

Mechanical And Three Body Abrasive Wear Behaviour of Nano-Filler Filled, Chopped Glass Fiber Filled Hybrid Composites

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ABSTRACT

Polymer matrix composites are developed for tribological applications due to the possibility of optimizing the properties with the addition of special filler materials as reinforcement. Filler filled polymer matrix composite systems are used in many automotive parts and components like brake pads or clutches which require high coefficient of friction coupled with low wear. However, in most cases it is the primary concern to develop polymer composites that possess low friction and low wear properties under dry sliding conditions against smooth metallic counterparts e.g., gears or bearings, chute liners and coal handling equipments in power plants, gear pumps handling industrial fluids, agricultural machine components. Polymer composites operate in applications where fluid and grease lubricants fail, and have superior tribological performance to traditional polymer composites. Particulate fillers or fibres or solid lubricants have been a part of notable reductions in the wear rate of the polymer matrix at very low loading, and there exists a wide scope on the development of polymer composites on the tribological behaviour. The incorporation of fillers in polymer could provide a synergism in terms of improving mechanical properties and wear performance, which has not been adequately explored so far. The research on nano fillers such as nano clay, Glass fiber and Titanium di-oxide (TiO₂) in epoxy system is scarcely reported and further they focus on a few specific issues only. The present study was focused on investigating Mechanical - Tensile, Hardness, and three body abrasive wear characteristics of thermosetting based Epoxy composites and the effect of incorporation of nano clay, TiO₂ nanoparticles and chopped glass fiber on microstructure, hardness, friction and abrasive wear are determined and analyzed. The main aim was to investigate the effect of applied load, sliding distance /abrading distance, sliding speed and percentage of filler. The surface morphology of the worn out surfaces were examined using SEM, to get a better understanding of the wear mechanisms.

Keywords : Abrasive Wear, Nano Composites, Epoxy, Nano Clay, Nano Tio₂, Glass Fibres

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I. INTRODUCTION

Hybrid Polymer matrix composites (PMCs) are extensively used in industrial applications and other engineering applications due to their special characteristics such as high specific strength and modulus. Properties of composites are strongly dependent on the properties of their constituent materials, their distribution and the interaction among them. The composite properties may be the volume fraction sum of the properties of the constituents or the constituents may interact in a synergistic way resulting in improved or better properties. Apart from the nature of the constituent materials, the geometry of the reinforcement (shape, size and distribution) influences the properties of the composite to a great extent.

Epoxy resin has good resistance to environmental degradation low shrinkage during cure, high adhesive strength and high mechanical properties, high electrical insulation and good chemical and water resistance. The epoxy resins have low impact resistance and toughness results in poor sliding wear behaviour. The use of reinforcements in the resin may provide increasing load withstanding capability, moderated coefficients of friction, improved wear resistance, better thermal properties and increased mechanical strength [1].

Glass fibres have good mechanical, thermal and tribological properties and the principal advantages of glass fibres are low cost with high strength. One of the challenges in the use of glass fibre-reinforced polymer (GRP) composites for primary structures is to avoid the damage propagation during conditions involving abrasive wear [2].

The main advantage of using nano fillers, compared to conventionally used micro-scaled fillers, is the significant smaller amount of filler needed to achieve or exceed a certain multifunctional property profile.

Due to the various possibilities for influencing material properties (e.g., mechanical and rheological properties, flame retardancy), layered silicates filled polymers could be employed in different industrial applications (e.g., automotive and construction area). The processing techniques used for the fabrication of nano composites is simple and the desired results can be obtained by maintaining proper process parameters.

One of the popular reinforcing fillers among various nanoparticles are nanoclay or nano-layered silicate minerals which provide several enhancement properties such as high aspect ratio, potentially exfoliation characteristics and better mechanical performance. It has an excellent aspect ratio and specific surface area. Many researchers found promising that nanoclay filler enables to strengthen and alter the mechanical properties of fibre reinforced polymer when nanoclay was well dispersed in epoxy resin composites [3].

Titanium dioxide (TiO_2) is used as a filler material by many researchers due to its potential applications across many different areas due to its good photocatalytic properties, hence it is used in antiseptic and antibacterial compositions, degrading organic contaminants and germs, as a UV-resistant material; this is due to its chemical inertness properties, non-toxicity, low cost, high refractive index, and other advantageous surface properties. The incorporation of TiO_2 nanoparticles into different types of the polymeric matrix could produce synergistic effects and various researchers have studied the effect of TiO_2 nanoparticle on several properties of polymeric composite, mainly to figure out whether the application of nanoparticles can enhance the mechanical performance of polymeric composites for applications in various fields.

