particulate content (A)	Wt.%	0	3	5	7
Sliding Speed (B)	m/s	0.85	1.6	2.35	4
Applied Load (C)	N	20	30	40	50

Taguchi is believed as one of the best tool to implement the design of experiments for conducting the various trails in very effective way by setting test conditions. Also, it is extensively considered as the powerful statistical tool to optimize the design parameters and their levels. In this study, L16 orthogonal array was used to evaluate the impact of control factors and levels towards the responses such as COF and Ks. The noted responses out of this study will be converted to signal to noise ratio (SNR) to find out the optimum combination of levels and control factors. The main intention behind that of the entire study was, to find the combination of control factors and levels which in turn reduce the Ks and COF, as well small value is better category than remaining combinations [23-24].

3.2 Scanning electron microscopic analysis

After the specimens were subjected for wear test, the worn surfaces were thoroughly investigated with help of the scanning electron microscope (JEOL JSM-6480LV). Before start the investigation, the entire worn surfaces were coated with thin gold film to expedite the conductive environment prior to observation.

4. RESULTS AND DISCUSSIONS

4.1 Wear analysis

The experiments were conducted based on L16 orthogonal array, in which three factors likely Filler loading (A), Sliding velocity (B), and applied load (C) and their various proportionate of different levels were considered. Upon this experimental study, specific wear rate was calculated by using the equations 1 & 2. Similarly COF can be calculated by using friction load and normal load. The obtained COF and Ks were entered in the Table 3(a) & (b) which was derived from Taguchi (Minitab V-18) methodology to avail the SNR.

Table 3(a). Experimental results and their SNR.

Expt. Runs	A	B	C	Ks ($x10^{-13}$) (m^3/Nm)
1	0	0.85	20	0.126
2	0	1.6	30	0.138
3	0	2.35	40	0.149
4	0	4	50	0.162
5	1	0.85	30	0.131
6	1	1.6	20	0.132
7	1	2.35	50	0.148
8	1	4	40	0.154
9	3	0.85	40	0.125
10	3	1.6	50	0.133
11	3	2.35	20	0.128
12	3	4	30	0.141
13	5	0.85	50	0.112
14	5	1.6	40	0.114
15	5	2.35	30	0.117
16	5	4	20	0.123

Table 3(b). Experimental results and their SNR.

Expt. Runs	Ks – SNR	COF	COF - SNR
1	19.8971	0.323	11.2559
2	18.9348	0.399	9.3345
3	18.3034	0.478	7.7434
4	17.5305	0.482	7.2895
5	19.5275	0.354	10.3852
6	19.4554	0.345	10.6362
7	18.3767	0.468	7.9128
8	17.9844	0.462	7.8276
9	19.9728	0.268	12.8451
10	19.3739	0.382	9.7236
11	19.7474	0.326	11.1449
12	18.8322	0.361	10.2297
13	20.8247	0.232	14.2233
14	20.8375	0.247	13.6529
15	20.5848	0.246	13.6529
16	20.1267	0.284	12.3878

The Table 3 encapsulates the obtained Ks in term of ($x10^{-13}$, m^3/Nm) and their COF through conducting the experiments along with SNR [25].

The response parameters of all control factors and their levels were quantified by using SNR on account to find the Ks and COF and their successive values were presented in Tables 4 and 5 respectively. From the graphs, it was found that the best wear resistance and their consecutive COF were found at greater S/N ratios in the response graphs. In the response graphs, it was clearly found that factor permutation of sliding velocity, filler loading and load had given minimum Ks and COF. According to the obtained values, minimum COF and Ks were seen when the filler loading was in highest level, also sliding velocity and applied load found to be low. Sudhir et al also found that, minimum Ks value possible while increasing the filler loading correspondingly reducing the sliding velocity and applied load [26].

Table 4. Response table of SNR upon Ks.

Levels	A	B	C
1	18.69	19.98	19.81
2	18.84	19.68	19.58
3	19.48	19.26	19.28
4	20.65	18.62	18.96
Delta	2.04	1.59	0.84
Rank	1	2	3

Table 5. Response table of SNR upon COF.

