

Study on friction and sliding wear behavior of woven S-glass fiber reinforced vinylester composites manufactured with different comonomers

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Abstract The effect of variations in sliding velocity and applied normal load on the friction and sliding wear behavior of glass–vinylester composite (G–V) is studied by measuring the weight change and observing the surface features of worn specimens using scanning electron microscopy (SEM). The G–V composites were manufactured with Bi-directional woven S-glass fibers (65 wt%) reinforced with vinylester resin with different comonomers. Friction and wear experiments were carried at ambient conditions on a Pin on disc machine arrangement. The wear in the experiment was determined from the weight loss measured after running against steel disc at sliding velocities of 1, 2, 3, and 4 m/s and applied normal load of 10, 20, 30, and 40 N. The experimental findings show increase in specific wear rate with increase the applied load. Specific wear rate was maximum for the sample A (Styrene as Comonomer), intermediate for sample B (Methyl acrylate as comonomer), and least for sample C (Butyl acrylate as comonomer). It was also observed that increasing normal load and sliding velocity the coefficient of friction decreases. The scanning pictures show features like tendency for the matrix to adhere towards the fiber, debris formation, network of cracks, agglomeration of debris, and broken fibers depending on the load and velocity employed.

Introduction

Fiber reinforced polymeric composites have gained importance due to their high specific strength and modulus. Besides a wider choice of materials and ease of manufacturing make them ideal for engineering applications. The organic composite systems generally which use thermosetting resins such as epoxy, phenolic, polyester, and polyimide as matrix material are important for many engineering applications [1–3]. Due to the account of their good combination of properties, fiber reinforced composites (FRCs) are used particularly in the automobile, aircraft industries, the manufacturing of ships and sea vehicles [3–5]. The other two important characteristics which makes polymer composites more attractive in comparison to metallic counter parts are relatively low density and ability to be tailored to have a stacking sequence that provide the strength and stiffness in directions of high loading [6]. Composite materials consist of resin and reinforcement and they are chosen according to the desired mechanical properties and the applications for which the composites are used [4, 7]. A fiber as a principal constituents occupy the maximum volume fraction in laminate composite materials and share the major portion of the load acting on the composite structure [7]. Among the various fibers, glass fibers are widely used. The main advantages of glass FRCs are good corrosion resistance, light weight, and better damping characteristics than metals. Polymer composites reinforced with glass fibers are usually one to four times stronger and stiffer than their unfilled matrices. The role of matrix in a fiber-reinforced composite is to transfer stresses between the fibers to provide a protection against an adverse environment and also to protect the surface of the fiber from mechanical abrasion [8, 9]. Among the resins, polyester, epoxy, phenolic, silicone, and polyamide resins

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are widely employed. The epoxy resins have low coefficient of friction and thermal expansion as well as higher load bearing capabilities compared to some thermal plastics. Polyesters are also commonly used as matrix material particularly with glass fiber reinforcement due to low cost and resistance to adverse environment [2–4, 10].

Polymers and polymer composites, especially FRCs form a very important class of tribo-materials and used in components which are run without any external lubricants. Such components have performance which is application sensitive. It is also well established fact that no material is universally resistant to all modes of wear. Hence during material selection for typical tribo application it becomes imperative to know its complete spectrum of behavior in various possible wearing situations [11]. Polymer composites find extensive use in tribological components such as brake pads, cams, gears and bearings because of their self-lubrication properties, lower friction and better resistance to wear. However, polymers find difficulty in use as bearing materials in its pure form, because neat polymer cannot satisfy the demands arising from the situations where combinations of good mechanical and tribological properties are required [12, 13].

Among the thermoset resin besides widely investigated resins like epoxy and polyester, the friction and wear characteristics of phenolic also has been investigated as an important friction and wear material for brake pads in automobile. In this investigation of tribological properties of phenolic matrix with addition of fillers like Cu Powder, BaSO₄, Al₂O₃, the improvement in friction and wear performance have been observed. The finding suggested the improvements in friction and wear properties but direct proportionately between hardness and wear resistance could not be established. The Boric acid used in phenolic deports the water and establishes the structural equilibrium. The performance by addition of barite in these phenolic matrix and boric acid, composites show improvements friction and wear performance in different environmental conditions [14, 15]. Among the glass fiber reinforced polymer composites the bidirectional fabric type of composites are gaining popularity in recent years because of their balanced properties in the fiber plane as well as their easy handling while fabricating the composites. Two-dimensional (2D) fabrics have been relatively well developed in terms of production, analysis and application and some of them have long been used in structural and tribological applications. However, most of 2D structures have weakness of laminated composites that are susceptible to delamination [16–18]. In recent years, more of research work is being directed to the exploring the potential advantages of thermoset matrix for composite applications [19]. Vinylester is one of this kind of matrix material, which has found a place in the family comprising