Wear is defined as damage to a solid surface generally involving progressive loss of material due to relative motion between that surface and contacting substance

or substances. Main types of wears are abrasive wear, adhesive wear, corrosive wear, erosion wear and fatigue wear, which are commonly observed in practical situation. When material is removed by contact with hard particles, abrasive wear occurs. The particles either may be present at the surface of a second material (two body wear) or may exist as loose particles between two surfaces (three-body wear). In three body abrasion, the loose abrasive particles abrade the solid surfaces between which they are situated only about 10% of the time, remaining 90% of the time in rolling. Most of the abrasive wear problems which arise in industrial equipment are three body, while two body abrasion is encountered primarily in material removal operation. The rate of material removal in three body abrasions is lower than that of two body abrasion [4]

Different types of polymer show different friction and wear behaviour. However, neat polymer is very rarely used as bearing materials and wear-resistant materials because unmodified polymer could not satisfy the demands arising from the situations wherein a combination of good mechanical and tribological properties is required [5]. Among the wear types, abrasive wear situation encountered in vanes and gears, in pumps handling industrial fluids, sewage and abrasive-contaminated water, roll neck bearings in steel mills subjected to heat, shock loading; chute liners abraded by coke, coal and mineral ores; bushes and seals in agricultural and mining equipment, have received increasing attention [6].

The design of a structural component using composites involves both material and structural design. Composite properties (e.g. stiffness, thermal expansion etc.) can be varied continuously over a broad range of values under the control of the designer. Careful selection of reinforcement type enables finished product characteristics to be tailored to almost any specific engineering requirement. Whilst the use of composites will be a clear choice in many instances,

material selection in others will depend on factors such as working lifetime requirements, number of items to be produced (run length), complexity of product shape, possible savings in assembly costs and on the experience & skills the designer in tapping the optimum potential of composites. In some instances, best results may be achieved through the use of composites in conjunction with traditional materials.

In the present study, the abrasive wear behaviour of epoxy composites reinforced by chopped glass fibres and variable percentages of nano clay and nano TiO₂ were investigated. The main objective was to examine the effect of applied load, sliding distance /abrading distance, sliding speed and percentage of filler.

II. MATERIALS AND METHODS

Materials: The matrix material used was medium viscosity epoxy resin (Lapox L12) and room temperature curing polyamine hardener (K-6) and was chosen because it provides good resistance to alkalis and good adhesive properties. The fibre reinforcement material employed was chopped glass fibres of 14µm filament diameter and 6-24mm length. The fillers - nano clay and nano TiO₂, were procured from Sigma Aldrich, Bangalore. The fillers were tested using X-ray diffraction method to measure the particle size and the maximum particle size of nano clay was 59nm and of nano TiO₂ was 39nm. The details of the material combination and percentage of filler materials are given in Table 1.

Table 1: Details of samples prepared

Sl. No.	PERCENTAGE OF FILLERS
1	Epoxy+0%Fillers+0%GF
2	Epoxy+2% Fillers+2% GF
3	Epoxy+4% Fillers+4% GF
4	Epoxy+6% Fillers+6% GF
5	Epoxy+8% Fillers+8% GF
6	Epoxy+10% Fillers+10%GF

Fabrication Methods: The composite laminates were fabricated using wooden moulds and a hand lay-up technique was adopted. To prepare the filled chopped glass fibre-epoxy composites, nano clay and nano TiO₂ fillers were mixed with a known amount of epoxy resin. The laminate was cured at ambient conditions for a period of about 24 hours. The cured materials are cut to yield abrasive wear test specimens of size 75mm X 25mm X 3mm and tensile test specimens of size 150mm X 25mm.

Testing Methods: The mechanical properties - tensile strength were evaluated by testing three samples for each combination of the composites, in accordance with ASTM D-638 using a Universal testing machine of upto 100kN capacity. The Rockwell hardness tester was used to measure the hardness of the filled and unfilled hybrid epoxy composites.

