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Mechanical and Tribological Properties of Epoxy Nano Composites for High Voltage Applications

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Abstract

The tribological and mechanical properties of organomodified montmorillonite (oMMT)-incorporated Epoxy (Epoxy-oMMT), vinyl ester (vinyl ester-oMMT) and titanium dioxide (TiO₂)-filled Epoxy (Epoxy-TiO₂) nanocomposites are discussed below. Implications of introducing oMMT and TiO₂ nanoparticles on mechanical and dry sliding wear properties are presented using micrographs of cast samples and through observations of wear affected surface of nanocomposites. Distribution of nanoparticles and their influence on properties are being emphasized for understanding the wear properties. The data on mechanical and tribological properties determined experimentally are compared with published literature. The main focus is to highlight the importance of nanofillers in the design of wear-resistant thermoset polymer composites. A detailed study of strength and moduli of Epoxy-oMMT, Epoxy-TiO₂ and vinyl ester-oMMT nanocomposites was taken up as a part of the investigation. A discussion on density, hardness, tensile, flexural test data, and friction and wear of nanocomposites and analysis of results by comparison with prevalent theoretical models and published results of experiments are presented.

Keywords: organomodified montmorillonite, titanium dioxide, tribological properties friction and wear d wear resistant

1. Introduction

The present day scenario is attracting the polymer nanocomposites with greater interest both in industry and academics because of significant improvement in the properties when compared to base matrix or traditional composites [1]. The important structural characteristics which give unique properties to nanocomposites are the nanosize and a huge interfacial surface area of the nanofillers.

Since 1980s, advantageous properties of polymer nanocomposites were realized at the Toyota research laboratories where they studied nylon-6 based composite system with nanoclay as the reinforcement material to improve the toughness and heat resistance of nylon-6 [2]. Since then, a large quantity of research work has been carried out on polymer composites system using nanoclays and nanoparticle fillers of metallic or nonmetallic type and remarkable enhancements and benefits in several properties have been reported. The intriguing fact is that the properties which were observed in nanocomposites were never anticipated.

Although, the broad group of nanocomposite polymer materials have been widely investigated and fairly understood, the development of thermoset polymer based nanocomposites which also belong to this group, still remains a challenge. This is mainly because of the difficulty in uniform dispersion of nanofiller particles in the thermoset polymer matrix.

Many studies shown improvements in the mechanical strength and modulus of nanoclay filled epoxy resin nanocomposites as compared to the Epoxy resin [3]. Wang and co-authors [4] reported another technique called unique “slurry compounding” to fabricated exfoliated nanoclay-Ethacure nanocomposites. This composites shows improvements in the modulus of epoxy resin by about 40% with 5 wt.% of nanoclay, but with decreased tensile strength. They attributed this partial achievement to the feeble surface interface and occurrence of air foam in the nanocomposite samples. Gefu and co-authors [5] studied vinyl ester with 1 wt.% nanoclay. They have compared five types of mixing combinations such as (i) 30 min manual stirring, (ii) 30 min manual stirring and 1 h sonification mixing, (iii) 30 min manual stirring and 1 h sonification mixing followed by three-roll mill shear mixing, (iv) 30 min manual stirring and 1 h sonification mixing followed by three-roll mill shear mixing and 1 h sonification mixing, and (v) 30 min manual stirring and 1 h sonification mixing followed by three-roll mill shear mixing with 1 h sonification mixing and three-roll mill shear mixing while holding other factors constant.

It could be generally concluded that intensive mixing improved exfoliation until a certain point (defined as optimum point) and then its positive effects diminish with further mixing efforts. Considering reduced mixing effort of method iv, it is being recommended. Kotsilkova and co-authors [6] studied the flexural strength and flexural modulus of epoxy-clay nanocomposites fabricated by direct method or without solvent method and indirect method or solvent processing techniques. Different organic modifiers were used to functionalize the clay. It has been observed that the mechanical properties are majorly dependent on nanocomposite structure and the type of solvent used. The fabricated samples by direct processing method or without using solvent technique showed increased flexural modulus. The modulus of the nanocomposites at 5 volume percentage of clay content increased significantly.

In compare, the flexural modulus of fabricated nanocomposites fabricated by adding solvent or solvent processing technique is not affected [7]. The flexural strength of the fabricated nanocomposites has increased when compared to the samples fabricated by the direct processing method or without using solvent. This is due to the strength of the solvent, acting as a plasticizer for the base resin and alters the interfacial adhesions. In earlier studies it is reported that nanoclay appears to have either the intercalated structure or the partially exfoliated structure. The maximum improvement achieved in the modulus was of the order of about 80% with 10 wt.% nanoclay [7].

While the addition of nanoclay to epoxy resin generally increases the tensile modulus, it decreases the tensile strength and fracture strain [8, 9]. This was mainly attributed to the agglomeration of nanoclay and presence of voids. Organically modified nanoclay effect on the physical, structural properties of organically modified clay-epoxy nanocomposites samples was examined by Dean and co-authors [10]. They reported that different morphological structures such as intercalation, partial and full exfoliation of nanoclay. The degree of intercalation morphology varies and interlayer expansion was observed. This is related to the mechanical properties of the fabricated nanocomposite samples. With the increase in the clay content, 35% increase in tensile modulus for the 2 wt.% sample, 15% increase in tensile modulus for 4 wt.% sample and 30% increase in the tensile modulus for the sample with 6 wt.% respectively.

Isık and co-authors [11] reported the clay loading effect on the tensile strength of organically modified clay filled epoxy nanocomposites. The tensile strength decreases with increasing content of clay. This is due to the nonuniform dispersion of clay, agglomeration of clay causes stress concentration and decreased polymer clay interactions. They also observed that the tensile modulus of the nanocomposite increases with increasing amount of clay. The mechanical properties of montmorillonite filled epoxy nanocomposites were studied by Akbari and Bagheri [12]. They found a decrease in flexural strength with increasing the content of organically modified clay and this is due to the fact that intercalated structure of clay in the nanocomposites. Kaya and co-authors [13] analyzed the flexural and tensile properties of clay filled epoxy nanocomposite samples. The surface modified and unmodified montmorillonite clay were added to the epoxy matrix with increasing clay contents from 1 to 10 wt.%. The author presented that both tensile and flexural modulus values tend to increase with increasing content of unmodified and modified clay. With the increase in clay loadings, surface modified clay showed a little higher values of elastic modulus when compared to surface unmodified montmorillonite clay. This is due to the fact that the effect of surface modification causes better level of penetration of the epoxy resin into the galleries of montmorillonite clay.

From the above studies, it was found that the mechanical properties such as flexural and tensile strength reduce for the fabricated nanocomposite samples with surface modified and not modified clay when compared to pristine base matrix. This is mainly due to the creation of voids, feeble adhesion points along the filler-matrix surface interface during nanocomposite processing. Velmurugan and Mohan [14] have reported the presence and effect of quaternary ammonium modified montmorillonite clay content on the mechanical properties of clay-epoxy nanocomposites. They reported that the tensile modulus of the fabricated samples increased about three times when compared to base pristine epoxy with the accumulation of 10 wt.% surface modified clay. This is due to the fact that the exfoliation structure of clay layers and surface interfacial adhesion between the nanoclay and the epoxy matrix, whereas, the tensile strength reduced in spite of the content of the clay as compared to pristine polymer (epoxy).

