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## Mechanical and Tribological Behavior of Vinyl Ester Hybrid Composites

P.A. Udaya Kumar<sup>a</sup>, Ramalingaiah<sup>b</sup>, B. Suresha<sup>c</sup>, R. Hemanth<sup>d</sup>

<sup>a</sup> Department of Mechanical Engineering, Sri Revana Siddeshwara Institute of Technology, Bangalore North, Bengaluru-562157, India,

<sup>b</sup> Cauvery Institute of Technology, Sundahalli, Siddaiahnakoppalu Gate, Mandya-571402, India,

<sup>c</sup> Department of Mechanical Engineering, The National Institute of Engineering, Mysuru- 570008, India,

<sup>d</sup> Department of Mechanical Engineering, NIE Institute of Technology, Mysuru- 570018, India.

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#### Corresponding author:

B. Suresha Department of Mechanical Engineering, The National Institute of Engineering, Mysuru- 570008, India. Email: sureshab@nie.ac.in

#### ABSTRACT

An attempt has been made to explore the effect of hybrid reinforcement on mechanical properties and dry sliding wear behavior of vinyl ester (VE) composites. Vinyl ester reinforced with alkali treated coir fibers (CF-VE), silanated aramid fibers (AFs) leading to fibridization and inclusion of coconut shell powder (CSP) with various weight proportions has been prepared resulting in hybridization. Effectiveness of treated fibers and CSP on mechanical and wear behavior of VE and their hybrids was evaluated. The density and hardness values of CF-VE composite increased steadily with inclusion of AFs and CSP. The tensile strength increased from 52 MPa at 0 wt.% AFs inclusion to a maximum of 76 MPa at 15 and 5 wt.% of AFs and CSP content. Further, it increased to a maximum of 78 MPa at 5 and 10 wt.% of AFs and CSP in CF-VE composite. The specific wear rate decreased more rapidly with the CSP content. Hence, it is deduced that hybrid VE (15CFs+5AFs) composite with 10 wt.% CSP is more effective against sliding wear. The type, geometry and dispersion of reinforcements contribute to the increased wear resistance. These results are important for the development of new composites incorporated with surface treated reinforcing materials such as CF and CSP, which will have significant application in automotive sliding or bearing components.

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#### **1. INTRODUCTION**

Since three decades, natural fiber reinforced polymeric composites (NFRPCs) are emerging as promising alternatives to replace the glass fiber reinforced polymeric composites (GFRPCs). Natural fibers namely sisal, jute, coir, banana and pineapple have attracted the attention of materials scientists and technologists for application in consumer goods, low cost structural materials and automotive components [1-5]. Natural fibers can have different advantages over synthetic fibers like low density, cost and acceptable specific mechanical properties. Also, they are renewable and biodegradable. In the past, extensive research studies have been found on fabrication and characterization of thermosetting polymeric composites reinforced with different natural fibers with and without chemical treatment by different methods [6-9]. Many polymeric resins exhibit poor mechanical properties and therefore several research teams have explored the possibilities to improve these properties of polymers by reinforcing them with various treated fibers, particulate fillers and with the combination of both fibers and particulates resulting in hybridization [10-14].

Among various natural fibers, coir fiber has shown a great importance for use in many composite parts. However, the characteristics like poor wettability, poor fiber/matrix adhesion, intrinsic polarity due to the presence of hydroxyl and carboxyl groups in their structure, and low moisture resistance reduced the scope for their use in medium load bearing applications. To overcome these drawbacks, efforts have been made by several researchers to improve interfacial adhesion of natural fibers by physical and chemical treatments [15-17].

Coconut based materials seem to have attracted much attention as possible reinforcements /fillers through not only by the attempts to understand their characteristic, but also for their possible use as hybrid fibers resulting in synergism in terms of improved properties such as tensile properties and tribological behavior has not been adequately addressed so far. A gentle survey of literature shows that little attention has been focused on the tribological behavior of vinyl ester based composites with fibridization (coir and aramid) and CSP filled hybrid composites in particular.

The wear behavior of the composites is not only dictated by the fiber/filler loading but also on the operating conditions. On the other hand, tribo-properties are not intrinsic material properties, nevertheless strongly depend upon the system in which material functions. So the influence of fillers and/fibers on the tribological behavior of composites cannot be predicted in prior and has to be tested in the laboratory. In many industrial applications of polymer based composites, an understanding of tribological essential along behavior is with an understanding of the mechanical properties.

The main goal of this investigation is to evaluate the mechanical and tribological performance of the hybrid thermoset based polymer composite reinforced with surface treated fibers and suitable for fabrication with simple hand-layup method.

In view of the said facts, the present research work was undertaken to study the influence of potential reinforcements i.e. alkali treated coir fibers, silane treated CSP and aramid fibers on the hardness, tensile properties and dry sliding wear behavior of vinyl ester composites. The main objective of the study is to evaluate the role of silane treated CSP, alkali treated coir fibers and silane treated aramid fibers as reinforcements on friction and dry sliding wear behavior of vinyl ester matrix. Experiments were conducted against stainless steel using a pin-ondisk tribometer, considering different operating parameters such as sliding distance, applied normal load and sliding velocity.

