

Improved mechanical and tribological behaviour of peepal–okra–madar fiber hybrid composites reinforced with TiO₂ nanoparticles

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Abstract. Hybridization of natural fiber composite is on the rise to improve the performance of the materials by the synergistic effect of multiple fibres. The wear resistance of hybrid composite has greatly been enhanced by the addition of filler materials. This paper will look at the mechanical and wear properties of a special hybrid composite reinforced with 6 wt. percent of Peepal Fiber (PF), and different weight percentages of Okra Fiber (OF) and Madar Fiber (MF). The artificial specimens were evaluated with mechanical characterizations such as hardness, impact resistance, flexural strength, interlaminar shear strength, and tensile strength. As a filler material to improve the tribological performance, different weight percentages (2, 4, 6 and 8 wt. %) of Titanium Dioxide (TiO₂) nano powder were added. The mechanical properties of the filler-free composite samples that were composed of 15 wt. % Okra and 15 wt. % Madar fibers were better compared to those of the other samples. Furthermore, out of all the configurations studied, the sample with 6 wt. % TiO₂ filler showed the lowest wear rate, which is a significant increase in the wear resistance. This paper highlights the potential of TiO₂ reinforced hybrid natural fiber composite in sophisticated structural and abrasive applications.

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1 Introduction

Natural fiber reinforced composites (NFRCs) are currently receiving a lot of attention in recent years as sustainable alternatives to traditional engineering materials. Their low density, biodegradability, corrosion resistance and positive strength-to-weight ratio make them suitable in the application in the automotive, aerospace and structural components [1]. Nevertheless, the extensive use of NFRCs remains insufficient because of the variable properties of fibers, methods of processing, and limited knowledge about the optimal reinforcement designs. The natural fibers are mainly made up of lignocellulosic constituents; cellulose, hemicellulose, and lignin, which determine the mechanical performance of the fibers, as well as their interactions with the polymer matrices [2]. The improvement of composite properties by the introduction of natural fibers and nano-fillers has been investigated in several studies. Indicatively, addition of peepal fiber with nano-scale fillers has been established to enhance mechanical as well as thermal properties of epoxy-based composites [3]. Likewise, okra fiber reinforced systems have shown a significant enhancement in tensile and flexural strength when mixed with appropriate nano-fillers and the appropriate processing conditions [4]. Hybrid constructions with okra fiber combined with other materials have also been shown to have better fatigue resistance and thermo-mechanical performance, which makes them suitable in terms of high-load applications [5]. In addition to structural applications, natural fiber derivatives have been considered in multifunctional applications, such as sensing and lubrication, due to their unique physicochemical properties [6,7]. Okra has been discovered among other natural fibers as a promising reinforcement material that has a relatively high tensile strength and eco-friendly nature [8]. Similarly, madar fiber-based composites have demonstrated an enhancement in mechanical strength and thermal stability, when used with appropriate fillers, which could be used in the development of sustainable composites [9, 10]. Though these studies indicate the prospects of an individual fibers composites or a simple combination of hybrids, most of the current research is confined to individual fibers composites or a simple blend of hybrids. The synergistic properties of several natural fibers, especially peepal, okra, and madar have not been studied thoroughly. Moreover, little is known about the effects that nano-fillers like titanium dioxide (TiO_2) have on mechanical and tribological properties when they are used in multi-fiber hybrid systems. Besides that, tribological performance, particularly wear resistance at different loading conditions is also an area that is not fully investigated in natural fibre composites. The adhesion of fiber reinforcement, matrix adhesion, and nano-filler dispersion are important factors that can be used to determine the durability and long term performance of such materials. Nevertheless, the optimization of such parameters has yet to be properly defined. To fill these gaps, the current research is centered on the creation of a hybrid composite system that is reinforced with peepal, okra and madar fibers in different proportions. Titanium dioxide (TiO_2) nanoparticles are proposed as an effective filler to improve wear resistance and mechanical properties. The aim is to systematically test the impact of fiber composition and filler content on tensile strength, flexural behavior, impact resistance, interlaminar shear strength, and wear characteristics. This work would help to gain a more thorough insight into the concept of multi-fiber hybridization and nano-filler reinforcement to improve the sustainable and high-performance composite materials in the engineering industry.