Yasmin and co-authors [15] conducted studies on the mechanical characteristics of surface modified montmorillonite clay added epoxy nanocomposite samples with variation in concentration of clay from 1 to 10 wt.% fabricated by shear mixing technique. Based on their report, the addition of surface modified nanoclay particles there is a significant improvement both in elastic and storage modulus of base epoxy resin. In addition, there is an increase in storage modulus with increasing in clay content. This was ascribed to intercalation or exfoliation structure of surface modified nanoclay (Cloisite 30B) in base epoxy matrix. The results reconfirmed that there is a direct relation between the degree of intercalation or exfoliation and the mechanical properties of the fabricated nanocomposites samples.

Basara and co-authors [16] analyzed the presence and effect of clay substance and morphological structure on mechanical properties of nanocomposites. The organically surface modified (Cloisite 30B) and natural or unmodified (Cloisite Na⁺) clay with the content varying from 0.5 to 11 wt.% was reinforced into base epoxy matrix and observed that tensile modulus increased with clay content. This is attributed to stiffening effect of montmorillonite clay in two types of clay reinforced nanocomposites. Cloisite 30B and Cloisite Na⁺ reinforced nanocomposite samples showed utmost tensile strength at 1 and 0.5 wt.% respectively. The reason for the decrease in tensile strength at larger montmorillonite clay content is due to the stress concentration effect of the agglomerated clay particles and lesser surface interactions.

Zhu and co-authors [17] investigated PA6/TiO₂ nanocomposites which resulted in improved spinnability and mechanical properties. It was observed that tenacity at break and the initial modulus of PA6 with modified nano-TiO₂ composites showed improvement by about 10, 20%, respectively, as compared to the pure PA6.

Mina and co-authors [18] studied TiO₂ filled isotactic polypropylene (iPP) composites with TiO₂ which was first single-extruded by an extruder and then double-molded by compression molding. Micro-hardness increased rapidly and then leveled off with increasing filler content. Variations with respect to molding conditions were also observed.

According to Dean and co-authors [19] the inter-gallery spacing of surface modified montmorillonite nanoclay increased efficiently. Ha and co-authors [20] have demonstrated that the tensile strength and elastic modulus of surface modified montmorillonite nanoclay Epoxy nanocomposites are elevated than that of unmodified montmorillonite nanoclay-Epoxy composites. Ha and co-authors [21] have stated that surface modification increases the interlayer gallery expansion, in turn improves the dispersion of nanoclay into the base polymer. Wang and co-authors [22] reported that improvement in the modulus and fracture toughness of base polymer such as epoxy was achieved by adding surface modified nanoclay using silane functionalizer. Lam and co-authors [23, 24] reported the effect of bunch of nanoclay and shows there is a best nanosized clay weight percentage in the nanocomposites in order to acquire maximum mechanical properties. The highest hardness is achieved for 4 wt.% of nanoclay. Furthermore, increasing the nanoclay content affected in reducing the hardness of the nanocomposites [23, 24].

Huang and co-authors [25] fabricated the composites of TiO₂ and epoxy by direct solution adding method, where directly epoxy and nanosized TiO₂ in the liquid form were mixed with the catalyst methyl isobutylketone. Based on the results obtained from XRD, the hydrogen bonds are formed by adding TiO₂ nanoparticles in epoxy resin. The Scanning Electron Microscopy result recommended that TiO₂ nanoparticles are uniformly distributed within the epoxy nanomaterial. The nanocomposite properties degrade if the nanofillers TiO₂ exceeds 3 wt.%. In addition, mechanical properties are enhanced by raising the concentration of catalyst such as methyl isobutylketone.

Elansezhian and co-workers [26] studied the wear and tensile strength behavior of vinyl ester with silica, alumina and zinc oxide nano fillers and reported that the addition of silica to the vinyl ester resin significantly improved wear resistance as compared to other two fillers. The functionalized silica nanoparticles showed an improved dispersion with vinyl ester resin. Functionalization caused particle dispersion more uniformly in the polymer matrix. As-received nanoparticles show lower tensile strength whereas functionalized nanoparticles show improved tensile strength by more than 15% at 5 wt.% loading as compared to unfilled resin.

Haque and co-authors [27] reported that the flexural strength of glass-vinyl ester-clay nanocomposites containing 1 wt.% nanoclay improved by 24% in comparison to conventional composites. The improvement in the mechanical properties of the nanocomposites mostly relies on achieving proper polymer/silicate layer (intercalated) or uniformly dispersed (exfoliated) morphology in the structure. It is observed that incorporation of higher percentage of nanoclay particles (> 2 wt.%) into the resin, may not improve mechanical properties.

Apoorva and co-authors [28] reported that tensile modulus of nanocomposites with nanoclay (cloisite 10A and Vinyl monomer) increases by about 25% with organically modified clay addition. The tensile modulus improved in the nanocomposites with Cloisite in a more effective manner. Whereas, at elevated clay load, cloisite 10A and Vinyl monomer clays emerge to execute uniformly good,

the mechanical strength appears to be marginally reduces with cloisite 10A nanocomposites. Vinyl monomer clay nanocomposites prove a little diverse trend [28]. The little concentration of Vinyl monomer clay in the nanocomposite system, the tensile strength show to enhance and this enhance is dedicated to the chemical link created connecting Vinyl monomer clay's surface functionalization and Vinyl ester manacles through the process of curing, which is additional useful in transfer pressure to the nanoclay platelets compared to an unreactive clay such as cloisite 10A. The succeeding small fall in tensile strength at 1 wt.% and two and half weight percentage clay content was due to lack of proper distribution of clay.

The attempts have been made to fabricate nanoparticle filled epoxy nanocomposites to progress the tribological properties of the base polymers. Recently, nanosized inorganic fillers such as nanometer sized Silicon Carbide (SiC), Silicon di-oxide (SiO₂), Zinc oxide (ZnO), Zirconium di-oxide (ZrO₂), Alumina (Al₂O₃) and Titanium di-oxide (TiO₂) be used as nanofillers in epoxy to attain excellent tribological property [29, 30]. The main benefit of nanosized filler filled composites over micro-sized filler filled composites lie down in its performance development which is attained at comparatively small percentage of the nanofillers. At higher filler loadings, due to higher percentage of particles or atoms present on the surface of the nanofillers, agglomeration takes place very easily because of nonuniform dispersion of nanofillers in the polymer by using conventional mechanical mixing techniques [31]. This results in the poor performance of the nanocomposites at higher percentage of loading than the micro filler based composites system.

Many authors reported that, polymer composites is widely used in wear applications such as cams, brakes, bearings and gears due to their self-lubrication property, low co-efficient of friction and improved resistance to wear. The inborn shortage of polymers is changed profitably by using special micro fillers and nanosized particles. More polymer micro and nanocomposites are in use as sliding devices which were formerly made of metallic materials. On the other hand, extreme improvements are in progress to find out different areas of purpose to modify their properties for higher load-carrying and ecological conditions [32, 33].

Extensive work has been approved in the development of nanofiller composites through the addition of nanosized particles namely ceramics and carbon in polymer based matrices. For example surface modified nanoclays [34], carbon nanotubes [35], alumina [36] ZnO and SiC nanoparticles [37] are incorporated in to the base polymers. Majority of these cases are on the effect of filler particle in polymer composites descending against metallic surface results in decrease of rate of wear, friction co-efficient and larger mechanical strength which is owing to incorporation of nanofillers.