thermoset engineering polymers due to its excellent mechanical properties with excellent chemical/corrosion resistance even at elevated temperature. The vinylester resins are stronger than polyester resins and cheaper than epoxy resins. These resins utilize a polyester resin type cross-linking molecules in the bonding process. The vinylester is a hybrid form of polyester which has been toughened with epoxy molecules within the main molecular structure. These resins offer better resistance to moisture absorption than polyester resins. It is also known that vinylester resins bond very well to fiberglass [20, 21].

Considering the above mentioned observations, an attempt is made to investigate the frictional and wear behavior of S-glass fiber reinforced vinylester composites, prepared with different comonomer namely Styrene, Methyl acrylate, and Butyl acrylate as comonomer.

Experimental

Preparation of vinylester resin

The reaction to form the vinylester oligomer is usually catalyzed by tertiary amines, phosphine, and alkali salts. The research shows that triphenyl phosphine is a more effective catalyst for this reaction as compared to other catalysts. Typical reaction conditions are 120 °C for 4–5 h, and hydroquinone is commonly used as the initiator. The conversion of the reaction is 90–95%.

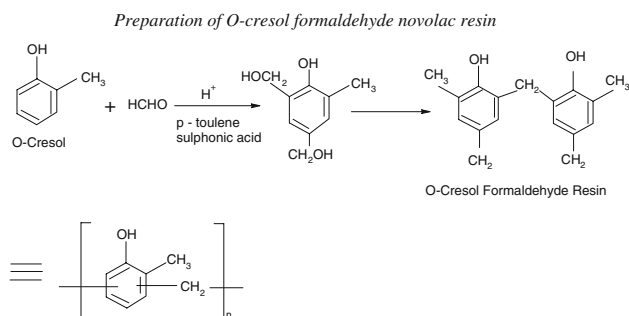
Experimental procedure

ortho-Cresol (S.D Fine Chemicals) and formaldehyde (37–41% solution, S.D. Fine Chemicals) were used for the preparation of novolac. Resorcinol (CHD), triphenyl phosphine (CHD), imidazole (CHD), Epoxy novolac resin (EPN-1138, Ciba-Giegy), methacrylic acid (Merck), triphenylphosphine (Fluka AG), butyl acrylate (Merck), and styrene (Ranbaxy), are used in the present study. Epichlorohydrin L.R. grade (CDH), sodium hydroxide pellets (Merck), used for the preparation of epoxy novolac.

Synthesis of *ortho*-cresol formaldehyde novolac

ortho-Cresol and formaldehyde in the molar ratio of 1:0.7 were used for the preparation of novolac resin. Weighed amount of *ortho*-cresol (108 g) was dissolved in 6 mL of water and its pH was adjusted to 1.5 with conc. H₂SO₄ under a pH scan 3+ double junction pH meter. This solution was charged into a 550 mL capacity, three-necked flask fitted with a Teflon coated blade-stirrer, a mercury thermometer and leibig condenser. The solution was heated to 80 °C and

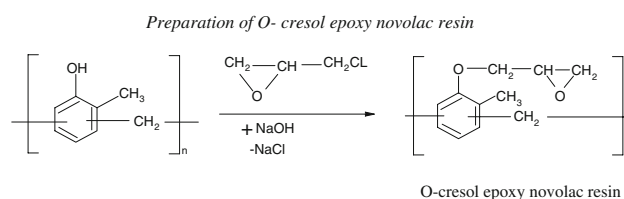
maintained at this temperature with constant stirring and formaldehyde solution (55.3 mL) was added in a drop wise manner over a period of 4 h. Heating was continued for an additional half an hour after the completion of the addition of formaldehyde solution. Forty milliliter of 10% sodium bicarbonate was added to neutralize the H_2SO_4 and arrest the reaction. The reaction mixture from the flask was then transferred to the separating funnel and washed with warm water to neutralize the pH removing excess sodium bicarbonate and salt. Four to five washings were generally adequate for the purpose. The resin was finally dried at 80°C under reduced pressure (35 ± 5 mm Hg).