## 2. EXPERIMENTAL DETAILS

## 2.1 Materials

Coir is a lingo cellulosic natural fiber and is a seed-hair fiber obtained from the husk of coconut, the fruit of a tropical plant of the palmae family. Brown coir fibers (CFs) obtained from Karnataka State Coir Development Corporation Ltd (KSCDC Ltd), Bengaluru, India were used in the present study. The intrinsic properties of CFs (suppliers data) used are given in Table 1. Coconut shell powder (CSP) of particle size  $< 50 \,\mu m$  was collected from Master Micron International, Bengaluru, India. The aramid fibers (AFs) (Kevlar 29-Type 964, density 1.45 g/cm<sup>3</sup>, diameter 12 µm, length 3 mm) used in this work were obtained from DuPont Company. Vinyl ester (VE) was used as the matrix material. Methyl ethyl ketone peroxide (MEKP) and cobalt naphthenate were used as curing catalyst and accelerator respectively. The above mentioned materials were supplied by M/s. S & S Polymers, Bengaluru, India. Trichlorovinyl silane and sodium hydroxide (NaOH) chemicals used for surface modification of CSP and CFs were obtained from M/s Sigma Aldrich, Bengaluru, India [4].

Fiber length (mm)	5-8
Fiber diameter (µm)	200 ±5
Color	Brown
Bulk density (g/cm <sup>3</sup> )	$1.1 \pm 0.1$
Ultimate tensile strength (MPa)	140 ±3
Young's Modulus (MPa)	3150 ±15
Elongation at break (%)	32.5 ±0.4

**Table 1**. Typical properties of coir fiber.

An alternative reinforcement in various polymers [18-20]. One of these is the coir fiber, which is the most used due to its excellent mechanical properties and availability (India, China and Sri Lanka). Two reasons for this could be that this fiber is an inexpensive among the natural fibers available in the world, besides being a strong, hard and stiff cellulosic fiber with high lignin content (> 30 wt.%) [21]. On the other hand, another coconut based material that has been extensively studied is the coconut shell powder (CSP) [22,23]. Most of the studies mentioned above have observed that the mechanical properties of polymer composite increased with increasing CSP content. Furthermore, there are some recently published reports of hybrid polymer based composites using CSP and other natural materials including coir fibers [24-28]. Vignesh et al. [24] carried out experiments using tensile and flexural tests of and coir fiber reinforced polyester CSP composites and found that the coir fiber predominates in enhancing the mechanical properties of CSP-polyester composites. Vinod Kumar and co-workers [25] concluded that the mechanical properties (tensile and impact strengths) were less affected by the addition of CSP filler. Kuburi et al. [26] and Bhaskar and Singh [27] concluded that the CSP and coir fibers are good reinforcing materials to reduce the consumption of resin as well as utilizing waste natural material without altering the properties required for load-bearing applications.

From the authors knowledge and from literature reviews [28-32], few reports are available on the usage of natural fibers and/fillers as reinforcement for tribological applications. Such polymer based composites can be used in many tribological applications such as bearings, sliders, machine beds and chute liners in power plants. In the recent work by Yusif and Tayeb [33], oil palm short fibers were used as reinforcement in polyester matrix for tribostudy. In this work, the authors concluded that introduction of oil palm fibers as reinforcement significantly reduced the specific wear rate. Recently, Nirmal et al. [34] investigated the effect of varying the loading of kenaf particles as reinforcement in epoxy matrix on the dry sliding friction and wear behavior. They found minimum wear scars with mild plastic deformation at higher loading situation. Further, they concluded that the formation of transfer films at higher loads was more significant, which would protect the composite from additional wear and thus reducing the wear rate. To obtain desired properties of a PMC, the the reinforcements and fillers are added to thermoset/thermoplastic matrices [35-37]. The synergistic improvements in mechanical and tribological properties in many cases are attained through the incorporation of glass or carbon fiber reinforcement and particulate fillers [38,39].

Aramid various polymeric resins epoxy, polyester and vinyl ester resins are the three most commonly used thermoset matrix in PMCs. With respect to vinyl ester based composites, some reports are available on both two-phase and hybrid composites with natural fibers and particulates (both organic and inorganic) as fillers. It was also observed by the authors that studies on vinyl ester reinforced with a filler and

## 2.2 Mould preparation

The fabrication of the various VE composites was carried out through hand lay-up procedure followed by compression moulding. The mould used for preparing VE composites was made from two rectangular chromium coated mild steel plates having dimensions of 250 mm × 300 mm. Four spacers were used to maintain a 3.5 mm thickness all around the mould plates. The role of these plates is to cover and compress the reinforcement after the VE is smeared. It also avoids the debris from entering into the composite slab during the curing process.