Recent reports on the wear behavior of surface modified nanoclay added polyamide6 (PA6) by Srinath and Gnanmoorthy [38] and Dasari and co-authors [39] has exposed lesser friction and higher rate of wear resistance, this is dedicated to the increase of the bond between nanofiller and the polymer matrix.

Nanofillers inclined to develop a persistent transmit layer on the opposite face which defends the nanocomposite sample outside surface from straight make contact with the opposite face thus reduces rate of friction and rate of wear. The incorporation of organically modified nanoclay into the base polymer matrix to formulate the nanocomposite is an alternative way to change the properties of composite material [40]. Ng and co-authors [41] directly dispersed TiO₂ nanoparticles in to the epoxy. The resultant composites appeared not only to be tougher than the traditional micro-particle filled epoxy but also to possess a higher scratch resistance. Rong and co-authors [42] also carried out wear trial on nanotitanium-epoxy nanocomposites and studied that the tribological characteristics is importantly depends on the state of dispersion and homogeneity of the filler.

Shao Rong and co-authors [43] have proved that the epoxy/SiO₂-TiO₂ nanocomposites are effective in lowering the frictional coefficient and wear rate. The results of these experiments [43] indicate that the wear mechanism of composites changed from adhesive wear to mild abrasive wear and fatigue wear with the increase of the SiO₂-TiO₂ content. Chang and co-authors [44] have proved that the addition of nanoTiO₂ apparently reduced the frictional coefficient and significantly enhanced the wear resistance of the composites, especially at contact pressures and sliding velocities.

The present study incorporates different evaluation techniques to encompass properties of mechanical and wear characteristics.

2. Results and discussion

2.1 Density of the nanocomposites

Density is an important property in several weight sensitive applications. In many applications, polymer composites are replacing traditional or conventional metals and metal based composites mainly for their low densities. Density of a composite depends on the relative proportion of matrix and the reinforcing filler. There is always a difference between the measured and the theoretical density values of a composite due to the presence of empty space and hole. These empty spaces or voids majorly affect the performance of the nanocomposites. A larger void content indicates lower value of fatigue resistance, greater susceptibility to water penetration and weathering [45]. The theoretical and measured densities of nanocomposites along with corresponding volume fraction of voids are presented in **Table 1**. The theoretical density was calculated for nanocomposite samples by weight additive principle, which states that:

$$d = w_1 d_1 + w_2 d_2 \quad (1)$$

where d is the density of the nanocomposite, w_1 and w_2 are the weight fractions of the fillers, epoxy and hardener, d_1 and d_2 are their corresponding densities. It

Samples (wt.%)		Density (g/cm ³)		Void fraction (%)
		Theoretical	Experimental	
Epoxy-oMMT	0	1.170	1.170	-
	2	1.184	1.176	0.65
	5	1.206	1.181	2.03
	7	1.221	1.182	3.13
Epoxy-TiO ₂	2	1.231	1.222	0.73
	5	1.323	1.284	2.91
	7	1.384	1.294	6.50
Vinyl ester-oMMT	0	1.346	1.346	-
	2	1.351	1.347	0.29
	5	1.368	1.348	1.46
	7	1.379	1.349	2.14

Table 1.
Theoretical, experimental densities and void fractions in nanocomposites.

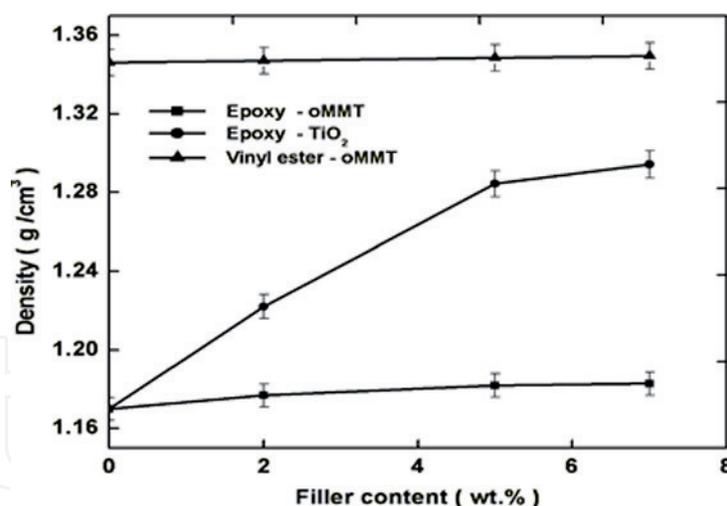


Figure 1.
Variation of density as a function of filler content for epoxy and vinyl ester nanocomposites.

may be noted from **Table 1** that density values of the nanocomposite calculated theoretically from weight fractions using Eq. (1) are not in agreement with the experimentally determined values. Theoretically calculated density values are larger when compared to equivalent experimental values due to empty space or voids and holes or pores.

The knowledge of void fraction is therefore essential for estimation of the quality of the composites. From the measured densities of Epoxy-oMMT, Epoxy-TiO₂ and Vinyl ester-oMMT nanocomposites (**Figure 1**), it is clear that pure Epoxy and Vinyl ester resin has an average density of 1.17 and 1.34 g/cm³ with an error of ± 0.0088 respectively. With the incorporation of oMMT and TiO₂ nanofillers, an increase in the density of the nanocomposites is observed. This is observed to be true in both Epoxy and Vinyl ester matrix with nanosized oMMT and TiO₂ filler inclusions.

It is evident from **Figure 1** that the density values for Epoxy-oMMT nanocomposite increases with the nanofiller loading content. However, the increase in the density was small in Epoxy-oMMT nanocomposites. With the addition of 7 wt.% oMMT, the density of Epoxy increases by 1% but the density of Epoxy-TiO₂ increases by 10%. This is due to higher density of TiO₂ as compared to oMMT. The density of Vinyl ester is higher than that of Epoxy due to the presence of bromine. Bromine is a heavy atom and there are four bromine atoms bonded in one molecule, and this result in density being higher for brominated Vinyl ester. Similar trends are noticed for Vinyl ester-oMMT nanocomposites.

2.2 Micro-hardness of the nanocomposites

Hardness is one of the significant factors that control the wear property of the composite samples. In this work, the data obtained for micro-hardness of the Epoxy and Vinyl ester composites with different types of weight percentage of fillers loading have been obtained. **Figure 2** shows the experimental results of measurements of micro-hardness of pure Epoxy and Epoxy-oMMT, Epoxy-TiO₂, pure Vinyl ester and Vinyl ester-oMMT nanocomposite samples with different weight percentage of nanofiller content.