The three body abrasive wear test was conducted using the rubber wheel abrasion test rig with angular silica sand used as the abrasive material. The schematic representation of the dry sand/rubber wheel abrasion test rig is shown in Fig. 1. The composite test samples were cleaned using acetone and dried before the test and was weighed using a high precision digital balance to note the initial weight of the test samples. The specimens were fixed to the specimen holder and a known amount of weight was added on the lever to have a firm grip between the specimen and the rubber wheel. The pivot axis of the lever arm lies within a plane, which is approximately tangent to the rubber wheel surface and normal to the horizontal diameter along which the load is applied.

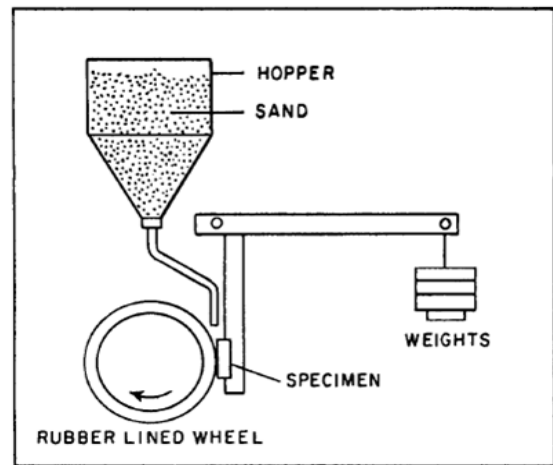


Figure 1: Schematic representation of dry sand/rubber wheel abrasion test rig.

The rubber lined to the wheel was chlorobutyl rubber tyre (hardness: 58–62, Durometer-A) and the diameter was 338.9mm. The tests were conducted at a rotational speed of 200 rpm for a duration of 300 seconds, load applied was 22N and rate of feeding the silica sand abrasive was 360±5g/min. The abrasives were introduced between the test sample specimen and rotating abrasive wheel, by maintaining a firm grip between the two by means of the lever arm. The rotation of the rubber wheel is in the direction of the sand flow. After the test duration, the test specimen is removed and cleaned, weighed again to note the final weight of the test samples. The difference in weights of the test specimens gives the weight loss of the specimen. Three tests for each combination of the composites were tested and the average values were reported. The wear was measured by the loss in weight, which was then converted into wear volume using the measured density data. The specific wear rate (K_s) (ASTM G-65) was calculated from the equation:

$$K_s = \frac{V}{L \times D} \text{ m}^3/\text{Nm}$$

where 'V' is the volume loss in m³; 'L' is the load in Newtons, and 'D' is the sliding distance in meters.

The worn surface morphology was studied using Scanning Electron Microscopy. The surface of the worn out specimens were gold sputtered prior to microscopy.

III. RESULTS AND DISCUSSION

Mechanical Properties: The tensile strength (MPa) of the nano clay and nano TiO₂, chopped glass fibre filled epoxy composites are reported in Table 2.

Table 2: Tensile Strength in MPa

Sl. No.	Specimen	Clay	TiO ₂
1	Epoxy+0%Fillers+0%GF	12.99	12.99
2	Epoxy+2% Fillers+2% GF	15.7	14.49
3	Epoxy+4% Fillers+4% GF	15.88	15.32
4	Epoxy+6% Fillers+6% GF	17.36	17.91
5	Epoxy+8% Fillers+8% GF	16.39	15.1
6	Epoxy+10% Fillers+10%GF	14.93	13.36

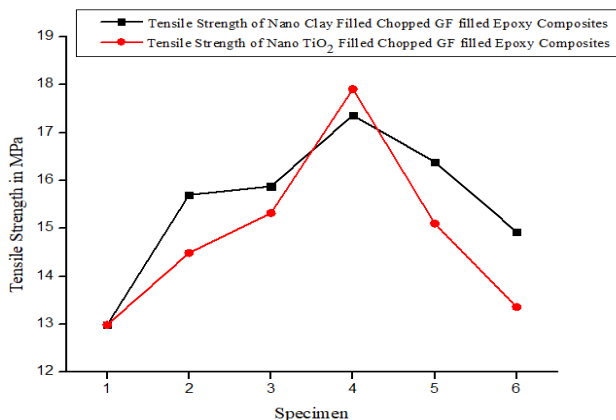


Figure 2: Variation of Tensile Strength (MPa) for different combinations of composites

The variation of the tensile strength for the different combinations of the epoxy composites filled with nano clay and nano TiO₂, chopped glass fibre are shown in Fig.2. The chopped glass fabric reinforcement in epoxy matrix increases the tensile strength. Further, addition of nano clay and nano TiO₂ fillers in chopped G-E composite enhances the tensile strength. As expected the chopped glass fabric reinforcement and inclusion of fillers lower the elongation at fracture indicating the

brittleness in composite materials. The results indicate that there is about 33% increase in the tensile strength of the epoxy matrix when the filler concentration is 6% clay and 38% increase when the filler concentration is 6% TiO₂. The tensile strength decreases as the filler concentration increases owing to agglomeration, improper distribution of fillers and also the fabrication methods adopted that plays a vital role in optimising the properties of the composites.