## 2.3 Chemical treatment

The coupling treatment is one of the important chemical treatment methods, which improves the interfacial adhesion between the matrix and the reinforcements/fillers. Accordingly, CFs was modified by NaOH. The coir fibers were washed with water and dried in an air circulated oven for 1 h at 70 °C. The dried coir fibers were treated with 2 wt.% NaOH solution in a water bath at room temperature for 2 h. Selected concentration of NaOH was based on earlier report [40]. The treated fibers were washed several times with fresh water until all the NaOH deposition over the fiber surface was removed. Then, the fibers were dried at room temperature for 1 h, followed by drying in a vacuum oven at 70 °C for about 1 h.

Coconut shell powder (CSP) was added slowly into the solution of trichlorovinyl silane of 0.5 wt. % solution dissolved in aqueous ethanol and stirred well for 1h for complete esterification. Silane aqueous solution was acidified to pH 3.5 with acetic acid as reported elsewhere [41]. Treated CSP was then filtered and dried in an oven at 80 °C for 2 h.

The surface of aramid fiber was treated with silane coupling agent to investigate the effect of improved adhesion on mechanical and tribological behaviour VE based hvbrid composites. Trichlorovinvl silane was prehydrolysed for 1 h in distilled water adjusted to pH 3.5 with acetic acid. The silane concentration was fixed at 0.5 wt.% and the aramid fibers were immersed in that solution for about 30 min. The silane treated fibers were dried in room temperature prior to use for about 24 h to ensure complete dry condition.

## 2.4 Fabrication of composites

For preparing the VE composites, two chromium plated tool steel molds of size 250 mm × 300 mm  $\times$  5 mm were used with hand lay-up technique. First CFs was cleaned to remove the pithy and other undesirable materials, if any, adhering to the surface of fibers. Then, the fibers were cut to uniform size of  $6 \pm 1$  mm long, which were subjected to chemical treatments. The fibers (coir) were then evenly arranged, in a mould measuring 250 mm × 300 mm × 5 mm and pressed into a mat. Then, it was wetted by pouring the resin mix solution with curing agents on to the fiber mat and squeezed using a roller to remove the air bubbles. The mould was then closed and cured at room temperature for 24 h under a constant pressure of 1 MPa. After the curing process, mechanical test samples were cut with a diamond cutter to the required dimensions following the ASTM standards.

Designation	Composition by wt. %	
CF-VE	VE (70) + CF (30)	
H1	VE (70) + CF (20) + AF (10)	
H2	VE (70) + CF (15) + AF (10) + CSP (5)	
H3	VE (70) + CF (15) + AF (5) + CSP (10)	

Similarly, other hybrid composites were fabricated with the required quantity in wt.% of fibers/fillers. Details of all the prepared VE composite slabs are listed in Table 2.

## 2.4 Hardness test

The hardness of all the prepared VE composites was determined according to ASTM E384 standard using a Leitz micro-hardness tester by applying a constant load of 50 gram-force for 30 seconds of dwell time. The indenter was made of diamond having right pyramid shape with square base and apex angle of 136°. Test specimens of size 25 mm x 25 mm x 3.5 mm were used to measure the hardness of all VE composite slabs prepared. The test specimens were held firmly in position and lens was arranged to get clear image at its focal length and finally, the indentation was made using set parameters. To obtain a statistically significant result for each condition, minimum five samples were used for hardness tests.

## 2.5 Tensile test

The tensile strength and the modulus of all VE composite slabs were determined according to ASTM D3039 standard following the test procedure as reported [41]. The test specimen with dimensions of 100 mm  $\times$  6 mm  $\times$  3 mm were subjected to tensile tests with a cross head speed of 2 mm/min at room temperature. To obtain a statistically significant result for each case, six samples from each set were tested to evaluate the tensile data.

## 2.6 Wear test

Dry sliding wear tests of prepared composite slabs are carried out using pin-on-disk apparatus, manufactured by Magnum Engineers, Bengaluru, India. The counter face is made of ground hardened steel disc EN-32, 64 HRC. The maximum diameter, thickness and surface roughness of disc are 150 mm, 06 mm and 0.5  $\mu$ respectively. Dry sliding wear test is performed as per ASTM G99-17 [42] standard. The test samples were glued to pins of size 8 mm × 8 mm × 25 mm length using suitable adhesive and their initial weights were recorded using high precision electronic balance after thorough cleaning. The sliding was performed under dry friction conditions at sliding distance ranging from 1000-3000 m, applied normal load of 15-60 N and sliding velocity varying from 1-3 m/s. Details of dry sliding wear tests and determination of specific wear rate have been presented elsewhere [42].

### 2.7 Fractography

Fractured samples of tensile test and worn surfaces of the composite samples subjected to wear were coated with a thin layer of gold and micro graphed using (E-SEM, Quanta 200 model, FEI-Netherland make) scanning electron microscope (SEM) with operational voltage of 20 kV, and the working distance was set to 6 mm. microscopic SEM helps in analysis and characterization of fibers/particulates on the basis of surface morphology and structural changes.