Figure 2 indicates that the addition of oMMT nanofiller into Epoxy, Vinyl ester and TiO₂ to Epoxy results in significant improvement in hardness of nanocomposites. In general, mechanical properties of nanocomposite depend primarily on following things namely filler content, filler size and shape, the degree of interfacial

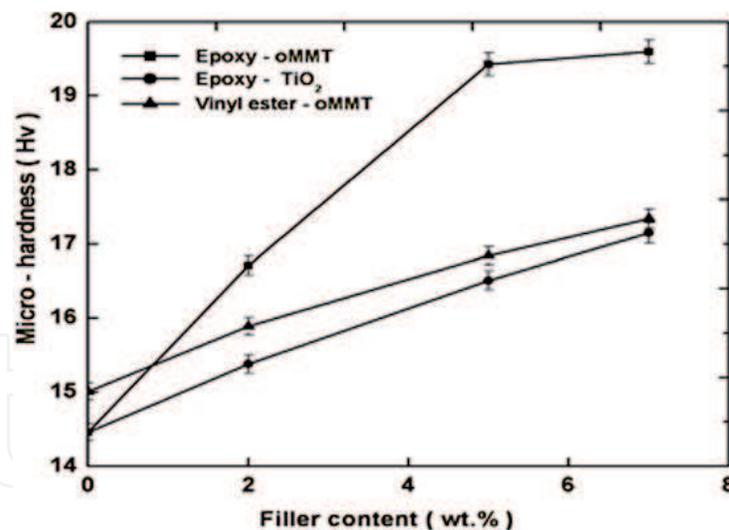


Figure 2.
Micro-hardness of epoxy and vinyl ester nanocomposites.

interaction or between the filler particles and polymer matrix and degree of dispersion of nanofiller within the polymer matrix. The improvement in the hardness property of the nanocomposites may be attributed to the intercalated and exfoliated clay platelets structure [46].

The intercalated/exfoliated clay platelets effectively restrict indentation and increase the hardness of the nanocomposites [46]. Similarly, incorporation of TiO₂ into Epoxy increases the degree of dispersion, thereby increases the hardness of nanocomposite. Organomodified montmorillonite has a much greater surface hardness because of its ceramic nature. Therefore, the contribution of oMMT to the hardness is greater as compared to TiO₂ filled nanocomposites. From the observed data it is clear that the increment in hardness is more significant up to 5 wt.% of filler loading. The increase in hardness for 5 wt.% filled polymer matrix may be partly attributed to the intrinsic hardness of nanofiller and the nanoparticles might be offering better resistance against Epoxy and Vinyl ester segmental motion under indentation.

On the other hand, increase in filler loading from 5 to 7 wt.% and the rise in value of the hardness is trivial implying that higher filler loading gives rise to undesired dispersion and agglomeration in the polymer matrix. When in comparison with the oMMT and TiO₂ nanocomposites loading is concerned, the Epoxy-oMMT nanocomposite reveal greater hardness values for the different filler loadings are concerned. Among all the nanocomposites investigated maximum micro-hardness value is recorded for Epoxy nanocomposite filled with 7 wt.% oMMT.

2.3 Mechanical properties

The measured tensile properties of Epoxy-oMMT, Epoxy-TiO₂ and Vinyl ester-oMMT nanocomposites are shown in **Table 2**. It is noticed from the **Table 2** that, lower values of tensile strength and tensile elongation are obtained for nanocomposites than those of pure Epoxy and Vinyl ester. However, the Young's moduli are evidently enhanced by the addition of fillers. As seen in **Table 2**, the tensile strength of these nanocomposites invariably decreases with increase in filler loading irrespective of the type of filler. Similar observations have been reported by some investigators [47].

The tensile strength and fracture toughness of a nanocomposite samples depend upon the filler shape, size and amount which is mixed with the polymer matrix, the strong bond between the nanofiller and the polymer matrix, the robustness of the

polymer matrix and the filler. Nanofillers affect the mechanical properties in accordance with their packing characteristics, size and interfacial interaction. The highest three dimensional packing of filler reflects the size distribution and shapes of the particles. The space between the particles is implicit to be packed with polymer matrix and no empty spaces or air bubbles are expected. With this condition, for a given nanocomposite, the matrix amount is fewer and it acts as separate segment or compartment to hold up tensile load. Properties of nanocomposites are manipulated by the individual properties of the filler and the matrix and also the filler-matrix interface.

Ability of the matrix to transfer the load to the filler particles, is its major purpose, and depends on the adhesion and compatibility between the filler and matrix. The tensile strengths of the nanoparticles filled Epoxy are less than that of pure Epoxy/Vinyl ester. The decreasing trend of adhesion with respect to relatively higher filler loading points towards the phenomenon of dewetting occurs at the interface. The figure shows a close up view of matrix region where more particles are concentrated. There can be two reasons for the decline in the strength properties of the filled nanocomposites as compared to the pure polymers which is evident from data shown in **Table 2** and discussed below;

Samples (wt.%)		Tensile strength (MPa) \pm 1.5%	Tensile modulus (GPa) \pm 1.5%	Elongation at break (%) \pm 2%
Epoxy-oMMT	0	47.50	1.44	10.39
	2	31.56	1.73	10.50
	5	37.95	1.90	11.30
	7	33.79	2.09	9.35
Epoxy-TiO ₂	2	32.60	1.61	10.41
	5	36.91	1.72	10.98
	7	32.47	1.82	11.18
Vinyl ester-oMMT	0	71.10	3.49	2.54
	2	69.40	3.55	2.38
	5	66.21	3.74	2.22
	7	64.10	3.91	2.08

Table 2.
Mechanical properties of polymer nanocomposites.

In the present study, the tensile strength of all nanocomposites is much lower than that of pure Epoxy and Vinyl ester may be assumed to be closely related to processing method. The compounding of oMMT in an Epoxy matrix with high shear mixing produces a highly viscous and foamy material. A higher content of oMMT leads to higher viscosity. Furthermore, the diethyltoluenediamine used to functionalize Nanomer 1.30E can voluntarily contribute in the curing reaction and delamination of the clay platelets. This results in highly viscous fluid with time and hampers the complete removing of gas before the process of curing. The nanocomposites with 7 wt.% of oMMT, the removal of gas turn out to be more critical. The presence of small gas bubbles in 2, 5 and 7 wt.% oMMT nanocomposites confirms the same.

The additional reason for voids can be air entered during transfer of the largely gelatinous material into the mold. Furthermore, the tensile strength of all the samples in the range of 30–70 MPa indicates break initiation from analogous types of defects. Consequently, it might be understood that under loading condition, breaks may begin from these tiny spaces or voids and result in sample failure at

fairly low strains [48]. The other reason is that the corner points of the irregular shape of the spherical filler namely TiO_2 particulate, may result in stress concentration in the Epoxy matrix. These two factors are perhaps responsible for significant reduction in tensile strengths of the nanocomposites. A comparison of the results reveals that the Vinyl ester-oMMT nanocomposites possess higher tensile strength confirming the effect of incorporation of oMMT filler which improves nanofiller-matrix surface adhesion or interface in the composite.

The tensile modulus shows a marked increase with increasing oMMT, TiO_2 content from 0 to 7 wt.% in Epoxy and Vinyl ester matrices. The improvement in the Young's modulus can be attributed to the exfoliation and good dispersion of nano-sized filler particles that restricts the mobility of polymer chains under loading as well as good interfacial adhesion between the particles and the polymer matrix [49]. The orientation of clay platelets and polymer chains with respect to the direction of loading can also contribute to the reinforcement effects [49]. Unlike the Young's modulus, nanocomposites of any nanoclay content show a lower tensile strength than that of pure Epoxy. Similar results are reported by Zerda and co-authors [50] but in contrast, an increase in tensile strength values of nanocomposites are also reported [51, 52].

As reported in literature, the strain at break of nanocomposites usually declines with increasing filler content. Low filler loadings cause a significant drop in fracture strain. It may be recalled that the composite is made up of partly filler and partly matrix. Due to the rigid nature of the fillers, most of the deformation comes from the polymer. The actual deformation is experienced only by the polymer matrix which is much larger than the measured deformation of the sample. With the result, that the polymer failure reaches failure strain limit at lower level of total deformation. Hence, the total composite strain-to-break decreases. However, it is interesting to observe that nanocomposites of the present study show contrary results that is strain-to-break behavior as comparable to conventionally filled composites. It tends towards slightly higher values for filler content of less than 5 wt.% (**Table 2**).