2 Methodology

2.1 Materials

The composite system was made using three different natural fibers, which are peepal (*Ficus religiosa*), okra and madar (*Calotropis gigantea*), all of which fall under the lignocellulosic fiber categories. These fibres are mainly made of cellulose, hemicellulose and lignin which make them stronger in terms of mechanics and biodegradability. Peepal fiber has a fairly balanced structure of the structural constituents, which allow good stiffness and moderate flexibility. Okra fiber, a compound derived by means of the stem of the Malvaceae plant family is characterized by a higher cellulose content, and thus appropriate in reinforcing biodegradable composites. Madar fiber which is extracted in *Calotropis gigantea* bark has a relatively higher tensile strength and has a role in enhancing load-bearing capacity. The natural fibers that were utilized in this research were obtained in Fiber Source Enterprises, Chennai, Tamil Nadu, India. The following table offers a brief description of the underlying properties of the materials used as reinforcement that are taken into account in the current work. The choice of epoxy resin (LY 556) is based on the fact that the material possesses high mechanical strength, good adhesion properties, and environmentally resistant environment. The resin system guarantees that there is good transfer of stress among the fibers and the matrix. Epoxy and hardener were purchased in Kovai Cheenu & Company, Coimbatore, Tamil Nadu, India. The filler material was titanium dioxide (TiO₂) nanoparticles with an average particle size of about 50 nm, which was sourced as filler material in Matrix Enterprises, Nagercoil, Tamil Nadu, India.

2.2 Composite Preparation

The hybrid composite laminates were made by compression molding technique and hand lay-up process. The surface of the mold was first washed and coated with a releasing agent that was easily removable to allow easy removal of the fabricated specimens. The first thing that was applied to the mold was a uniform layer of epoxy resin with a thickness of about 2 mm. Then, measured amounts of peepal, okra and madar fibers were applied evenly over the resin layer so as to reinforce the resin evenly. Fig. 1 shows the fabrication process of the composite samples.

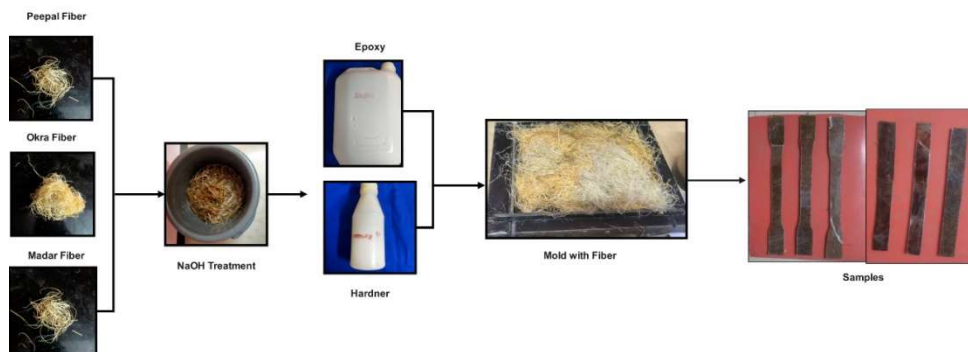


Fig. 1. Fabrication of composite samples using hand lay-up technique with controlled fiber loading, uniform dispersion of fillers, and room-temperature curing conditions.

This was followed by application of a second layer of resin in order to completely encapsulate the fibers and lastly, gentle rolling was done to reduce air entrapment and enhance fiber wetting. The ready laminate was compressed with the pressure of 3 MPa using a hydraulic press. Curing was done at room temperature during a period of 24 hours under constant pressure so as to ensure proper consolidation and bonding. Upon curing the composite plates were demolded very carefully and cut into standard specimen sizes to undergo additional mechanical and tribological tests. The fabrication process also provided uniform distribution of fibres and a uniform dispersion of TiO₂ nanoparticles in the matrix.

2.3 Mechanical Properties of the Composite without TiO₂Nano Particles

Mechanical behavior of the fabricated composites was measured based on the accepted ASTM standards. Tensile testing was done according to the specifications ASTM D3039/D3039M with the help of universal testing machine. Specimens of dimensions 250 mm x 25 mm x 3 mm with a gauge length of 150 mm were tested until failure at a constant crosshead speed of 2 mm/min. The flexural properties were tested based on ASTM D790 standards using three-point bending test. The test specimens, which are 150 mm x 12.5 mm x 3 mm, were held between a span of 100 mm and a central load of 20 kN load cell on a constant crosshead speed of 2 mm/min. Interlaminar shear strength (ILSS) was measured to determine the bonding strength of the layers of composite. The ILSS values were determined using the applied load, and specimen dimensions with consideration of the relationship between shear stress and cross-sectional area.