Synthesis of epoxy novolac from ortho-cresol formaldehyde novolac resin

Epoxy novolac resin was prepared by reacting the novolac resin with epichlorohydrin (5 mol of for every phenolic group of the novolac resins). Sodium hydroxide in the mole ratio with epichlorohydrin of 0.2:1.0 was used as catalyst. The apparatus used for the preparation of epoxy novolac consisted of an oil bath, a 500 mL capacity 3-necked flask fitted with a glass stirrer having Teflon coated blade in the

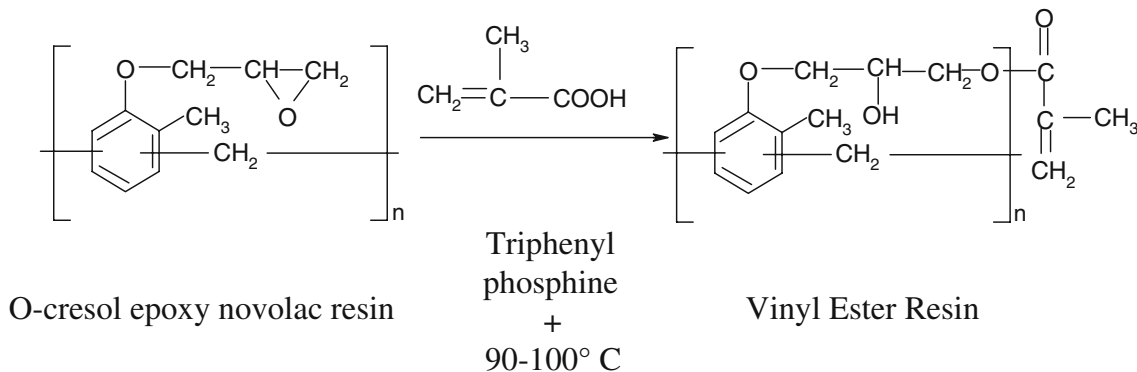
central neck, Dean and Stark assembly in one side neck, and in another side neck a thermometer pocket and a dropping funnel to deliver sodium hydroxide at a regular rate by the side of thermometer pocket. Novolac resin was (50 g) with epichlorohydrin (160.632 mL) was charged into the flask and heated under stirring to temperature of $112^\circ\text{C} \pm 1^\circ\text{C}$. While maintaining this temperature for epoxidation to proceed, 16.3934 g of sodium hydroxide (40% w/w) was added gradually to the reactants in the flask over a period of three and half hours. Heating was continued for an additional 16 min, after which the contents were dissolved in toluene and the solution filtered using Whatman filter paper no. 42 to remove the salts. Toluene was then removed by heating under reduced pressure.



Synthesis of vinyl ester resin form ortho-cresol epoxy resin

Vinylester resin was prepared using 1:0.9 mole ratios of *ortho*-cresol epoxy novolac resin prepared (epoxide equivalent weight: 259, determined by pyridinium chloride method and methacrylic acid in the presence of imidazole (1 phr by weight of the epoxy resin) and hydroquinone (200 ppm) at $85^\circ\text{C} \pm 1^\circ\text{C}$. The etherification reaction was carried out for a 3 h to obtain a product with an acid value of 20 mg KOH/g solids determined. The VER samples were stored in a refrigerator at 10°C .