This increase suggests that the nanoparticles are able to introduce additional mechanisms of failure and energy consumption without blocking matrix deformation. Particles may induce matrix yielding under certain conditions and may furthermore act as inhibitors to crack growth by pinning the cracks [15]. Nevertheless, if the fillers exceed 5 wt.%, the failure strain undergoes a slight decrease. Such a reduction leads to that the large proportion of fillers start dominating, and they reduce the matrix deformation by mechanical restraining.

The decrease in tensile strength and the upgrading of values of hardness with the filler addition is due to the following reasons: under the action of a mechanical (tensile) force, the interface between filler and polymer matrix will tend to debonding, depending on interfacial bond strength and this can direct to a break in the nanocomposite material. On the other hand, in case of hardness, pressing stress is in act. The matrix phase and the nanofiller phase would be hard-pressed together. Thus the interfacing bond can transfer pressure more effectively and thus there is an enhancement in the values of hardness.

2.4 Flexural properties

Table 3 lists the average values of flexural modulus for different weight percentages of oMMT and TiO_2 nanofillers. The flexural modulus of Epoxy resin is 2.30 GPa. The addition of oMMT and TiO_2 to Epoxy resin is expected to reinforce the resin and increase its elastic modulus and the results are on expected lines. The flexural modulus of nanocomposites shows increase with oMMT and TiO_2 concentrations. For 7 wt.% of oMMT and TiO_2 loading, improvement in the flexural

Samples (wt.%)		Flexural modulus (GPa) \pm 1.5%	% change	Flexural strength (MPa) \pm 1.5%	% change
Epoxy-oMMT	0	2.30	—	89	—
	2	2.34	1.70	91	2.20
	5	2.57	10.50	95	6.31
	7	2.69	14.49	100	11.0
Epoxy-TiO ₂	2	2.52	8.73	96	7.29
	5	2.85	24.12	102	12.74
	7	3.07	33.77	105	15.23
Vinyl ester-oMMT	0	3.56	—	99	—
	2	3.79	6.06	63	36.36
	5	4.05	12.09	56	43.43
	7	4.26	16.43	45	54.54

Table 3.
 Flexural properties of the polymer nanocomposites.

modulus is 14 and 33% respectively. The addition of 7 wt.% oMMT into Vinyl ester increases the flexural modulus by 16% and the flexural strength of Epoxy resin and Vinyl ester resin are observed to be 89 and 99 MPa respectively. The maximum improvement in the flexural strength is observed with the addition of 7 wt.% oMMT and TiO₂ and it is 11 and 15% as compared to pure polymer. Significant improvement in the flexural strength of around 54% is observed at 7 wt.% Vinyl ester-oMMT nanocomposite.

The gradual increase in flexural strength and modulus, of nanocomposites, reveals that mechanical stresses are efficiently transferred via the interface. The significant uniqueness of nanocomposites is considered to explain this phenomenon about quality of the interface in the nanocomposites, (static adhesion strength) usually take part in significant responsibility in the materials' capability to convey stresses and elastic deformation from the base polymer matrix to nanofillers [15, 16].

This is correct for nanofillers filled composites, because they have high part of interfacing. If the interaction between filler and matrix is poor, then filler particles are not capable of carrying out any part of the outside weight. Further, the power of the nanocomposite will not be superior as compared to that of pure polymer matrix. Instead of this, if the fillers and matrix bond is strong, then the tensile strength of composite can be higher than that of polymer matrix [15, 16]. The comparison of the results reveals that Vinyl ester-oMMT nanocomposite shows higher flexural strength and modulus.

2.5 Friction and dry sliding wear behavior

In **Figure 3**, the variation of the coefficient of friction can be observed for the three nanocomposite materials, as a function of the sliding distance, for load of 30 N and sliding velocity of 1 m/s. Initially, an increase in friction coefficient is associated with the initial running-in period is observed. After the initial running-in period the friction coefficient stabilizes as a function of sliding distance. Epoxy-TiO₂ shows lesser coefficient of friction, followed by Epoxy-oMMT. Among the three nanocomposite systems studied, pure Epoxy shows higher coefficient of friction for all the sliding distances.

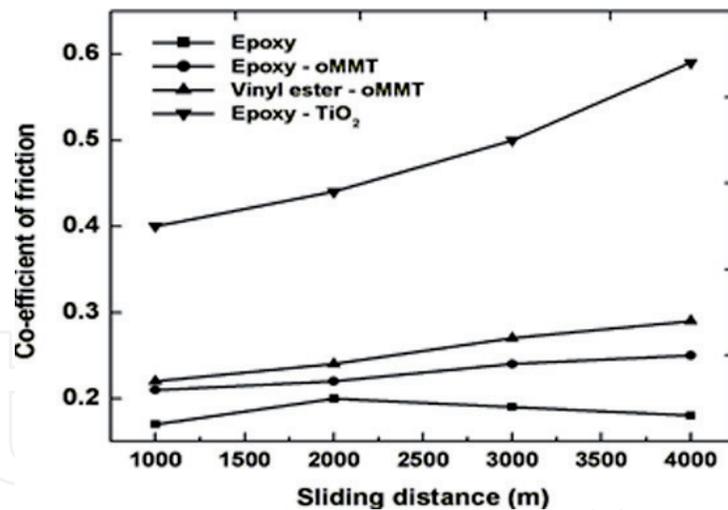


Figure 3.
Coefficient of friction of 5 wt.% filler filled polymer nanocomposites.

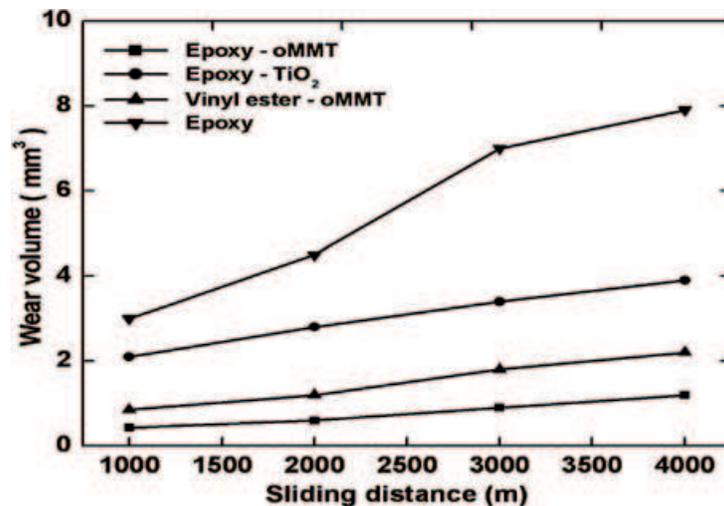


Figure 4.
Wear volume as a function of sliding distance of 5 wt.% filler filled polymer nanocomposites.

The variation of wear volume as a function of sliding distance is shown in **Figure 4**, for the three nanocomposites, for load of 30 N and sliding velocity of 1 m/s. As seen in the **Figure 4**, a linear increase of the wear volume loss as a function of the sliding distance is observed. This increase is more significant for the pure Epoxy. Nanoparticles filled polymer composites such as Epoxy-oMMT, Epoxy-TiO₂ and Vinyl ester-oMMT have shown a lesser wear volume loss during the sliding processes.