#### 3. RESULTS AND DISCUSSION

#### 3.1 Surface treatment of reinforcements

The surface topography of untreated and NaOH treated were shown in Figs. 1 and 2 respectively. The coir fiber is composed of spindle-shaped cells or ultimate fibers aligned along the fiber axis are as shown in Fig. 1a. There are some impurities on the outer surface of untreated CF. The surface of the fiber consists of circular pit openings and few well defined fibrillar pattern visible from the micrograph. Initially, the fiber surface will be covered with a layer of substances; like pectin, lignin, pores and other impurities (Fig. 1a). The fiber surface is not smooth and has nodes, irregular stripes and, fibrils. Figure 1b shows the typical cross section of the coir fiber, which is not completely circular. Fiber cells showing a layered spiral structure act as though they are composed of concentric helical springs of inextensible materials could be seen from Fig. 1b.

The treated coir fiber SEM micrographs in length direction and cross sectional views are shown in Figs. 2a and b respectively. After NaOH

treatment, most of the lignin, pectin and other impurities are removed resulting in rougher surface and also tiny voids are produced on the fiber surface (Fig. 2a).





**Fig. 1**. SEM pictures of untreated CF: (a) length direction; (b) cross section.





**Fig. 2**. SEM pictures of treated CF: (a) length direction; (b) cross section.

Treated CF with NaOH shows very clean from wax and pectin of CF. This could be attributed to the NaOH, which reacts with hydroxyl groups of the cementing material like cellulose, and it brings on the destruction of cellular structure and hence the fibers split into fine yarns. The tiny voids present in the fiber will increase the mechanical bonding between the matrix and fibers during composite slab preparation.

#### 3.2 Density and hardness

The density and hardness of short coir fiber reinforced vinvl ester (CF-VE) and their hybrid composites are summarized in Table 3. The bulk density of the VE based composites are 1.10 g/cm<sup>3</sup> for two phase composite (CF-VE), 1.13 g/cm<sup>3</sup> for VE hybrid (H1), 1.15 g/cm<sup>3</sup> for VE hybrid (H2), and 1.17 g/cm<sup>3</sup> for VE hybrid (H3) composites. It is possible to note that, as the amount of aramid fiber and CSP loading in CF-VE increased, the density of the hybrid composites are also increased. Further, it can be noticed that addition of higher wt.% of CSP (10 wt.%) filled hybrid composite (H3) increased the density by about 10 %. The results demonstrated that the fiber and filler dimensionality have dramatic influence on the density as well as hardness of CF-VE composite.

Table 3. Density and hardness of CF-VE composites.

Samples	Theoretical Density (g/cm³)	Measured Density (g/cm³)	Void fraction (%)	Hard- ness (HV)
CF-VE	1.13	1.10	1.77	13.20
H1	1.15	1.13	1.74	16.40
H2	1.17	1.15	1.71	17.30
H3	1.19	1.17	1.68	18.10

It can be seen from Table 3 that the hardness of two-phase CF-VE increases from 13.2 HV to 16.4 HV when it is filled with 10 wt.% of aramid fibers (H1). The slight increase in the hardness may be attributed to the higher strength and hardness of aramid fiber and also the uniform dispersion and better interfacial bonding between the treated AFs in the VE resin. The incorporation of 5 and 10 wt.% of CSP with hybrid fiber (H1) reinforcement had no significant increase in the hardness compared to CF-VE two phase composite. Addition of CSP and CFs/AFs augmented the hardness compared to that of 30 wt.% CFs in VE matrix alone. This is in agreement with earlier findings [43].

### 3.3 Mechanical properties

Mechanical properties of natural fiber/filler reinforced polymeric composites depend on several factors viz., inherent mechanical properties of reinforcements as well as matrix phase, loading of reinforcement, orientation and distribution of reinforcements and fiber/fillermatrix interfaces. Further, good control of the interface morphology of polymer based composites (PMCs) is one of the most critical parameters to impart the desired mechanical properties in multi-phase composite materials. The tensile strength, modulus and elongation at break of short coir fiber reinforced hybrid VE composites are tabulated in Table 4.

**Table 4**. Mechanical properties of CF-VE composites.

Specimen code	Tensile Strength (MPa)	Young's modulus (MPa)	Elongation at break (%)
CF-VE	58±1.6	2110±4.0	3.21
H1	72±1.4	2425±3.6	3.11
H2	76±1.4	2490±3.8	2.85
H3	78±1.3	2625±3.9	2.65

Figure 3a demonstrates the tensile test failed CF-VE and their hybrid composites. Figure 3b shows the mechanical properties such as tensile strength and modulus of VE based composite slabs prepared in this study. It can be seen that the tensile strength and elastic modulus of these composites increase with incorporation of small amount of silane treated aramid fibers as well as increase of CSP loading. Further, an improvement in tensile strength and modulus of CF-VE two-phase composite with the addition of AFs can be witnessed. The tensile strength of CF-VE increased from 58 MPa to 72 MPa and further

slight increase in values (76-78 MPa) for AFs and AFs with 5 and 10 wt.% of CSP loading. The modification of CF-VE with AFs and CSP, results in the increase in tensile strength and modulus. This is attributed to the higher strength, modulus and higher aspect ratio (1/d; 956) of AF combined with treated CSP, resulting in strengthening the interface of VE matrix and reinforcement materials. The maximum tensile strength for 10 wt.% CSP loaded composite (H3) was higher (78 MPa) compared to other VE based composite slabs.