Preparation of vinyl ester resin based on O-cresol novolac resin

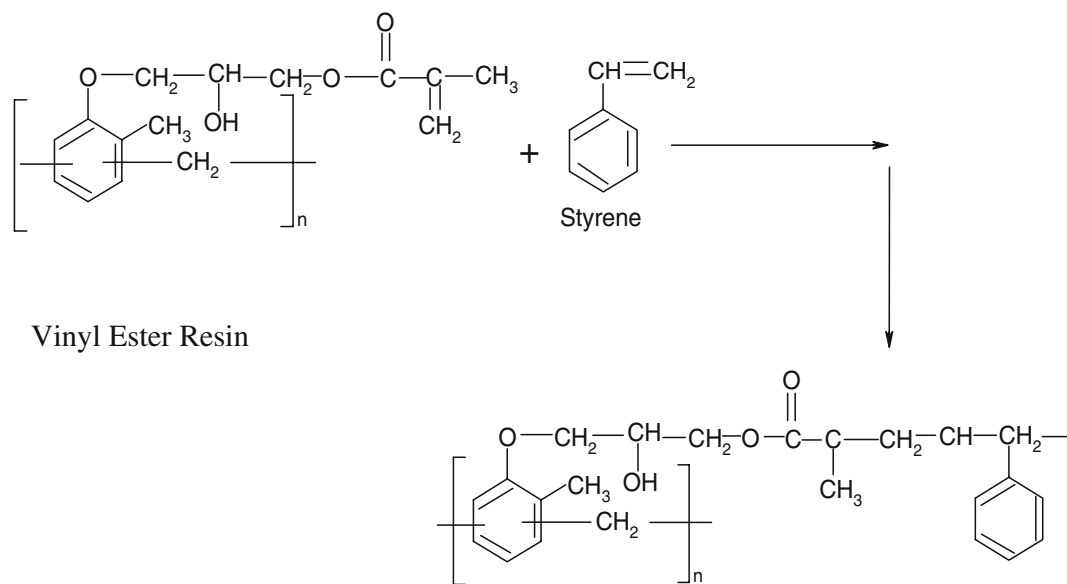


Cross-linking reaction with different comonomer

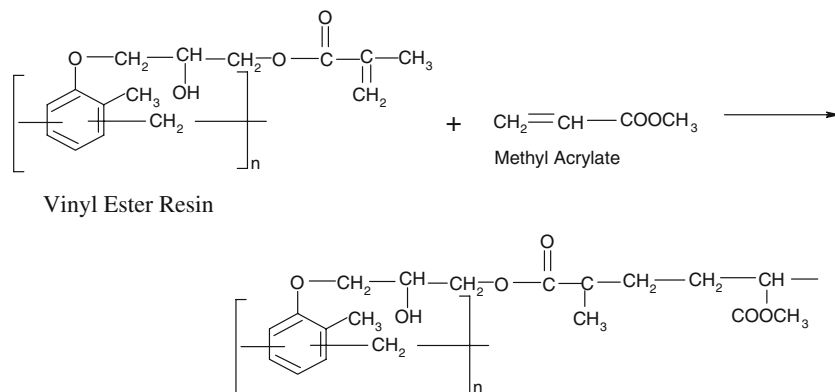
The vinylester resins are cross linked by free radical copolymerization of methacrylate end groups with styrene. It is a system of copolymerization of vinyl/divinyl monomers. The gelation occurs when a three-dimensional network or an infinite molecular weight polymer (gel) is formed. A gel molecule cannot be dissolved in any solvent. Experimentally, gel times (the time to reach the onset of gelation at a particular temperature) are measured using solubility experiments. Vinylester resins usually contain 30–50 wt% styrene and 70–50 wt% vinylester oligomers. Early work on the model phenyl–glyciyl–ether methacrylate showed that it reacted more rapidly than styrene in bulk or in solution because of hydrogen bonding between the polymer radical and the methacrylate monomer. Reactivity ratios for this copolymerization with styrene

show that, at least in the early stages of the reaction, the methacrylate content is high relative to styrene and methacrylate’s copolymerization. Due to the very high concentration of divinyl monomers, gelation begins at very low conversions. For vinylester resins, the onset of gelation is usually rapid-right after the induction period with little or no appreciable increase in viscosity prior to gelation. The time to the onset of gelation is dependent on the concentration of free radical. Increasing the concentration of the initiator and/or accelerators and the use of elevated temperatures will shorten the time to onset of the gel. In this work three different monomers, i.e., Styrene, Methyl acrylate, and Butyl acrylate are used for preparing three different samples so as to compare the friction wear properties of the three samples. In all the three cases the ratio of resin to comonomer (reactive diluent) is kept 10:4 by weight.

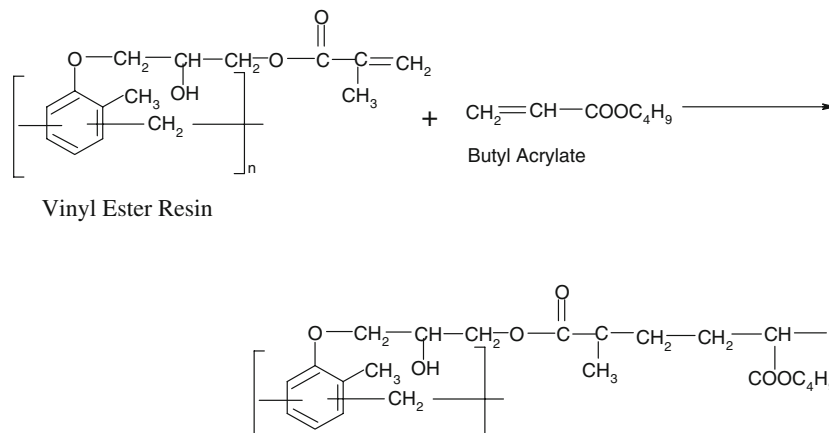
Cross linking of vinylester with styrene as comonomer