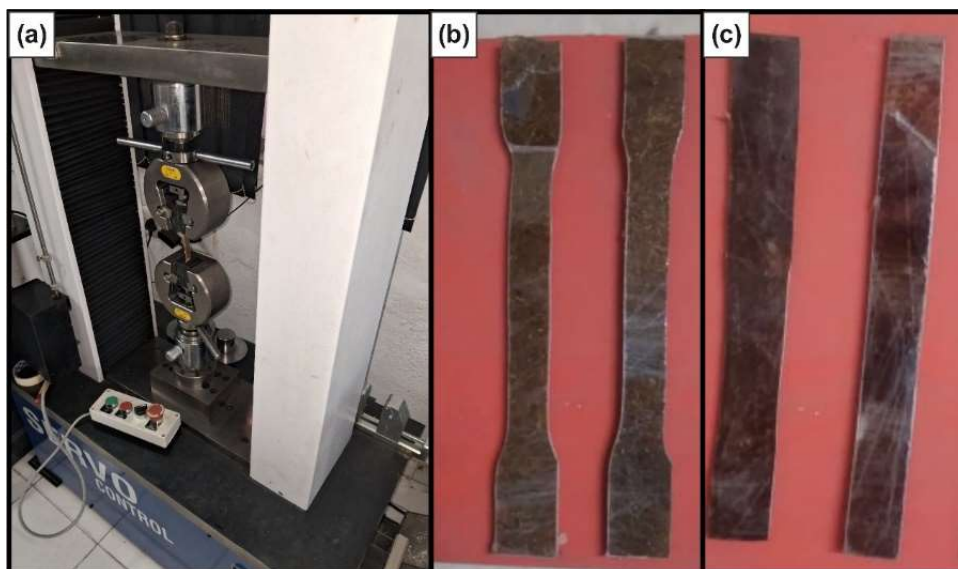


Fig. 2. (a) Universal testing machine under controlled loading conditions(b) ASTM D3039/D3039M tensile specimen tested at constant crosshead speed(c) ASTM D790 flexural specimen under three-point bending test.

A resistance to impact was determined by the Izod impact testing method as per ASTM D256. A series of tests with pendulum impact tester to identify the energy absorption capacity were conducted on specimens of size 65 mm x 12.7 mm x 3 mm with a notch angle of 2 mm of 45 o. Using the equation below (1), the ILSS can be calculated:

$$ILSS = \frac{3F}{4BT} \quad (1)$$

The measurement of hardness was done according to the ASTM D2240 standards using a Shore D durometer. This test measures the resistance of the composite surface to indentation and gives an indication of stiffness of the material used. In all mechanical tests, three specimens were tested in each configuration and the average values were reported in order to assure accuracy and repeatability.

2.4 Wear Behaviour of the Composites with TiO₂Nano Particles

According to the ASTM G99 standards, the wear behavior of the composite samples was studied with the help of a pin-on-disc tribometer. The experiment was carried out on dry sliding to determine the specific wear-rate and frictional properties of the composites. During testing, a hardened EN31 steel pin (69 HRC hardness, 8 mm diameter) was used as the counterface material. The speed at which the disc was rotated was 600 rpm with a track radius of 50 mm. All the experiments had a constant sliding distance of 1200 m to maintain consistency. Applied loads were changed with 15 N, 20 N and 25 N to examine the impact of loading conditions on wear performance. The frictional force that was generated when sliding was continuously measured using a digital data acquisition system that was in-built with the tribometer. The special wear rates were determined using equation (2).

$$W_{sp} = \frac{\Delta V}{NS} \quad (2)$$

In this case, ΔV represents the material lost (mm³), N represents the applied load (N), and S represents the distance traveled by the material (m). Fig. 3(a) and Fig. 3(b) respectively represent the pin on disc setting and wear test samples respectively.