Levels	A	B	C
1	8.886	12.182	11.356
2	9.245	10.837	10.815
3	10.876	10.113	10.567
4	13.479	9.484	9.787
Delta	5.684	3.788	2.678
Rank	1	2	3

The Figure 3 and 4 represents the main effect plot of SNR for Ks and COF respectively. It was found that, from the graphs, higher the SNR value gives the lower rate of variance. Also the graph shows the values of variance with respect to different levels of SNR. This part further helps in determining the best combination of experimental procedures to get the optimum Ks. While doing so, to get the best combinations of control factors of levels to arrive the Ks was seen as 5 wt.% of filler content, 0.85 m/s of sliding speed and 50 N of applied load. From the entire experimental procedure exhibit that, incremental in filler loading value successively reduces the Ks abruptly. In the case of COF, found optimized combination was 5 wt. % filler loading, 4 m/s of sliding velocity and 20 N loads, was smaller value than the remaining arrived values. Similarly, Tables 6 and 7 were briefing the analysis of variance exhibited results upon Ks and COF of the IPN composites for the foresaid control factors in the experiments [27].

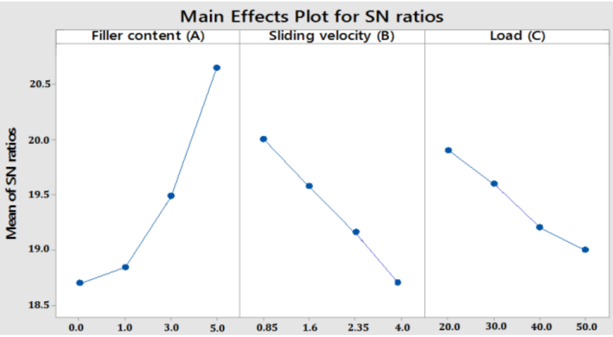


Fig. 3. Main effect plot for SN ratio towards Ks.

Fig. 4. Main effect plot for SN ratio towards COF.

From the Table 6, it was seen that, contribution of particulate loading, sliding velocity and load for Ks was 58.64%, 32.91% and 9.42% which influences on the experimental values of Ks. Similarly, from the Table 7, impact of particulate loading, sliding velocity and load for COF was 64.65%, 23.35% and 12.21%. From the obtained values it was clearly observed that, filler loading plays the vital role in increasing the wear resistance as well decrease in COF. The particulates have evenly distributed across the matrix and fiber and thus the way creates the better rubbing surface against the wear and load. The fillers (TiO2) predominantly work as the wear protector for the composite. Also observed that, the interpenetrating polymer networks provides the better dissipation for the fillers across matrix and gives the good interfacial adhesion [28]. Furthermore, from the experimental analysis shows that, small increase in the applied load causes the decreasing trend of wear resistance, similarly the sliding velocity also shows the same kind of trend set, small incremental value in the sliding velocity significantly reduces the wear resistance and DOF. Actually, increase in the applied load adversely affects the matrix medium which in turn induces the heat thus the way matrix softens itself due to temperature rise. So the bonding or interfacial adhesion between the particulate matrix loosens and correspondingly rapid wear observed across the surface of the IPN composite. The same trends of observation were also found by many researchers in their experimental analysis by changing the various filler materials and changing successive load upon many laminates.

Table 6. ANOVA responses for Ks.

Source	DF	Adj SS	Adj MS	F-Value	P-Value	P (%)
Regression	3	0.003213	0.001145	287.83	0.0000	-
A	1	0.001908	0.001918	528.18	0.0000	58.64
B	1	0.001142	0.001121	297.78	0.0000	32.91
C	1	0.000284	0.000284	85.28	0.0000	9.42
Error	12	0.000043	0.000003	-	-	1.34
Total	15	0.003245	-	-	-	-
S =0.0019768 R-sq = 97.76% R-sq (pred) = 96.95%						

Table 7. ANOVA responses for COF.

Source	DF	Adj SS	Adj MS	F-Value	P-Value	P (%)
Regression	3	0.123414	0.038512	123.94	0.0000	-
A	1	0.074842	0.074762	252.72	0.0000	64.65
B	1	0.026957	0.026957	85.79	0.0000	23.35
C	1	0.013814	0.013814	43.38	0.0000	12.21
Error	12	0.003774	0.000315	-	-	3.41
Total	15	0.125276	-	-	-	-
S =0.0185746 R-sq = 96.55% R-sq (pred) = 95.35%						

Likewise, Gnanamoorthy and ray also pointed out three types of wear mechanisms and their allied problems in their study part, majorly noted (i) matrix wear, (ii) fiber and filler wear (iii) fiber and matrix debonding throughout their study, this three mechanisms have had played majorly in disturbing the entire laminates irrespective of particulate addition. Despite the titanium dioxide is bit harder (spherical in shape), improves the tribological properties of IPN laminate, the interfacial force between the filler and matrix lessens due to the thermal softening of the matrix. Though researches say various predictions upon the failure of the composite, the common mechanism towards wear was proposed by all the investigators that, surface breaking, particulate separation, thick and thin transfer layers across the surface induces the wear on the IPN laminate [29].