**Fig. 3.** (a) Tensile test failed CF-VE and their hybrid composites, (b) Mechanical properties of CF-VE and their hybrid composites.

In order to improve the mechanical properties of natural fiber reinforced polymer composites (NFRPCs), several researchers have been considered alkali treatment as a better technique to modify the fiber surface to obtain improved bonding between the fiber and matrix [44-46]. Rout et al. [47] characterized the influence of fiber treatment on the mechanical properties of coir/polyester composites. They found 26 % improvement in tensile strength, 15 % in flexural strength and 20 % in impact strength after the coir fibers were treated with 2 % NaOH solution for 1 h. In the present work, the NaOH reacts with hydroxyl groups of the cementing material hemicellulose, and it brings on the destruction of the cellular structure and thereby the fibers split into filaments. The difference between the fibers before and after alkali treatment is exhibited in Figs. 1 and 2. It was observed that the filaments in the untreated fiber were packed together but got fibrillated after the alkali treatment. The fibrillation and tiny voids formed during alkali treatment on the coir fiber increases the effective surface area available for contact with the VE matrix and hence the interfacial adhesion was improved. Same observation on coir fiber has been reported by Rout et al. [47].

The modulus increases from 2110 MPa to 2425 MPa, 2490 MPa and 2625 MPa with the incorporation 15 wt.% AF into CF-VE matrix (H1) and 5 and 10 wt.% CSP based hybrid composites (H2 and H3) respectively. On the other hand, percentage elongation decreased from 3.21 to 2.65 for the CF-VE based hybrid composites. These observations reveals that incorporation of filler/fiber reinforcements increases strength and modulus of neat polymer while percentage elongation decreases. This is because the fibers have much higher strength and stiffness than those of the polymer matrices [48,49]. Another reason for the improvement in the strength and modulus of hybrid CF-VE composites may be due to the attainment of improved bonding between the fiber and filler with VE matrix. Furthermore, the fiber surface is treated with NaOH solution that formed a bridge of chemical bonds between fiber and VE matrix. Sodium hydroxide treatment improved the fiber-matrix interface. This treatment creates porous surface leading to a rough surface texture which allows coir fibers to reinforce strongly with VE matrix (Fig. 2). Further, the improved adhesion at treated filler (CSP)-matrix and treated fiber-matrix interface results in efficient load transfer from the matrix to filler/fiber reinforcement. The optimum hybrid reinforcement weight percentage varies with the nature of the fiber, particulate filler, matrix, fiber aspect ratio and interfacial bonding between the fiber/filler and matrix [50].

From the tensile behaviour of VE based hybrid composite materials, 30 wt.% reinforcement (15 wt.% CFs, 5 wt.% AFs and 10 wt.% CSP) was found to be the optimum for improved tensile strength as well as modulus. At the optimum fiber and filler weight percent, the load transfer from the fiber and filler to VE matrix was high. Thus, the hybrid composite (H3) exhibited highest tensile strength and modulus. Surface modification of fibers and CSP fillers improved the tensile properties by removing lignin and cellulose part that can be wet easily with the VE matrix. The tensile properties obtained in this work agree well with the reported tensile properties of sisal/coir fiber reinforced epoxy hybrid composites [51].

## 3.4 Effect of filler on coefficient of friction

Figure 4 illustrates the plots between the friction coefficients ( $\mu$ ) and the sliding distance at 15 N as applied load on different CF-VE based hybrid composites.



**Fig. 4**. Coefficient of friction of CF-VE and their hybrid composites.

The  $\mu$  increased from a low value to a nearly stabilized value with increase in sliding distance. The incorporation of CF in VE matrix increased the  $\mu$  as compared to VE hybrid composites. The value of  $\mu$  becomes stable after an initial running-in period. Addition of aramid fiber (AF) in VE matrix (H1) reduced the  $\mu$  of CF-VE composite as shown in Fig. 4. Further, the  $\mu$  decreased on increasing the content of CSP loading (H2 and H3) in VE based hybrid composites. In the initial sliding wear process, the specimen and the counterface are at room temperature. The temperature of the specimen increased due to frictional heat generated during wear process. The thermal softening of specimen with the applied load improved the surface contact between contacting pair. The increased surface contact area increased the u that was stabilized with further increase in sliding distance. The µ of two-phase CF-VE first showed an increasing trend from the 500 to 1500 m of sliding distance, with further increase in sliding distance decreased the  $\mu$  and then attains stability with increase in sliding distance up to 3000 m as shown in Fig. 4. From the above figure it is clear that the µ of CF-VE hybrid composites greatly depends on the filler and type of fiber content. Modification of CF-VE composite by treated AFs and CSP (H1, H2 and H3 composites) demonstrated lower friction coefficient values (Fig. 4). This was attributed to the development of transfer film, which provides an effective coating over the hard steel counterface asperities.