Cross linking of vinylester with methyl acrylate as comonomer



Cross linking of vinylester with butyl acrylate as comonomer



Preparation of composite samples

After the cross linking and gelation, vinylester resin with different comonomers is used for preparation of composites. The vinylester resin prepared from *ortho*-cresol formaldehyde novolac resin having density 1.42 g/cc is used for making composites. The bi-directional woven S-glass fiber (modulus 85.5 GPa, density 2.49 g/cc) is used as reinforcement (supplied by Saint Gobian Ltd.). Before preparing the composites the vinylester is mixed with comonomers along with an initiator Benzoyl-peroxide (Central Drug House Ltd. India) in order to have pre gel formation of the resin. Similar procedure is followed for all the three samples. Hand layup technique was employed to fabricate the composites. The stacking procedure consists of placing the fabric one above the other with the resin mix well spread between the fabrics on a mould release sheet. A porous Teflon film was again used to complete the stack. To ensure uniform thickness of the sample, a 3 mm spacer was used. The mould plates were coated with release agent in order to aid the ease of separation on curing. This procedure was repeated in all three cases unless thickness of 3 mm was obtained. A metal roller was used so that air entrapped could be removed and compactness could be improved. The whole assembly is placed in the compression molding machine at a pressure of 60 Kgf/cm² and allowed to cure at room temperature for 12 h initially. The post curing carried as per the schedule shown in Table 1.

Curing of glass–vinylester composites

The composite laminates are prepared and then they are air dried for about 4 h so that pre-gelation takes place. The curing reaction is a free-radical chain growth cross-linking polymerization between the reactive diluent and the

Table 1 Curing schedule for the composite samples

Sl. No.	Temperature (°C) (curing process)	Pressure (kgf/cm ²)	Time (h)
01	Room temperature	60	12
02	60	60	8
03	90	60	4
Post curing process			
04	120	60	4

terminal ethylene group of the vinylester resin. The vinylester monomer contributes in cross-linking capacity and branch points for the developing network, while the styrene monomer provides linear chain extension. Depending on the temperature and other curing conditions, some of the species remain unreacted after the curing in the form of residual monomer, soluble polymer and do not contribute towards the cross-linked network. The samples are then cured in a compression molding machine. During the curing process, initially laminates are cured at room temperature and pressure of 60 Kgf/cm². The post curing is carried at higher temperature as per the curing schedule mentioned in Table 1. Three different composite laminates manufactured are designated on the basis of type of comonomer used. Sample A—Styrene, Sample B—Methyl acrylate and Sample C—Butyl acrylate are used as comonomers. The laminate sheets of sizes 300 mm × 300 × 3 mm were prepared. These samples were analyzed for sliding wear characterization as per ASTM standard. The specimens used for testing were cut from the respective sheets using diamond cutter.

Friction and wear measurements

To evaluate the friction and sliding wear performance of G–V composites A, B, and C under dry sliding condition,

wear tests are carried out in a pin-on-disc test rig (DU-COM) as per ASTM G 99. The counter body is a disc made of hardened ground steel (EN-32, hardness 72 HRC, surface roughness 0.7 μ Ra). The specimen is held stationary and the disc is rotated while a normal force is applied through a lever mechanism. During the test, friction force was measured by a transducer mounted on the loading arm. The friction force readings are taken as the average of 100 readings every 40 s for the test duration. For this purpose a microprocessor controlled data acquisition system is used. The test conditions for conducting experiments for these samples composites A, B, and C are given in Table 2. Sliding wear data reported here is the average of at least three runs. The average mass loss is used to calculate the specific wear rate. A series of test are conducted with sliding velocities of 1, 2, 3, and 4 m/s under different normal loading of 10, 20, 30, and 40 N. The material loss from the composite surface is measured using a precision electronic balance with accuracy ±0.01 mg.