The regression expression was also formulated to identify the relation upon various input parameters and output responses, which was given as the equation of (3) and (4).

$$Ks = 0.11758 - 0.008391A + 0.009516B = 0.000264C \quad (3)$$

$$COF = 0.2755 - 0.06815A + 0.04684B + 0.001783C \quad (4)$$

The obtained mathematical expressions were verified by using normal probability plots for the responses of Ks and COF, their graphical representation also shows in the Figure 5 and 6.

From the graph, it was observed that the residual were distributed all along the line and the so called expressions were found satisfactory. Based on the experimental analysis observed, because of hybrid lamination, laminates proves the reinforcement effect, through by giving better wear resistance and shown lesser value of COF [30]. As well, to substantiate the conclusions arrived from Taguchi technique, confirmation test was performed which was based on SNR plots. The taken values were 5 wt.% of particulate, 0.85 m/s sliding velocity and 50 N of applied load and their corresponding results and development were mentioned in the Table 8 and reduction in SWR was found by confirmation test standards.

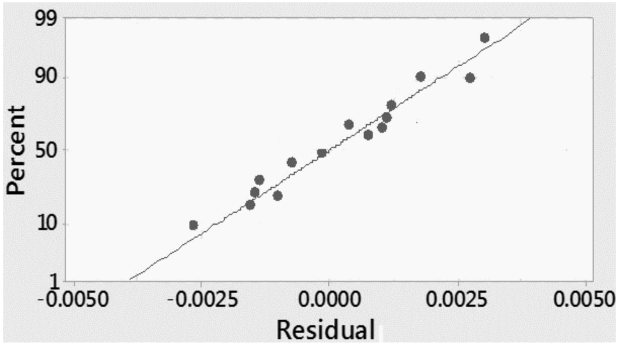


Fig. 5. Normal probability graph for Ks.

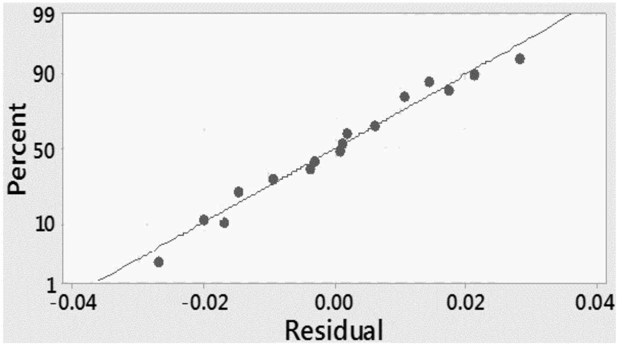


Fig. 6. Normal probability graph for COF.

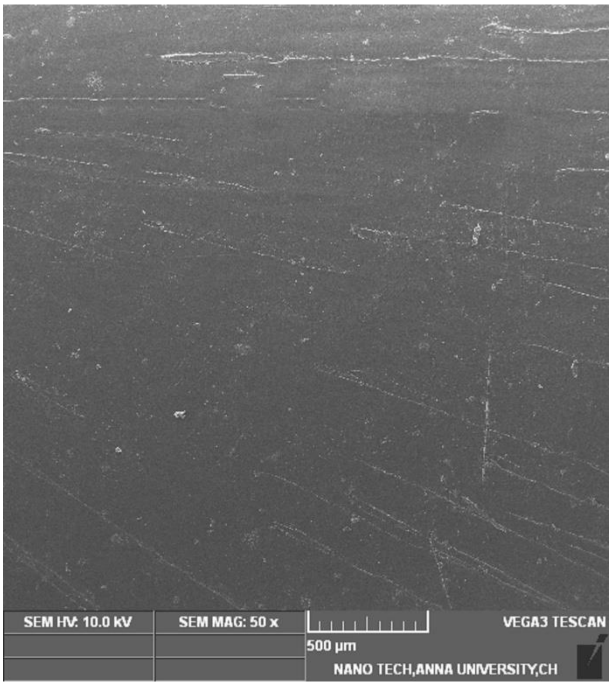
Table 8. Confirmation test.