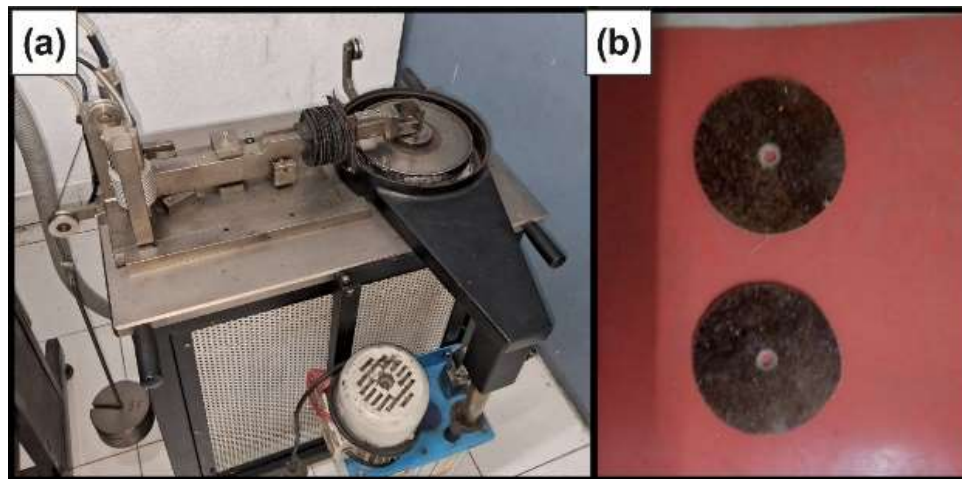


Fig. 3. (a) Pin-on-disc setup showing controlled load, sliding speed, and distance under dry sliding wear conditions (b) Wear test specimens prepared and tested under identical loading and sliding conditions.

Wear loss was calculated by recording the difference in weight of specimen before and after testing. The wear rate was determined using the loss in volume, force exerted, and the distance covered. The experimental design provided the control of the testing conditions and the possibility of accurate evaluation of tribological performance in various composite configurations.

It was anticipated that incorporation of TiO₂ nanoparticles would affect wear resistance by enhancing interfacial bonding, minimizing surface damage and redistributing loads during sliding contact.

3 Result and Discussions

3.1 Mechanical Properties of the Composites

Mechanical response of fabricated PF/OF/MF composites indicates that the mechanical response is strongly dependent on the fiber composition and the interfacial interaction. As shown in the tensile behavior, performance improves with an increase in the contents of okra fibers up to an optimum level, beyond which there is a decline in the performance. Sample S3 had the highest tensile strength (52.12 MPa) and better elongation properties compared to all samples. This improvement is due to the balanced ratio of okra and madar fibers which enhances efficient transfer of stress and minimizes the concentration of stress in the matrix. Conversely, samples that had either too much okra fiber or had too little madar content experienced a loss in tensile strength. This behavior can be interpreted as the madar fiber causing large contribution to the load-bearing capacity because it has higher intrinsic strength and thus excessive content of okra may weaken the interfacial bonding. The observed trend indicates the significance of having an ideal hybrid ratio in order to get a better mechanical performance. The same trend is followed in flexural properties. Flexural strength rises with fiber reinforcement to a maximum of 139.78 MPa which is the maximum value of flexural strength. This is linked with an increase in crack initiation and propagation resistance under bending loads. This is due to the uniform distribution of fibers in S3 leading to improved distribution of loads across the composite cross-section. Nonetheless, any further enhancement in the okra fiber content leads to a decreased flexural strength, probably because of a lack of adhesion between the matrix and the okra fiber, and an increase in the agglomeration of the okra fibers.