The specific wear rate ($\text{mm}^3 \text{N}^{-1} \text{m}^{-1}$) is expressed on ‘volume loss’ basis

$$K_S = \Delta m / L \rho F_N$$

where K_S is the specific wear rate ($\text{mm}^3 \text{N}^{-1} \text{m}^{-1}$), Δm is the mass loss in the test duration (g), ρ is the density of the composite (g/mm^3), t is the test duration (s), V_s is the sliding velocity (m/s), F_N is the average normal load (N), L is sliding distance.

Scanning electron microscopy examination

The SEM pictures of G–V composite samples A, B, and C are shown in Figs. 7, 8, 9, 10, 11, and 12. The surfaces of the specimens were examined directly by scanning electron

microscope JEOL JSM-6480LV. The composite samples were mounted on stubs with silver paste. To enhance the conductivity of the samples, a thin film of platinum was vacuum-evaporated onto them before the photomicrographs are taken.

Results and discussions

Table 2 presents details of composites, testing conditions and parameters used for conducting the various experiments of G–V composite samples A, B, and C. G–V composites A, B, and C are tested at room temperature under dry sliding conditions at applied normal loads of 10, 20, 30, and 40 N and sliding velocities of 1, 2, 3, and 4 m/s. Table 3 shows the specific wear rate values calculated from mass loss data obtained during experimentation. Experimental results of coefficients of friction and specific wear rates are plotted in Figs. 1, 2, 3, 4, 5, and 6 for different applied normal loads (10, 20, 30, and 40 N) and sliding velocities (1, 2, 3, and 4). Figures 1, 2, and 3 show the variation of friction coefficients with normal load of G–V composite A, B, and C as function of load for different sliding velocities. Figures 4, 5, and 6 show the variation of specific wear rate with load of G–V composite A, B, and C at different sliding velocities. Scanning Electron Microscopic pictures of typical worn surfaces for the tested specimens are shown in Figs. 7, 8, 9, 10, 11, and 12 in which different wear mechanisms can be seen. Within the present testing conditions it can be seen that there is considerable dependence of the friction coefficients and wear rates on the normal load and sliding velocity conditions.

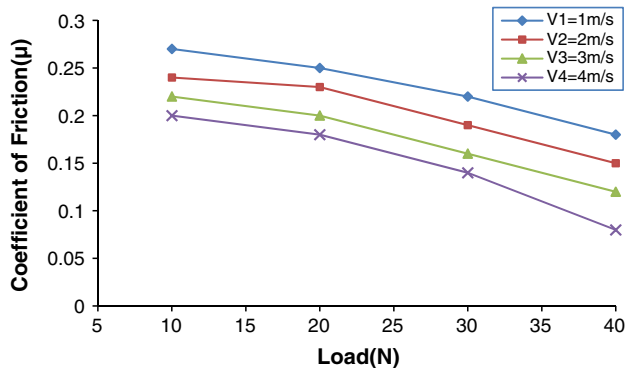
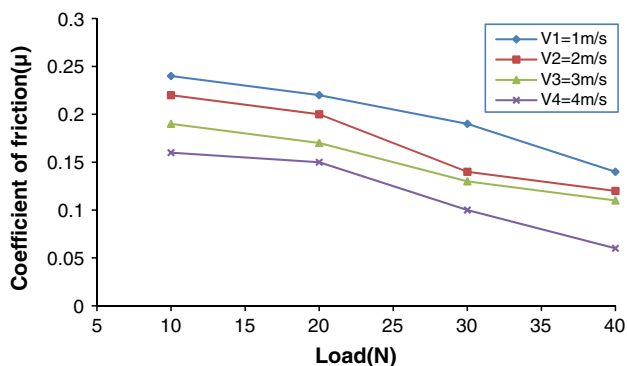
By comparing friction coefficients and specific wear rate of the G–V composites A, B, and C in Figs. 1, 2, 3 and