In general, larger surface area contact between polymer and counterface can give rise to a higher  $\mu$  during dry sliding. Therefore, the presence of fiber and filler (AFs and CSP) on the surface can lower the  $\mu$  of CF-VE composite by reducing a direct contact between the composite surface and steel counterface. It is therefore implicit that the addition of AF and CSP content decreases the  $\mu$  of the two-phase composite due to the reduced contact between the composite surface and steel counterface which promoted free rolling effect of AF debris in addition to lubricating effect of the fine coconut shell particles.

Table 5. Friction	coefficient for	CF-VE composites.
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Composites	Friction coefficient (μ)			
code	15 N	30 N	45 N	60 N
CF-VE	0.60	0.62	0.71	0.75
H1	0.56	0.59	0.55	0.54
H2	0.51	0.50	0.48	0.46
H3	0.46	0.44	0.43	0.41

Table 5 summarizes the effect of applied normal load on  $\mu$  of CF-VE and their hybrid composites. The  $\mu$  of CF-VE increased with the load and decreased for VE based hybrid composites (H3). It is well known that thermoset polymers on heating; flow and cross-link to form rigid material which do not melt or soften on further heating [52]. The softening combined with the

application of applied normal load improved the surface area contact between specimen and the steel counterface. The increased pressure further increases the contact area and consequently the value of u. Significantly higher  $\mu$  was found for CF-VE composites. The addition of fiber and filler (AF and CSP reinforcements) in the composite reduced the  $\mu$  as evident from Table 5. An increased sliding distance decreased the  $\mu$ , which was latter-on stabilized (Fig. 4). This may be attributed to the formation of transfer film of CSP and fiber debris on steel counterface to facilitate lowering the value of  $\mu$ .

# 3.5 Effects of sliding distance and applied load on specific wear rate

The tribological properties of thermoset polymer composites can be improved significantly by the incorporation of surface treated natural fillers/fiber as reinforcements. However, this is not true for all cases. Therefore, sliding wear tests for predicting the tribological behaviour are complex owing to numerous test parameters and environment conditions that can affect the tribological behaviour. The effect of sliding distance on specific wear rate (K<sub>s</sub>) of CF-VE and their hybrid composites is shown in Fig. 5.



Fig. 5. Variation of K<sub>s</sub> CF-VE composites at 15 N, 1 m/s.

From the above figure, it is evident that the  $K_s$  of CF-VE composites increases with increasing sliding distance. The lowest  $K_s$  was found in case of 10 wt. % CSP loading (H3 composite) and the  $K_s$  are steady up to 3000 m and thereafter increased abruptly. However, from the result, the  $K_s$  is initially high (at 500 and 1000 m) and

reduces considerably at a distance of 1500-2000 m. The debris formed during long run will clog in the asperities of the counterface of the steel disc thereby, reducing the  $K_s$ . The decrease in the  $K_s$  with increasing CSP loading (H2 and H3) can be attributed to the uniform dispersion of particles within the VE matrix and could give benefit of combining the properties of hybrid composite. Further, this could also be attributed to the surface modification of specimen as well as the steel counterface during the wear process.

To show the effect of applied load and sliding velocity on the Ks of CF-VE and their hybrid composites, results are extracted from the wear data at 3000 m sliding distance at three different velocities are presented in Figs. 6, 7 and 8 respectively. It is observed that the K<sub>s</sub> increase with the increase in the applied normal load, sliding velocity for CF-VE and their hybrid composites. However, the K<sub>s</sub> were lower than that of CF-VE composite compared to that of their hybrid composites (H1-H3). Figure 6 depicts that during sliding at low velocity the K<sub>s</sub> increases non-linearly with an increase in applied normal load for CF-VE and AF reinforced hybrid composites (CF-VE and H1). It is generally accepted that the wear behavior of short fiber reinforced polymeric composite is dominated by the process of matrix wear, wear of fiber and fiber peeling off, which typically occurs such as fiber thinning, fiber cracking, and fiber removal [54].



Fig. 6. Variation of K<sub>s</sub> CF-VE composites at 3000 m, 1 m/s.

Figures 7 and 8 show plots between the  $K_s$  and the applied load on CF-VE and their hybrid composites (H1, H2 and H3) at sliding velocity of

2 and 3 m/s respectively. The  $K_s$  shows linear increasing trend with increase in CSP loading. Further, the  $K_s$  increases with increase in load up to 45 N and further increase in load i.e. beyond 45 N, almost constant  $K_s$  was found for H2 and H3 composites (Figs. 7 and 8).



Fig. 7. Variation of  $K_s$  CF-VE composites at 3000 m, 2 m/s.



Fig. 8. Variation of  $K_s$  CF-VE composites at 3000 m, 3 m/s.

The two-phase VE composite demonstrated the poorest wear performance as compared to hybrid fiber (H1) and hybrid fiber + CSP filled VE samples (H2 and H3) tested in this work, are as shown in Figs. 6, 7 and 8.