The surface hardness of the nano clay and nano TiO₂, chopped glass fibre filled epoxy composites are reported in Table 3.

Table 3: Rockwell Hardness Number of Chopped Glass fibre reinforced epoxy with nano clay and nano TiO₂ fillers

Sl. No.	Specimen	Clay	TiO ₂
1	Epoxy+0%Fillers+0%GF	47	47
2	Epoxy+2% Fillers+2% GF	51	49
3	Epoxy+4% Fillers+4% GF	52	50
4	Epoxy+6% Fillers+6% GF	55	58
5	Epoxy+8% Fillers+8% GF	50	49
6	Epoxy+10% Fillers+10%GF	49	46

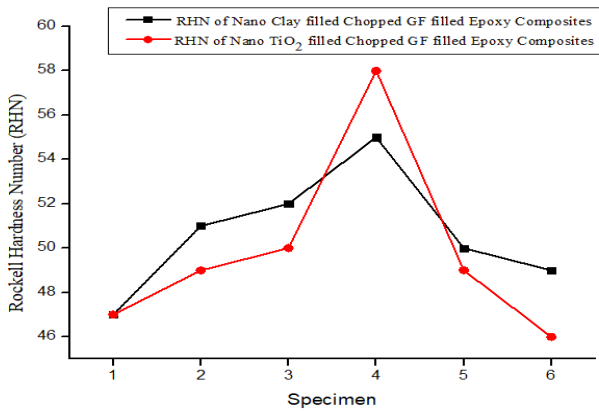


Figure 3: Variation of Rockwell Hardness Number (RHN) for different combinations of composites.

The variation of the hardness – Rockwell hardness Number (RHN) for the different combinations of the epoxy composites filed with nano clay and nano TiO₂, chopped glass fibre are shown in Fig 3. The plots of tensile strength and hardness show similar patterns as both hardness and tensile strength have a very close relationship. The hardness of fibre and filled epoxy composites increases as compared to the neat epoxy. The results indicate that there is 9% increase in the hardness of the epoxy matrix when the filler concentration is 6% and glass fiber 6% and 23% increase in the hardness when the filler concentration is 6% glass fiber and 6% TiO₂. The decrease in the hardness of the specimens can be attributed to the filler distribution and fabrication techniques.

Abrasive Wear Tests: The specific wear rate at 22N for chopped glass fibre reinforced epoxy composites filled with varying percentages of nano clay and nano TiO₂ is as shown in table 4.

Table 4: Specific wear rate of Chopped Glass fibre reinforced epoxy with nano clay and nano TiO₂ fillers

Sl. No.	Specimen	Specific Wear Rate X10 ⁻⁶ m ³ /min	
		Clay	TiO ₂
1	Epoxy+0%Fillers+0%GF	0.2026	0.2026
2	Epoxy+2% Fillers+2% GF	0.1350	0.0986

3	Epoxy+4% Fillers+4% GF	0.1183	0.1152
4	Epoxy+6% Fillers+6% GF	0.0996	0.1051
5	Epoxy+8% Fillers+8% GF	0.1619	0.1848
6	Epoxy+10% Fillers+10%GF	0.1391	0.1399

The variation of the specific wear rate for the different combinations of the epoxy composites filed with nano clay and nano TiO₂, chopped glass fibre are shown in Fig.4.

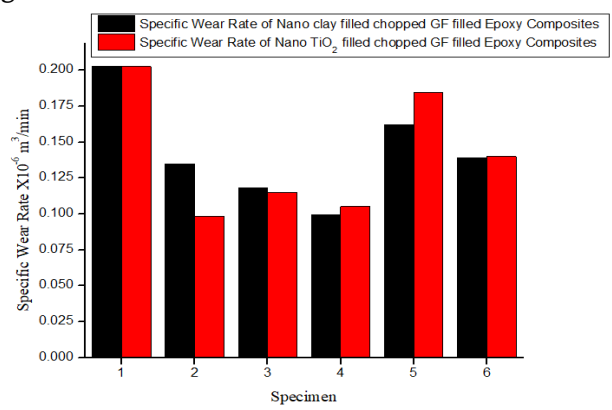


Figure 4: Variation of Specific wear rate for different combinations of composites.