	Primary test based on L16 array	Confirmation test results	Improvement in the results
Level	A4B1C4	A4B1C1	
SWR	0.112	0.087	22.32 %
COF	0.232	0.193	16.81 %

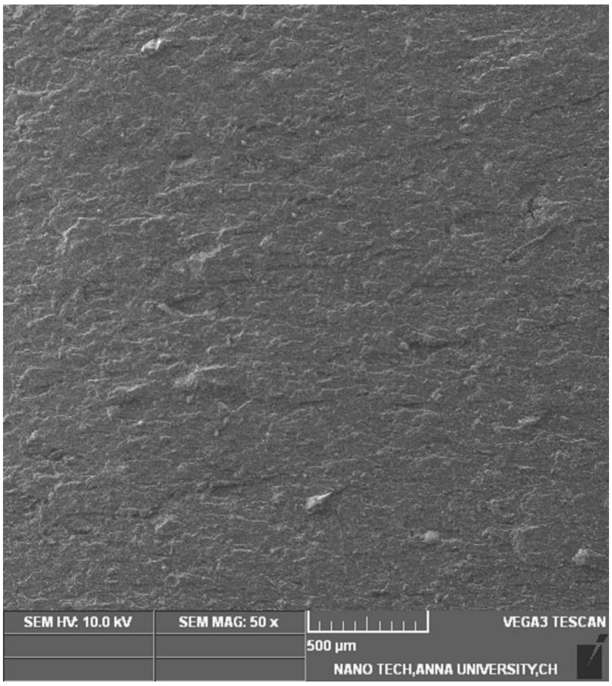
Generally epoxy resin is considered as the brittleness (hard segment) in nature, because its unique nature of brittleness, it comes under the rapid wear once slide against the rough steel counterpart. Similarly the polyurethane is having the presence of soft segment (SS), in spite of the soft segment presence it exhibits good interfacial strength with the particulates. But, soft segment presence forces the entire matrix comes across thermal stress in turn, the matrix plasticizes early and losses the interfacial adhesion between the particulate and fiber. In order to overcome the negative issues encountered, the IPNs are reinforced with the natural fiber along the particulate addition. Natural fiber gives the strength, stiffness and rigidity to the laminate and particulate exhibits the better wear resistance against the sliding velocity and load with respect to the particulate addition [31].

4.2 SEM analysis

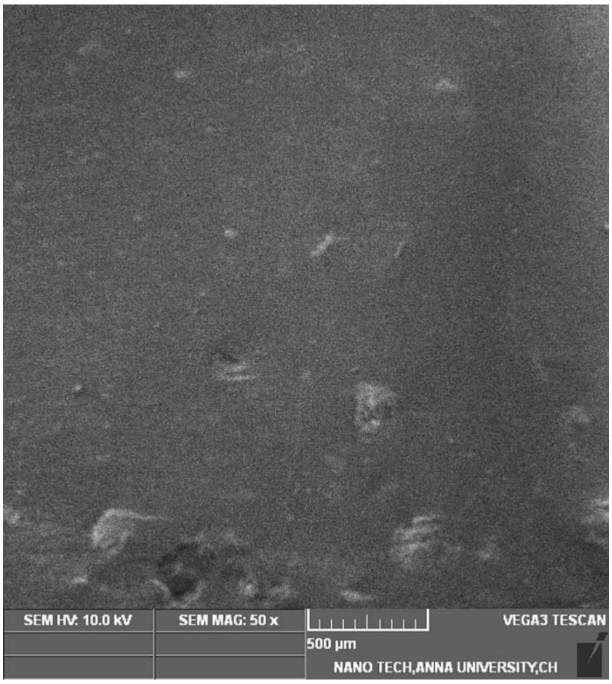
Figure 7(a) exhibits the distinctive worn surface of jute fiber reinforced particulate composite under 5 wt.% particulate loading, 085 m/s sliding velocity and 50 N load. The image shows considerable smooth surface. As well, it was noted that, the fiber were completed secured with matrix, there was no evidence of fiber stripping-off on the surface of the worn out surface. It shows that, the IPNs are well accustomed with the particulates filling and unveils better interfacial adhesion between the substitutes. As well it was proved that, the particulates were well dispersed into the matrix and gives the better wear resistance to the specimen. The Fractographic wear image of Figure 7(c) shows the 1 wt.% particulate loaded worn out surface with 0.85 m/s sliding velocity and 30 N loaded experimental procedures. The images showcase the shear cups and matrix damages on the image captured.