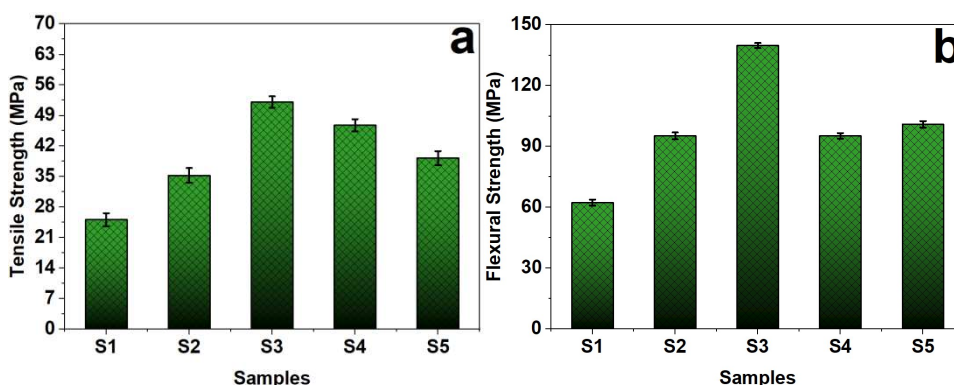


Fig. 4. (a) Tensile strength of composites without TiO₂ nanoparticles, measured under constant crosshead speed as per ASTM D3039/D3039M standard (b) Flexural strength of composites without TiO₂ nanoparticles, evaluated using three-point bending as per ASTM D790 standard.

Above the content of 17% madar fiber (MF) content, a decline in flexural strength is observed which may be attributed to poor interfacial adhesion between the fiber and the epoxy matrix. The change in flexural strength between PF/OF/MF composites (Fig. 4(b)) demonstrates the effect of fiber proportion on bending performance. Sample S3 out of all the configurations show a remarkable increase with about 122 percent increase in flexural

strength as compared to the composition at the baseline. This increased performance could be attributed to its increased resistance to crack initiation and propagation, which is caused by a balanced fiber distribution. The same can be said to the interlaminar shear strength (ILSS) results depicted in Fig. 5(a), where the variation is very similar to the flexural strength. The ILSS values of the composite samples vary with S3 having better interfacial bonding and load transfer efficiency. Conversely, sample S4 and S5 have lower ILSS, which means that the fibers and the matrix do not adhere to each other as well, which negatively impacts the stress distribution in the composite layers. On the whole, the mechanical performance of the developed NFRCs is the best in sample S3, where okra fiber (OF) and madar fiber (MF) are observed in almost equal amounts. This balanced structure increases fiber-matrix interface, which results in an increase in strength properties. With either of the two contents (OF and MF content) disproportionately higher in other samples, the mechanical properties are likely to be reduced because of the inefficient bonding and transmission of stress. The anisotropic nature of the composites can also be linked to the observed differences in the performance. The fibers are randomly oriented and therefore when there is a variation in the fibers in terms of their alignment and distribution, the variation will be felt in the load bearing behavior. This is the reason sample S5 is slightly better than S4 although they exhibit similar composition trends because local fiber orientation can have some contribution towards improved stress resistance in some directions.

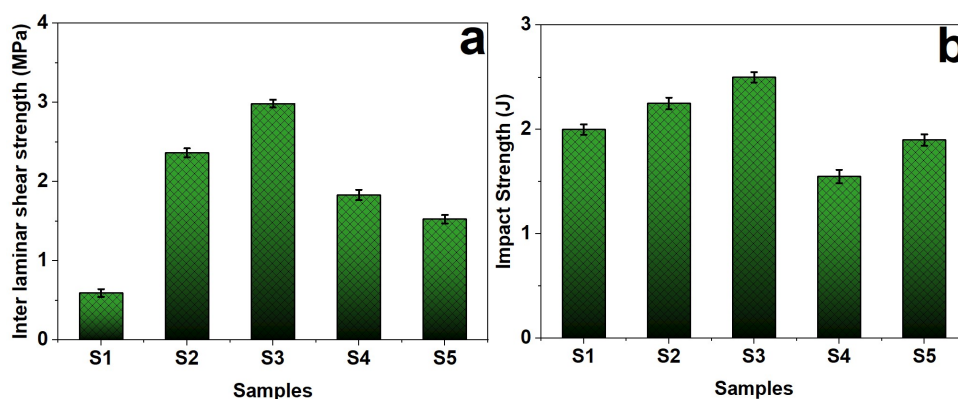


Fig. 5. (a) Interlaminar shear strength of composites without TiO₂ nanoparticles, evaluated using short-beam shear test as per ASTM D256 standard (b) Impact strength of composites without TiO₂ nanoparticles, measured using Izod method under standard impact testing conditions.