The wear volume and specific wear rate (K_s) as a function of sliding distance for pure Epoxy and oMMT filled Epoxy nanocomposites are shown in **Figures 5** and **6** respectively. From **Figure 5**, it is observed that the wear volume loss increases with increase in sliding distance for all the nanocomposites. Wear volume loss showed an upward trend in the gradient, as the sliding distances were increased in case of pure Epoxy (**Figure 5**). The sliding over a pure epoxy surface with a flat steel, the counter surface of which is ground to a roughness of 0.25–0.30 μm (arithmetic mean) under a load of 30 N and at a velocity of 1 m/s, resulted in a specific wear rate of $10.54 \times 10^{-5} \text{ mm}^3/\text{Nm}$ for 1000 m sliding distance (**Figure 6**).

The result obtained possibly may be due to enhanced temperature occurs for the duration of the process of wear. The wear resistance varies in the order of weight percentages as follows; 5 > 2 > 7 > 0% by weight of oMMT filler in polymer matrix. It is clear from the data in the **Figure 6** that the specific wear rate value of pristine

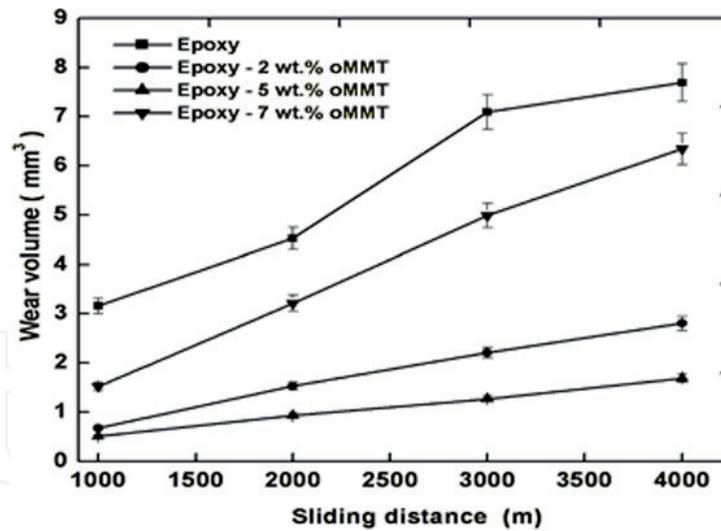


Figure 5.
 Wear volume as a function of sliding distance of pure epoxy and oMMT filled epoxy nanocomposites.

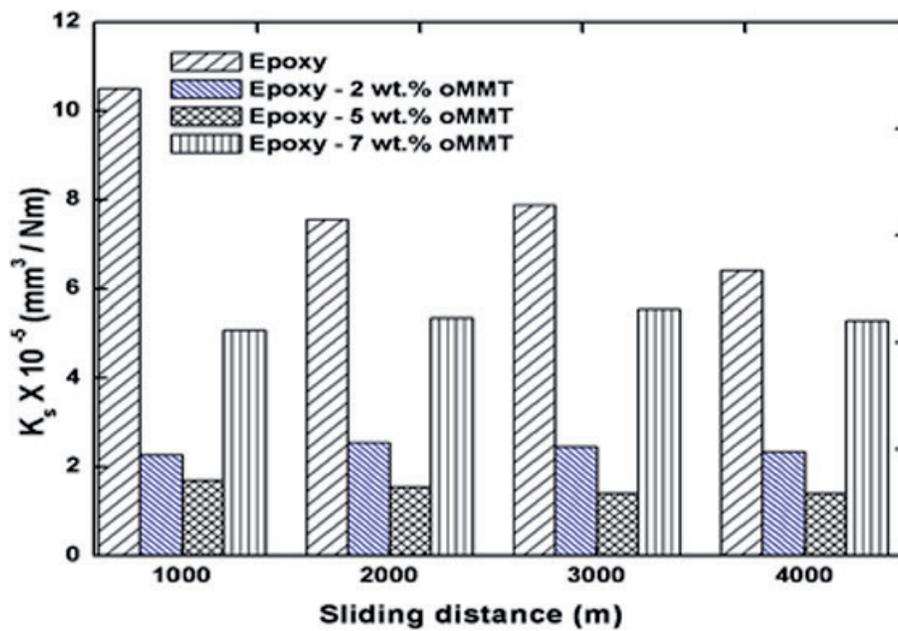


Figure 6.
 Specific wear rate as a function of sliding distance of pure epoxy and oMMT filled epoxy nanocomposites.

Epoxy be linear up to 2000 m and rises nonlinearly up to 4000 m. The specific wear rate of oMMT nanofillers filled Epoxy nanocomposites decreases linearly with increase in sliding distances except in the case of 7 wt.%. Epoxy resin with 5 wt.% of nanofillers (oMMT) has the smallest specific wear rate, whereas specific wear rate of pristine Epoxy rises above 5 wt.%. This is due to the poor interaction or adhesion Epoxy matrix nano-particles and the accumulation of the nanoparticles at 7 wt.% filler content.

The incorporation of 5 wt.% of oMMT in Epoxy, results in decrease in specific wear rate by 70–80%. The tendency is in agreement with that of the tensile strength and hardness results and is listed in **Table 2** and **Figure 2** respectively. It is well known that majority of micro-fillers used are generally very successful in the reduction the wear property of different polymers. In the pristine Epoxy, wear trash consists of deformed polymer matrix containing broken down and powdered matrix particles and wear powder of the metallic opposite surface. The constituent parts can either be lost from the contact region or stay there for unchanging time as a

“transport layer.” In such cases, the polymer constituent can cushion the opposite surface and reduces the toughness, but the broken matrix particles and wear powder of the metallic opposite surface will take steps as a third body abrasive guiding to increase the abrade of the opposite surface. Thus, specific wear rate of the pristine Epoxy depend on presence of various elements in the wear trash. During the process of wear, no film was formed on the opposite surface and thus maximum specific wear rate was observed in pristine Epoxy.

The wear volume loss is low for oMMT filled Epoxy nanocomposites as compared to pure Epoxy. At the beginning of sliding, the two surfaces of all the asperities are in contact with each other. When shear forces are applied, the asperities get deformed. The oMMT particles protrude out from the surface of the sample and initially, the Epoxy matrix wears out and only oMMT nanoparticles remain in contact with the countersurface. As sliding distance increases, the wear rate starts decreasing and the oMMT nanoparticles wear out the steel countersurface. Due to extreme hardness of the countersurface, oMMT nanoparticles adhere to the matrix and excess filler concentration is observed on the exposed composite surface after prolonged sliding. During sliding, a rolling effect of nanoparticles may reduce the shear stress, the frictional coefficient, and the contact temperature.

For the oMMT filled Epoxy nanocomposites, a three-body contact condition is induced by the addition of nanoparticles between the contact surfaces, which is evidenced by presence of the grooves on the worn surfaces (**Figure 8a** and **b**). Hence, it is proposed that during the sliding process, many of the hard particles are embedded in the soft polymeric transfer film on the counter surface which creates grooves on the sample surface. In this way, the distance between the countersurface and the sample gets enhanced, i.e., the particle act like as spacers. This in turn, can cause reduction in the adhesion between the contacting surfaces. Therefore, the coefficient of friction of oMMT filled Epoxy is always less than that of pure Epoxy. Moreover, as the nanoparticles are free to move and they tend to be dispersed uniformly over the transfer film during the wear process. This can result in a more uniform contact stress between the contact surfaces and in turn minimizes the stress concentration [53–55].