Table 2 Details of composite samples, testing conditions and parameters

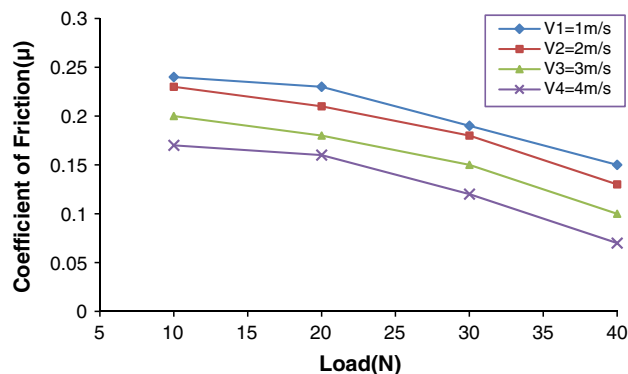
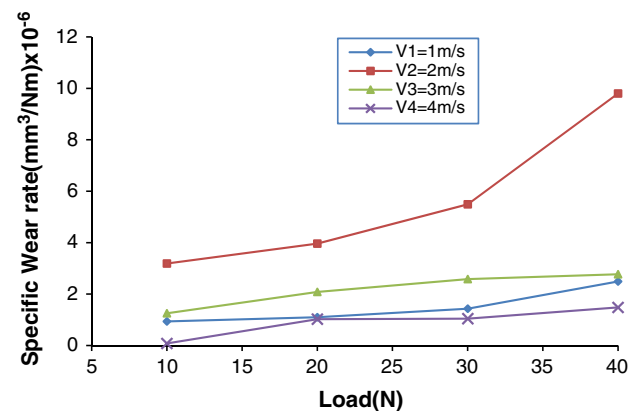
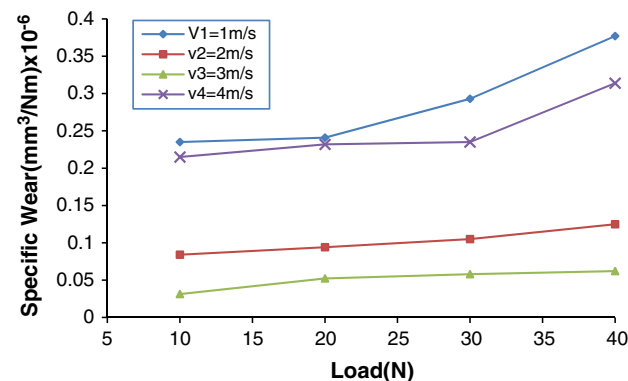
Material	Composition	Color	Fiber weight fraction (%)	Density (g/cm^3)	Test temperature ($^{\circ}\text{C}$)	Load (N)	Speed (m/s)	Humidity (%)
A	Vinylester + Styrene	Brown	65	1.87	27.6	10	1	65.1
						20	2	
						30	3	
						40	4	
B	Vinylester + Methyl Acrylate	Brown	65	1.77	26.5	10	1	66.2
						20	2	
						30	3	
						40	4	
C	Vinylester + Butyl Acrylate	Brown	65	1.82	27	10	1	66.3
						20	2	
						30	2	
						40	3	

Table 3 Specific wear rate for G–V composites tested at different load and speed values

Composite material	Load (N)	Specific wear rate (mm^3/Nm) $\times 10^{-6}$			
		Sliding speed			
		1 m/s	2 m/s	3 m/s	4 m/s
A	10	0.935	3.190	1.250	0.081
	20	1.100	3.960	2.080	1.020
	30	1.430	5.490	2.580	1.040
	40	2.490	9.800	2.770	1.480
B	10	0.235	0.084	0.031	0.235
	20	0.241	0.094	0.052	0.262
	30	0.293	0.105	0.058	0.235
	40	0.377	0.125	0.062	0.314
C	10	0.366	0.168	0.012	0.081
	20	0.440	0.183	0.012	0.069
	30	0.549	0.210	0.092	0.107
	40	0.550	0.214	0.173	0.122

**Fig. 1** Coefficient of friction versus applied load of G–V composite A**Fig. 2** Coefficient of friction versus applied load of G–V composite B

Figs. 4, 5, 6 and Table 3, the G–V composite A shows maximum value for friction coefficients and followed by composite B and lowest for composite C. By comparing the

**Fig. 3** Coefficient of friction versus applied load of G–V composite C**Fig. 4** Specific wear versus applied load of G–V composite A**Fig. 5** Specific wear versus applied load of G–V composite B

three different G–V composites A, B, C, the highest values of coefficient of frictions and specific wear rate were evident for dry sliding against steel surface containing maximum debris formation in A and exposure of glass fibers at higher loads. This is attributed to the possible effects of loose damaged particles which contain glass fragments from G–V composites at the contacting surface interface. It is well established fact that the glass fibers have relatively