Fig. 5(b) depicts the behavior of the PF/OF/MF fiber-reinforced composites on the behavior of the impact strength under controlled loading conditions. The results indicate that the energy absorption capability varies significantly with fiber composition. Specifically, sample S4 shows a pronounced decrease in impact resistance, implying behavior of brittle fracture and low capability to dissipate applied energy. In samples S1, S2 and S3, where okra fiber (OF) and madar fiber (MF) are present in similar proportions, the impact strength takes moderate values, ranging between around 2 J to around 2.5 J. This difference substantiates the fact that the hybridization of fibers affects the toughness properties of the composites. All in all, the variation in the impact performance in Fig. 5(b) represents that impact performance is sensitive to the distribution of fibers and the bonding between the interfaces. Similar observations have been made in previous reports where hybrid composite using natural fibers and bio-fillers were found to have better impact resistance. As an example, composites reinforced with peepal and Indian almond fibers and 7.5 wt.%

jujube seed filler was found to be improved in terms of mechanical performance with an impact strength of about 5.2 kJ/m² indicating their suitability in sustainable engineering applications [15].

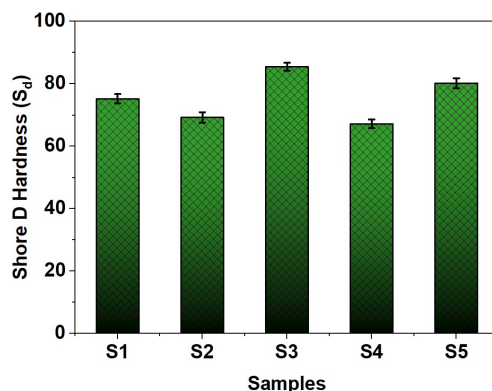


Fig. 6. Shore hardness of composites without TiO₂ nanoparticles, measured using Shore D durometer under standard load and ambient laboratory conditions as per ASTM D2240.

Interlaminar shear strength (ILSS) outcomes is another evidence that supports the significance of fiber–matrix interaction. S3 records the highest ILSS, which means that there is a strong interfacial bond and an enhanced delamination resistance. Samples S4 and S5 have relatively low ILSS values, implying a weak adhesion and poor stress transfer between layers. This decrease can probably be explained by the unbalanced distribution of fibers and a higher void content in these designs. The results of impact strength indicate that the ability of fiber to absorb energy is also dependent on the fiber composition. The S3 sample exhibits the best impact resistance over any of the other configurations and hence the best toughness and ability to arrest cracks. When fiber proportions are imbalanced in samples, low impact strength is exhibited because of poor stress dissipation and brittle fracture behavior. The measurements of hardness in all samples indicate that there is a comparably small difference. It should come as no surprise because hardness is largely controlled by the epoxy matrix and not the fiber composition. Because the content of the matrix is the same in all configurations the hardness values tend to be more similar, that is, surface resistance to indentation is constant.

3.2 Specific Wear Rate and Frictional Force

The tribological behaviour of the composites was measured by examining the frictional force and specific wear rate under different load conditions. The findings suggest that both parameters are greatly affected by applied load and the incorporation of TiO₂ nanoparticles. All samples increase their frictional force with an increase in applied load, which is also expected since the real contact area between the sliding surfaces increases with increasing applied load. Nonetheless, the relatively stable frictional properties of composites reinforced with TiO₂ nanoparticles can be explained by relative stability. Out of the tested configurations, sample W, Y and Z exhibit a relatively lower frictional variation which implies that the surfaces are more stable during the sliding process. Presence of TiO₂ nanoparticles is important towards adjusting the surface properties of the composites. These nanoparticles are micro-scale load bearing components and less direct contact between the surface of the composite and the counterface is reduced by these nanoparticles and thus the

variation in frictional fluctuations is minimized. By contrast, filler-free composite (S3) exhibits a stronger augmentation in friction under heavier loads because of direct exposure to the matrix and augmented reduction in the surface. Specific wear rate analysis shows that there is an apparent enhancement of wear resistance upon incorporation of TiO₂ nanoparticles. The filler-free sample (S3) has the highest wear rate in all loading conditions which confirm the susceptibility of the matrix-dominated surface to material loss. Contrarily, wear rates of composite materials that incorporate TiO₂ are found to be much lower, and thus durability is also improved. The composite with 6 wt.% TiO₂ (sample Y) shows the most promising wear characteristics. This optimum concentration ensures a uniform spread of nanoparticles in the matrix resulting in enhanced interfacial bonding and decreased removal of material during sliding. The nanoparticles also help in a protective layer at the contact interface which reduces abrasive wear and stabilizes the sliding mechanism. With increased concentrations of fillers (e.g., 8 wt.% TiO₂), there is a slight decrease in performance. This is due to the agglomeration of nanoparticles that contributes to the creation of stress concentration points and weak interfacial regions. Consequently, the wear resistance is not further enhanced but can even deteriorate when subjected to greater loads.