The specific wear rate decreases with increasing filler and fiber concentrations for the fixed abrading distance and applied load. The results revealed higher abrading nature of neat epoxy composites compared to filler and fibre reinforced specimens. The phenomenon of decrease in specific wear rate is due to the nature of fibres and fillers. In the present study, chopped glass fibre reinforced epoxy composite filled with nano clay and nano TiO₂ was fabricated by hand lay-up technique and wooden moulds, characterized by the resin rich top layer. Abrasive wear tests were performed on as cast surface of the composite without disturbing its original surface. Thus, in the initial stage of abrasion, abrasive is in contact with matrix and has less hardness compared to that of angular silica sand. When the chopped glass fibers and fillers are in contact with abrasive particles both provide better resistance

to the process of abrasion. It is seen that the specific wear rate for all the samples is high for the composite with no reinforcements and low for epoxy composites reinforced with chopped glass fibres, nano clay and nano TiO₂. This is attributed to the fact that at lower abrading distance low modulus matrix was exposed and at higher abrading distance high modulus fiber and fillers was exposed to abrasion. These exposed fibers and fillers that are pulled out act as lubricants, because of their high hardness values, provide better resistance against the abrasion and in turn, abrasive particles have to work more that is much higher energy is required to facilitate further failure.

The correlations of wear volume loss with selected mechanical properties such as (σe) factor (where σ is the ultimate tensile strength and e is the ultimate elongation), hardness (H) have been reported for single-pass studies of polymers without fillers and composites [18, 19]. Lancaster [18] stated that the product of σ and e is a very important factor which controls the abrasive wear behavior of composites. Generally, fiber/filler reinforcement increases the tensile strength (σ) of neat polymer, they usually decrease the ultimate elongation (e) and hence the product (σe) may become smaller than that of neat polymer. Hence, reinforcement usually leads to deterioration in the abrasive wear resistance.

Scanning Electron Microscopy: To correlate the wear data better, SEM photomicrographs shown in Figs. 5, 6 and 7 pertaining to a higher load of 22 N for neat epoxy, filler and chopped glass fibre filled epoxy composites are presented.