The decrease in the  $K_s$  at 5 and 10 wt. % CSP loading (H2 and H3 composites) can be attributed to uniform dispersion of CSP within the matrix and could give full benefit of the combination properties of the hybrid composite. Further,  $K_s$  is sensitive to load of hybrid VE composites than CF-VE under dry sliding wear conditions. At higher loads (>45 N), more heat of friction was generated during sliding process that made the hybrid samples softer and caused the transfer of film easily on the steel counterface.



**Fig. 9**. Photomicrographs showing the microstructure of (a) CF-VE, (b) H1, (c) H3 composites.

This transfer film decreased the wear loss and improved the wear resistance. The  $K_s$  was however found nearly constant in the CSP filled VE composite (H2 and H3). The silane treated CSP

and hybrid fiber modified VE composites reduced the K<sub>s</sub> and the samples could be tested easily up to 60 N without damaging the hybrid reinforcements. The increased weight percent of CSP in composite reduced the K<sub>s</sub> and lower u demonstrating lubrication effect of values coconut shell particles. This effect could be explained by the orientation of fiber as well as effectiveness of the transfer film formed on the rubbing pair. The fiber distribution and orientation of CF, CF/AF and CF/AF/CSP in VE is shown in the photomicrograph as in Figs. 9 (a), (b) and (c) respectively. From the photomicrographs, it is seen that the fiber (both coir and aramid) distribution in VE is random and tangled orientation (Figs. 9a and 9b). The dispersion and interfacial adhesion of fibers and CSP filler is relatively better in hybrid composites (H3) as shown in Fig. 9c.

#### 3.6 Worn surface morphology

The wear behaviour of polymer matrix composites is a very complicated phenomenon in which various mechanisms like adhesion, abrasion, surface fatigue and tribo-chemical processes. Suh [54] stated that wear of materials occurs by many different mechanisms depending on type of material, the environmental and operating conditions and also the geometry of the wearing process. To recognize the adhesive wear mechanism, the worn out surfaces of selected composite samples were studied using scanning electron microscopy (SEM) and micrographs of CF-VE and their hybrids are illustrated in Figs. 10, 11 and 12.

Photomicrographs of CF-VE worn surface after sliding against EN 32 steel counterface under 15 N applied normal load and at a sliding distance of 3000 m for 1 and 3 m/s sliding velocities are shown in Figs. 10 (a) and (b) respectively. Arrow indicates sliding direction. In Fig. 10(a) shows the high wear rate as compared to hybrid composites (H1, H2 and H3), this may happen due to the presence of cellulosic structure in coir fiber which is preventing the property of interfacial adhesion between the fiber and the matrix composites. For the improper adhesion, more fiber damage is seen in Figures 10 (a) and (b). Fibers are severely damaged and caused discontinuity and debonding in CF-VE composites (Fig. 10 (b)). The surface of VE suffers from fragmentation

(marked as F in Fig. 10 (a)) at lower sliding velocity. The major wear failure mechanisms identified in CF-VE samples are; network of micro cracks (indicated as MC in Fig. 10a) in the VE matrix, fiber breakage in length direction (indicated as FB in Fig. 10a), multiple grooving in the sliding direction (indicated as G in Fig. 10a), fiber cutting (indicated as C in Fig. 10b), microgrooves in the sliding direction (indicated as FG in Fig. 10b) and drastic matrix damage (marked as MD in Fig. 10b). Further, the fiber damage is associated with micro-peaks in the wavy surface, narrow grooves and surface fatigue. Matrix micro-cracks and fiber damage are the main mechanisms identified during sliding of CF-VE composites.





**Fig. 10**. Worn surface morphology of CF-VE composites: (a) 1 m/s, (b) 3 m/s.





**Fig. 11**. Worn surface morphology of H1 composites: (a) 1 m/s, (b) 3 m/s.

Figure 11 shows the photomicrographs of the worn surfaces of hybrid fiber reinforced VE composites (H1), indicating that there is a sign of film transfer (Fig. 11b) and micro-cracks (indicated as MC in Fig. 11a) can be seen on the photomicrograph. Gentle observation of Figs. 11a and 11b, reveals the less amount of wear of VE matrix material, and is due to hybrid reinforcements at 1 m/s sliding velocity compared to Fig. 10a. Further, lesser network of micro-cracks, less debris formation and lower trend towards agglomeration of the matrix debris are noticed. The spread out of the VE matrix embedding the hybrid fibers and fillers in the worn surface region is clearly seen. From these photomicrographs (Figs. 11a and 11b), it is noticed that majority of the fibers are masked by the matrix at lower sliding velocity. Few fibers damaged (marked as FG) in the sliding direction as shown in Fig. 11a. From photomicrograph shown in Fig. 11b, due to severe wear of the VE matrix region the reinforcement becomes increasingly visible and partly delaminated (marked as D) from the VE matrix, slid at higher sliding velocity of 3 m/s. Further, matrix cracks (marked as MC) very fine stepped regions along the sliding direction, partly bulged fiber (marked as B) can be noticed.