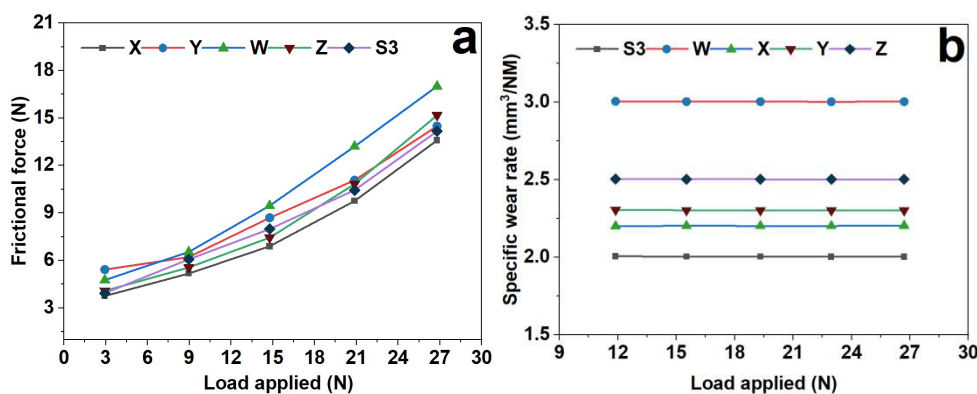


Fig. 7. (a) Frictional force of TiO₂-reinforced composites under varying applied loads, measured using pin-on-disc setup under dry sliding conditions. (b) Specific wear rate of TiO₂-reinforced composites at different applied loads, evaluated under controlled speed and sliding distance conditions.

The wear mechanisms observed indicate that TiO₂ nanoparticles increase mechanical interlocking between fibers and matrix, improve load-distribution, and serve as barriers to crack-propagation. There are also enhancements in wettability and a decrease in the formation of voids, which contribute to overall stability of the composite in the process of tribological interaction. The overall findings reveal that both fiber hybridization and nano-filler incorporation are key factors that can be used to establish the mechanical and tribological capabilities of the composites. The optimized setup (S3 6 wt.% TiO₂) offers a balanced solution of strength, toughness, and wear resistance and can be used in structural applications that are subjected to mechanical loading and surface wear.

4 Conclusions

This research conducted an inquiry into the mechanical and tribological performance of hybrid natural fiber composites which are reinforced with peepal, okra and madar fibres using epoxy as a matrix. The effects of fiber composition and incorporation of titanium

dioxide (TiO₂) nanoparticles on composite behavior were systematically investigated. The findings prove that fiber hybridization is significant in the improvement of mechanical characteristics. The composite with balanced proportions of okra and madar fibers (sample S3) had better performance as it achieved tensile strength of 52.12 MPa, flexural strength of 139.78 MPa, and enhanced interlaminar shear and impact properties. The main factors that have led to this improvement include the ability to transfer stress, enhanced interfacial bonding, and crack propagation resistance. On the contrary, the performance was low because of the poor adhesion and the inefficient distribution of the loads when the fiber ratio was not optimal. The addition of TiO₂ nanoparticles considerably enhanced tribological performance of the composites. There was a significant decrease in wear rate in the samples which were reinforced with nanoparticles than the filler-free composite. One of the configurations tested was the composite with 6 wt.% of TiO₂, which showed the highest wear resistance, showing an ideal balance between particle dispersion and interfacial interaction. Filler concentration resulted in minor performance losses, probably because of the agglomeration of nanoparticles and the development of local defects. In general, the synergistic effect of optimized fiber hybridization and nano-filler reinforcement led to the development of composites characterized by an increase in the mechanical strength of the composite and, at the same time, improved wear resistance. The hybrid composite system of PF/OF/MF/TiO₂ developed has a high potential of being used in structural and tribological applications where durability and sustainability is paramount. Future research can be aimed at improving the methods of dispersion of nanoparticles, new methods of treating the surface of fibers, and a long-term study of the workability of such composites under various environmental conditions.

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