In the present work, the wear-resistant of Epoxy nanocomposite filled with small size oMMT particles (<40 nm) transfers well to the counterface and its transfer film is thin, uniform and adheres strongly to the countersurface. Thus, the improvement in the tribological behavior of Epoxy-oMMT nanocomposite is related to the improved characteristics of the transfer film.

2.6 Morphology of wear affected surface

SEM assessment of worn surfaces of pure base resin and organically modified montmorillonite nanoclay (oMMT) added nanocomposites against metal counter surface with a load of 30 N and sliding velocity of 1 m/s are given in **Figures 7a, b** and **8a, b** respectively. **Figure 7a** and **b** represents the features worn surfaces of base resin. Projectile marked depicts the direction of sliding. At lesser enlargement, the worn surface is moderately rough and allied with micro-cracks in the epoxy (**Figure 7a**). The advanced microscopy image indicates (**Figure 7b**), break to the epoxy matrix is elevated ensuing in extra base material elimination from the nanocomposite surface. The loss and contact temperature are significantly enlarged, leading to increase in fracture of the base epoxy near the region of interface.

The interface surface break extremely increases with channel by the Epoxy removed (**Figure 7b**). A comparison of results of **Figure 7a** and **b**, indicates that the damaged outside surfaces are highly smoother at the mentioned sliding conditions and separation of matrix is very much limited with the content of organically modified montmorillonite nanoclay particles. **Figure 8** indicates shape and size of the wear debris

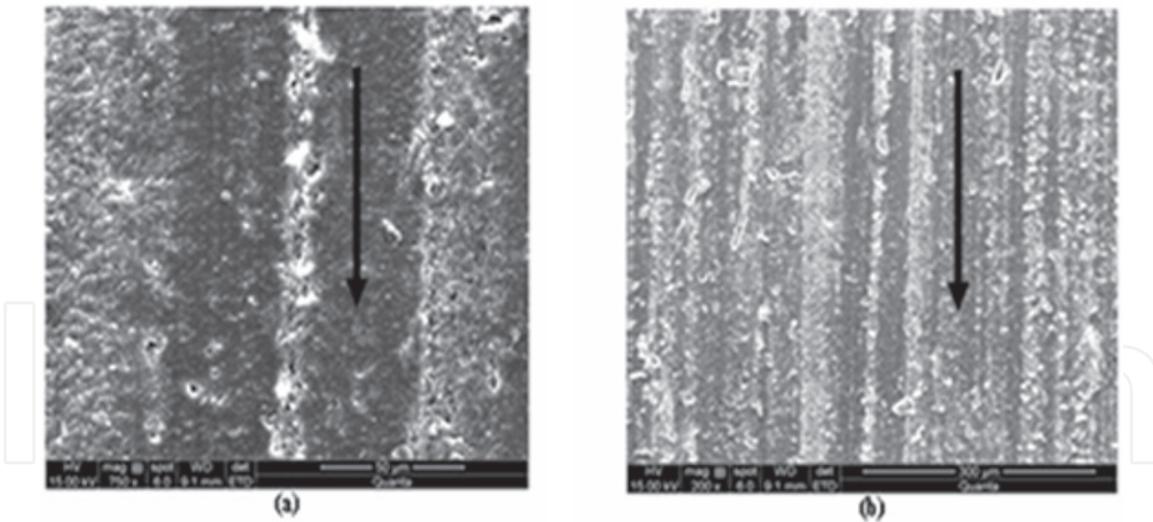


Figure 7. Scanning electron micrograph of pure epoxy at 30 N, 1 m/s. (a) Lower magnification and (b) higher magnification.

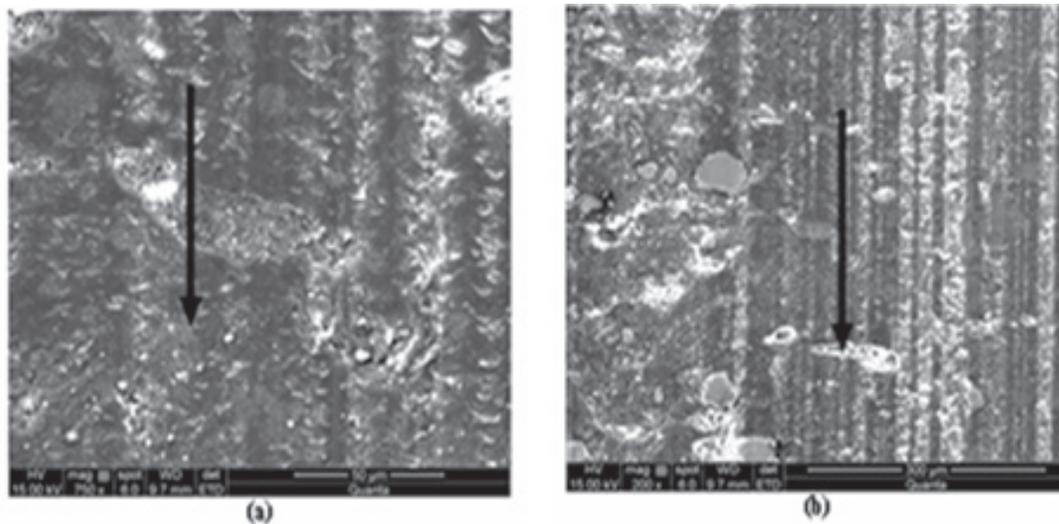


Figure 8. Scanning electron micrograph of 5 wt.% oMMT filled epoxy at 30 N, 1 m/s. (a) Lower magnification and (b) higher magnification.

and debris shape changes for organically modified nanoclay composite. The shape of the debris changes to spherical and finer in comparison to that seen in **Figure 7** when the nanofillers were not present. These small or fine particles build up marks of dense material as seen in **Figure 8a** and **b**. Enlarged magnification (**Figure 8b**) shows the makeup of the packed material formed by addition of the wear debris.

The surface worn is comparatively soft and illustrated by surface damage due to fatigue, usually taken places at elevated temperatures and eliminates the surface deposit by microcracks. A space consists of worn out particle are held jointly by thermal and mechanical processes implicated in sliding, particularly roughing due to frictional heat, and compression due to the load applied. Consequently, due to the adding up of Organically Modified Montmorillonite nanoclay (oMMT) nanoparticles, outside surface reliability is preserved in the polymer matrix with a steady process of wear is taking place even at elevated load and velocity situations and thus guide to an increased load bearing capability of the nanocomposite. In addition, for nanocomposite samples, the width of traces of the wear for the nanocomposites throughout the process of sliding was considered using scanning electron microscope.

The highest and lowest trace width for the pristine Epoxy resin is 50 and 25.35 μm correspondingly. In the case of, 5 wt.% of organically modified Monmorillonite nanoclay (oMMT) filled Epoxy nanocomposite, the width of the trace is 27.69–18.46 μm respectively. It was accomplished from the width of the trace that organically modified Monmorillonite nanoclay (oMMT) filled Epoxy nanocomposite has the smallest width of trace and consequently observed that improved resistance to wear.

2.7 Characteristics of Wear by analysis using Taguchi method

The research results were studied by Taguchi method and important factors affecting rate of wear are identified. The results of the Taguchi analysis are listed in Table 4 and in Figure 9.