**Fig. 12**. Worn surface morphology of H3 composites: (a) 1 m/s, (b) 3 m/s.

Figure 12 exhibits the photomicrographs of the worn surfaces of CSP filled hybrid fiber reinforced VE composites (H3). The wear-scars on the VE surface characterized by obvious scale-like marks (marked as T in Fig. 12 a) and smooth surface (marked as S). A close examination of Fig. 12b, shows that micron-size blocks of VE resin have left the sample surface. This is an indicative for fatigue delamination occurred under repeated loading during sliding process. Fatigue wear has been regarded as one of the main mechanism

responsible for the sliding of H3 sample against EN 32 steel counterface. The model is based on the sub-surface crack nucleation and coalescence due to shear deformation of the softer surface induced by the grip of the harder asperities [59]. Due to repeated loading at higher sliding velocity for sample H2 splitting of fiber (marked as FB) and fiber delaminated from VE matrix (marked as D). For filled composites, the worn surfaces are composed of smoothly polished grooves (Figs. 12a and b). This is particularly evident in the case of H3 sample slid at higher sliding velocity (Fig. 12b). By comparing Figs. 11 and 12, it is possible to highlight the effect of increased velocity on the wear surface features of hybrid composite. Thus, the low velocity situation reveals (Figs. 11a and 12a) more matrix damage; complete masking of reinforcements with scattered debris. The high velocity (Figs. 11b and 12b) situation displayed thinning of fiber, fiber delamination with some having their sides sliced. This slicing of fiber and the lack of retention of the matrix material, like those are seen for matrix rich regions, together with loss of CSP filler accounts for the higher K<sub>s</sub> for larger sliding velocity.

# 3.7 Correlations between tribological and mechanical properties

The mechanical properties of neat vinyl ester were improved upon with the incorporation of reinforcements such as surface modified coir/aramid fibers and coconut shell powder. Figures 13 and 14 show the dependence of wear resistance (reciprocal of specific wear rate) on the tensile strength and hardness, respectively. The tensile strength has been increased by about 31-34 % by aramid and CSP reinforcing materials. Higher strength and hardness indicates lower wear rate generally. Mechanical properties such as tensile strength and hardness (Hv) of CF-VE and their hybrid composites may correlate with their wear resistance.

Figures 13 and 14 show the correlation of tensile strength and hardness with the wear resistance for CF-VE, H1, H2 and H3 hybrid composites, under room temperature (24 °C).

As seen from the graphs, the wear resistance with tensile strength and hardness of CF-VE and their hybrid composites were found to be almost linear with an  $R^2$  value of 0.74 and 0.79 respectively.



**Fig. 13**. Correlation of wear resistance with tensile strength of CF-VE composites.



**Fig. 14**. Correlation of wear resistance with hardness of CF-VE composites.

The combined effect of reinforcements (coir, aramid and CSP) appear to have more influence on the hardness rather than that of tensile strength of VE composites and this may be attributed to the modification of the surfaces of reinforcements helped in better interfacial adhesion with the VE matrix and good filler/fiber-matrix interaction.

#### 4. CONCLUSIONS

In this study, the effect of surface treated CSP, coir fiber and aramid fiber on mechanical and tribological performance of the VE composites were investigated and the following conclusions are derived:

- 1. Successful fabrication of vinyl ester based polymer composites reinforced with treated coir, aramid and particles of CSP by simple hand lay-up technique has been achieved.
- 2. From the microstructure of untreated and treated coir fiber, it is clear that the interfacial adhesion between the coir fibers and the vinyl ester matrix is strong enough to overcome the pullout of fibers at the interface.
- 3. Chemically treated CSP and coir/aramid fiber reinforced VE composites (Samples H2 and H3) showed good hardness, tensile strength and tensile modulus compared to CF-VE and H1 composites, indicating the effect of incorporation of AFs and CSP in CF-VE composites.
- 4. Coir fiber reinforced VE hybrid composite (Sample H3) showed 46 % increase in hardness, 35 % increase in the tensile strength and 24 % increase in the modulus indicating good interfacial bonding between the fibers/filler and VE matrix. Silane treated aramid fibers and CSP act as loadbearing reinforcement in the CF-VE composite. These are well corroborated by the fractography features of the VE based composites.
- 5. Specific wear rates of the composites mainly influenced by the applied normal load, sliding velocity, sliding distance and the type of fiber/filler material.
- 6. The relationship between mechanical properties (hardness and tensile strength) and wear resistance was corroborated. The obtained correlation coefficient of hardness to wear resistance was 0.79, thus, the hardness dominates the wear phenomenon of the CF-VE and their hybrid composites.
- 7. The SEM studies of worn surfaces have revealed various wear mechanisms such as micro-cracking, grooving, formation of debris, fiber slicing, surface fatigue and fiber matrix de-bonding. Also, SEM observation showed no sign of pullout, tear, or breakage of fibers.
- 8. The hybrid composite (H3) exhibits a better mechanical and wear performance than CF-VE and other hybrid composites. Hence, composite H3 can be suggested for bearing as well as for structural applications in automotive industry.

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