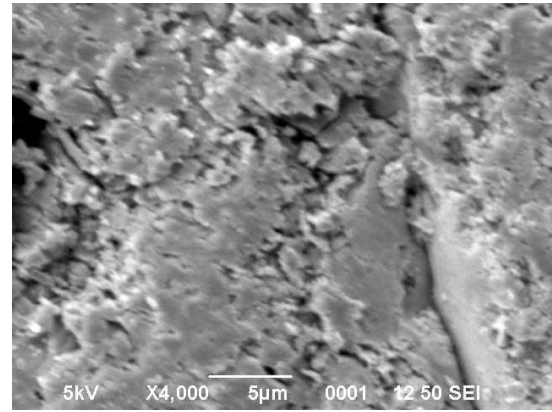


Figure 5: Epoxy without fillers

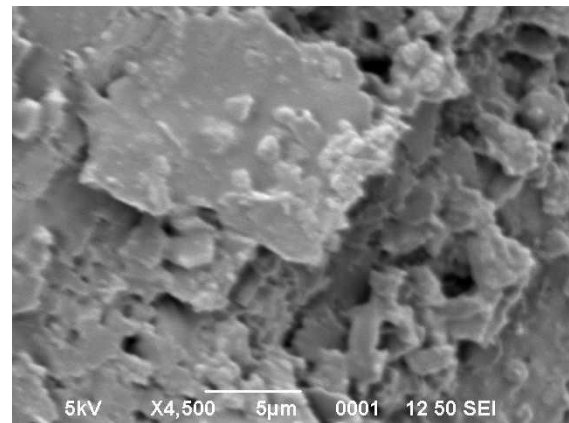


Figure 6: Epoxy with Clay & chopped GF

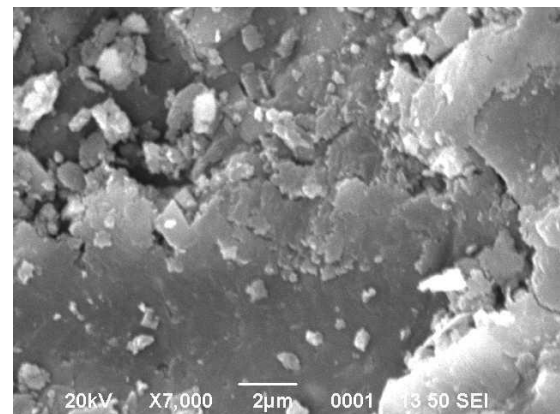


Figure 7: Epoxy with TiO₂ & chopped GF

Observations of the worn surfaces by scanning electron microscope (SEM) provide knowledge about both, the role that nanoparticles play, in the reduction of wear rate and the wear mechanisms. The above figures depict the SEM micrographs of epoxy filled with nano - clay/TiO₂ & chopped glass fibre composites. The pure epoxy had a relatively smooth morphology on the fractured surface upon the addition

of nano - clay/TiO₂ & chopped glass fibre to epoxy, and the morphologies of fractured composites changed dramatically and very rough surface was observed. The nano - clay/TiO₂ & chopped glass fibre are covered with epoxy matrix indicating strong interfacial adhesion between the fillers and polyimide. The fracture surface did not show large voids or cracks.

Main features on the worn surfaces of the neat epoxy resin are severe damage characterized by the disintegration of the top surface, wear debris and deep grooves in the sliding direction (Fig 5). Polymer fragments of the size of micrometres detach and leave small craters behind. These polymer fragments, which might be captured between the counterface and the sample, abrade the sample surface leading to even more substantial loss of material.

In the case of filled nano-composites, the appearances are comparatively different and become rather smooth. Although the ploughing grooves are still visible on the nano-composite sample surface, the groove depths are shallow (Fig 7) and at some region, the grooves are simply invisible. It seems the low filler loading is insufficient and cannot bring significant improvements in wear resistance. The wear performance of a composite material is known to volumetric dependent. It is noted that some cracks across the wear tracks are visible on the composites' worn surface (Fig 6). Very fine scratches are visible resulting from the nano-fillers pulled out of the matrix, and then further moved across the surface by scratching and rolling. Thus it can be concluded that the abrasive wear of neat epoxy is replaced by fatigue wear when the nano particles are introduced. Due to their size, the particles should be able to move into gaps in the counterface and may be exposed to rolling rather than sliding/ scratching movement. Large matrix fragments are not found. During sliding, a rolling effect of nano particles could reduce the shear stress, the friction co-efficient and the contact temperature. Thus, the improvement in the tribological behaviour of nano - clay/TiO₂ & chopped

glass fibre filled epoxy composite is related to the improved characteristics of the transfer film.

IV. CONCLUSION

Tribological properties of nano clay and nano-TiO₂ filled epoxy matrix composites, incorporated additionally with chopped glass fibres, were systematically studied under different sliding conditions. The following conclusions can be drawn:

- The filler morphology, size, particle amount and the dispersion homogeneity influence extensively the composite's Clay/ TiO₂ performance. Composite strength and toughness are strongly affected by three factors – particle size, particle/matrix adhesion and particle loading.
- The addition of Nano Clay/ TiO₂ particles increases the mechanical properties of epoxy composites.
- Abrasive wear of Epoxy filled with Nano Clay/ TiO₂ composites strongly depends on the experimental test parameters such as load and sliding distance.
- Abrasive wear response of these composites under different operating speeds can be successfully investigated by the Dry Abrasion test. The operating speed emerges as the most significant factor affecting wear rate of these composites, other factors like filler content and normal load and their interactions have been found to play significant role in determining wear magnitude.
- Based on the microscopic observations of the worn surfaces, a positive rolling effect of the debris (matrix and nanoparticles) between the sample and the counterface, which led to remarkable reduction of wear. This effect becomes more remarkable when the filler concentrations is 2 wt. %.
- Nano Clay/ TiO₂ filled epoxy composites show signs of mild abrasive wear due to the hard

ceramic particles. The main wear mechanism of composite changed from the severe abrasive wear (for neat epoxy matrix) to mild abrasive wear.

- These composites using fillers Clay and TiO₂ have adequate potential for tribological applications. With the reinforcement of fillers, they exhibit significantly improved abrasive wear resistance.

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