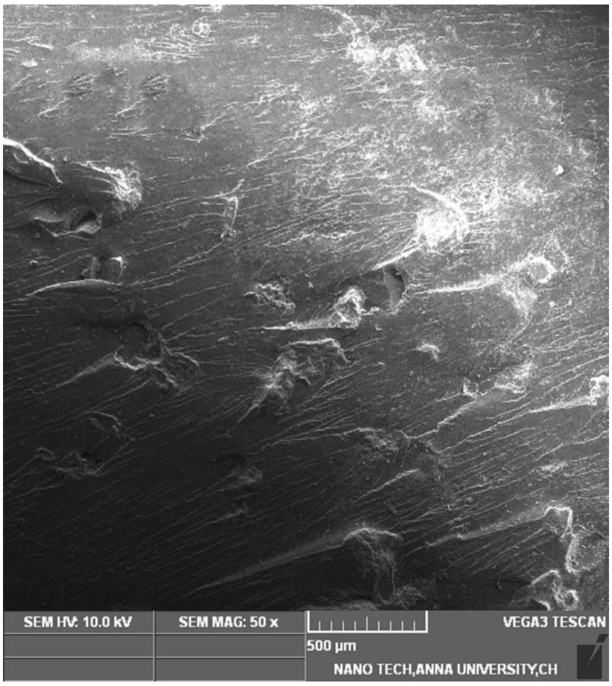
(a) 5% TiO_2



(b) 3% TiO_2



(c) 1% TiO_2



(d) Neat sample

Fig. 7. Scanning electron microscope images (a) 5% TiO_2 (b) 3%TiO_2 (c) 1% TiO_2 (d) Neat Sample (Pristine).

Even though, the particulates were well disbursed with the matrix it could not give that much amount of wear resistance as compared with the remaining higher combinations like 3% and 5% particulate addition. Figure 7(b) shows the 3 wt.% particulate loaded worn out surface, which subjected with the 0.85 m/s sliding velocity and 40 N load. Their corresponding image show the fiber thinning, fracture of fiber, wear scars were noted down, this effect shows the moderate wear behaviour on the surface of the IPN laminate [32]. Since less amount of particulate disbursed into the matrix, the matrix surface alone come across the direct wear as it could be seen as the cavity image on the surface of the image. Since the specimens were incorporated with only less amount of particulate addition, it will not able give much wear resistance against the load, sliding velocity as well it quickly come across the massive wear than the higher particulate added specimens.

The Figure 7(d) shows the image of 0 wt.% particulate loaded specimen, in that image, findings of massive wear, fiber scissoring and fiber breakages were commonly seen across the worn out surface. Since matrix had the direct contact with the metallic surface, it could not give complete cover to the fiber as it plasticizes due to the thermal stresses, due to this phenomenal effect, large amount of fiber breakage and matrix damages were commonly found across the entire surface. From the images, it was concluded that, as much as addition of particulates into the matrix gives the complete wear resistance to the specimens. The interpenetrating polymer network also gives the complete binding energy to fiber and particulates [33].

5. CONCLUSIONS

The influences of titanium dioxide on dry sliding wear characteristics by using Taguchi methods were investigated. Based on the present investigation results, conclusions have been drafted as follows:

- The jute fibers have been successfully reinforced with epoxy and polyurethane matrix (interpenetrating polymer networks) material along with the various proportionate of titanium dioxide.
- By adopting the Taguchi design of experiments different optimized combinations for the lower value of Ks and their corresponding COF were noted. While doing the experimental analysis on both the output parameters (Ks & COF) identification have been done on control factor and levels. The arrived optimized levels were 5 wt.% particulate loading, 0.85 m/s sliding velocity and 50 N applied load.
- From the formation of ANOVA, it has been observed that the major influence in curbing the Ks and COF was seen as particulate loading.
- Moreover, it has been found that titanium dioxide plays the major role in improving the wear (tribological) performance of jute fiber reinforced IPN composites. The worn out surfaces of the formed composite shows, particulate inclusion into the matrix enhances the robust improvement in turn creates possible wear resistance with respect of particulate addition. Also found that, the upon the inclusion of particulates, matrix shields the worn surface form severe wear mechanisms.
- The assessed values calculation was done, by using the optimum test parameters for SWR and COF, upon doing this good agreement was seen between the predicted and actual values with confidence interval of 95%.

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