Test run	Velocity (A) m/s	Load (B) N	Filler content (C) %	Distance (D) (m)	$K_s (\text{mm}^3/\text{Nm}) \times 10^{-5}$	S/N ratio (db)
1	0.5	10	0	1000	3.41	89.34
2	0.5	20	2	2000	2.97	90.54
3	0.5	30	5	3000	4.70	106.55
4	1.0	10	2	3000	2.00	94.00
5	1.0	20	5	1000	2.54	91.90
6	1.0	30	0	2000	8.40	101.51
7	1.5	10	5	2000	2.99	90.48
8	1.5	20	0	3000	8.50	101.41
9	1.5	30	2	1000	4.51	86.91

Table 4. Experimental design (L_9 orthogonal array) with output and S/N ratio.

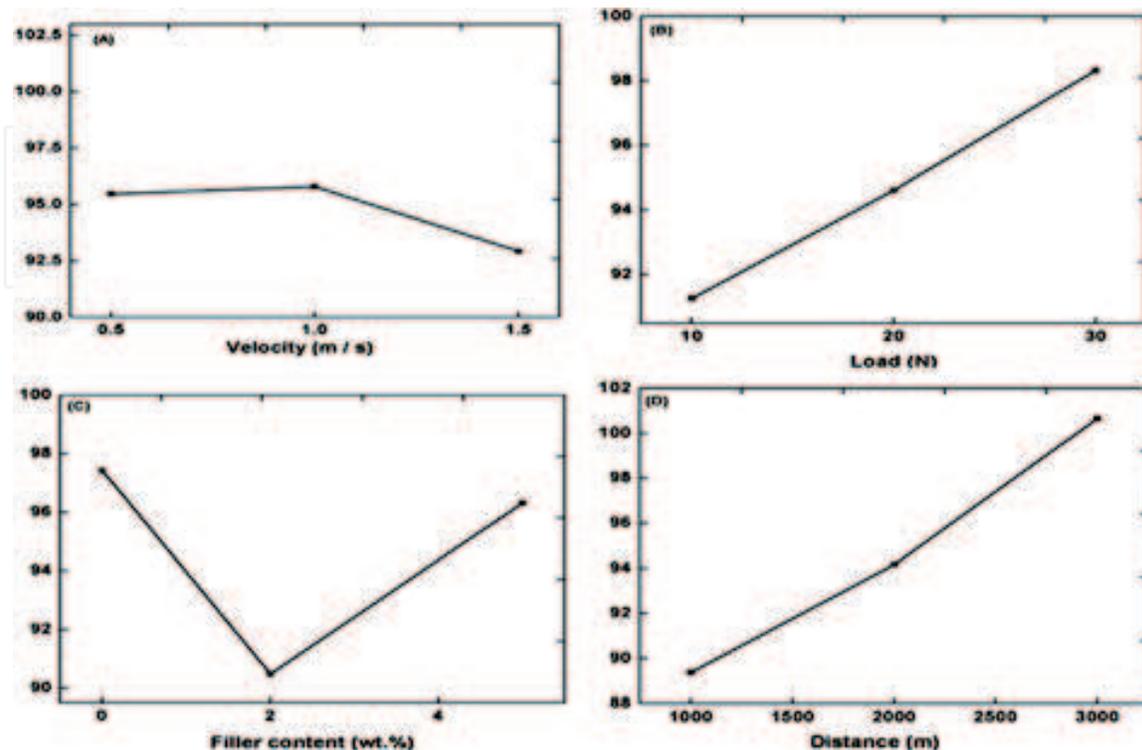


Figure 9. Effect of control factors on specific wear rate of epoxy-oMMT nanocomposites.

Level	(A) Velocity	(B) Load	(C) Filler content	(D) Distance
1	95.48	91.27	97.42	89.94
2	95.80	94.62	90.48	94.18
3	92.94	98.33	96.32	100.65
Delta	2.86	7.05	6.94	11.26
Rank	4	2	3	1

Table 5.
Response table for signal to noise ratios.

From **Table 4** the overall mean for the S/N ratio of the wear rate is found to be 85.264 db. **Figure 9** shows the effect of the three control parameters on particular rate of wear. The analysis was made using MINITAB 15, a popular software tool particularly used for plan of experimental applications.

The probable interactions among the control parameters have to be taken into account before any effort is made to utilize this model as a predictor tool for the monitoring of performance.

The S/N ratio result are listed in **Table 5**, it is concluded from table that amid every single one of the parameters, sliding distance is mainly important factor subsequently normal load and content of the filler while the velocity of the sliding has the smallest amount to the rate of the wear of the organically modified montmorillonite nanoclay (oMMT) added nanocomposite.

Investigation of the outcomes guides to the conclusion that combination of factors A_2 , B_3 , C_1 and D_3 gives minimum specific wear rate as shown in **Figure 9**.

3. Conclusions

This study shows that a fair degree of success has been achieved in production of pristine base Epoxy resin and organically modified nanoclay (oMMT) and TiO_2 added base Epoxy resin and Vinyl ester nanocomposites. The mechanical and tribological properties of the pristine Epoxy and organically modified nanoclay (oMMT) and Titania (TiO_2) added nanocomposites have been analyzed by series of tests and also by means of Taguchi's design of experiment technique. The following conclusions were drawn from the research work:

- i. Incorporation of nanofillers modifies the tensile and flexural strengths of the composites both for Epoxy as well as for Vinyl ester matrix.
- ii. The mechanical strength, modulus, hardness and physical density of the epoxy based nanocomposites are very much influenced by the nano filler type and content.
- iii. The nanoparticles filled Epoxy composites possess higher hardness values as compared to the Vinyl ester-oMMT nanocomposites.
- iv. The tensile strength of the Epoxy-oMMT, Epoxy- TiO_2 and Vinyl ester-oMMT nanocomposites decreases with increase in oMMT and TiO_2 content, whereas, the hardness and modulus increased with filler loading into the matrix.
- v. The oMMT filler has the ability to replace TiO_2 in some applications where high load bearing capabilities are not required.

- vi. Fabrication of polymer nanocomposite of specific requirements, there is a need for the choice of appropriate nanofiller material for optimizing the properties of the composite system.
- vii. The tribological results of pristine Epoxy and organically modified nanoclay (oMMT) filled nanocomposites are in the arranged susequently: Epoxy < Epoxy-7 wt.% oMMT < Epoxy-2 wt.% oMMT < Epoxy-5 wt.% oMMT. From the assessment of deviation in specific wear loss and wear rate, with larger sliding space, the abrasive wear mechanisms can control the interfacial interaction between the surfaces in contact. In this state, wear resistance of pristine polymer possibly is improved by adding the epoxy with organically modified nanoclay (oMMT) nanofiller.
- viii. Evaluation are conducted on the wear behavior, where 5 wt.% of organix-ally modified nanoclay (oMMT) added polymer nanocomposites samples illustrate enhanced results in contrast with pristine polymer Epoxy and other samples with higher filler content.
- ix. Dry sliding wear result of nanocomposites over different loads and different velocities can be effectively studied using Taguchi's experimental design technique. Taguchi's technique gives easy, organized and proficient technique for deciding the factors of control.
- x. Sliding distance come out as the important factor which affects rate of wear of Organically modified nanoclay (oMMT) filled nanocomposites, other parameters such as specific load, nanofiller content and sliding distance and velocity have taken part in a considerable position in finding the specific wear rate (K_s).

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