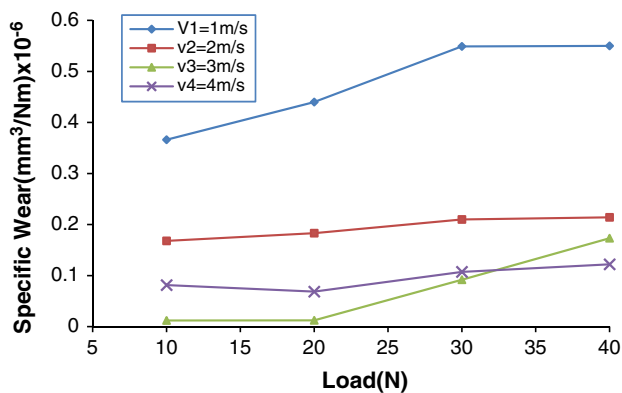


Fig. 6 Specific wear versus applied load of G-V composite C

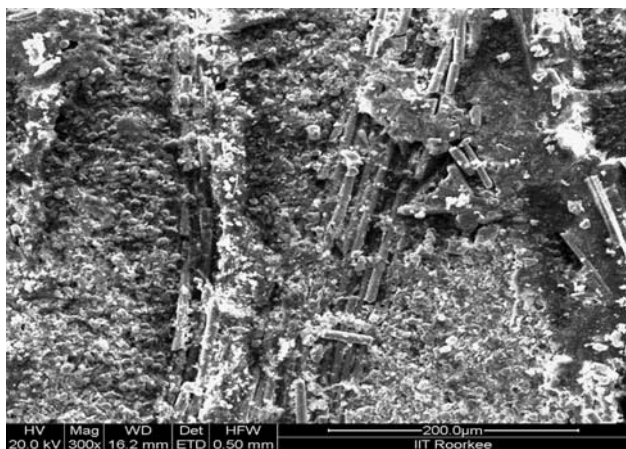


Fig. 7 SEM picture of sample A at load (20 N) and sliding speed (2 m/s)

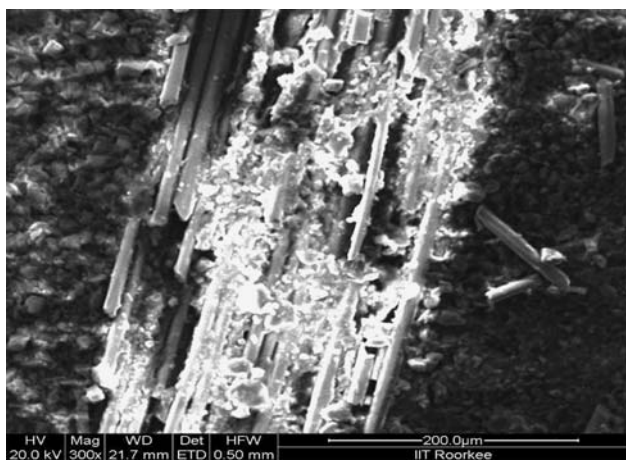


Fig. 8 SEM picture of sample A at load (40 N) and sliding speed (2 m/s)

higher friction coefficients than the matrix such as vinyl-ester, epoxy, and polyester [5]. Due to the presence of this loose debris of the glass fiber give rise to more loose



Fig. 9 SEM picture of sample B at load (20 N) sliding speed (2 m/s)



Fig. 10 SEM picture of sample B at load (40 N) and sliding speed (2 m/s)

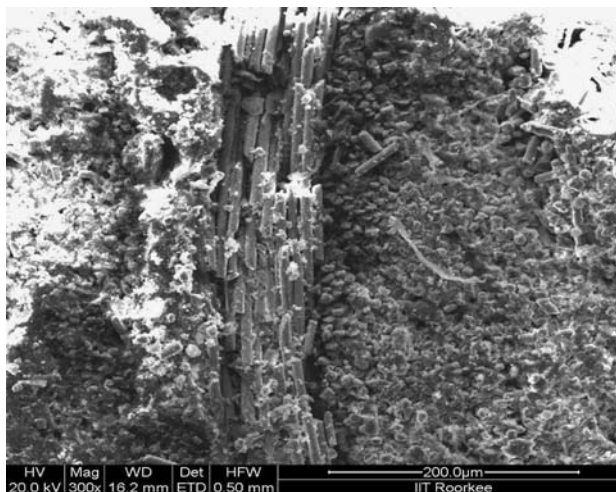


Fig. 11 SEM picture of Sample C at load (20 N) and sliding speed (2 m/s)

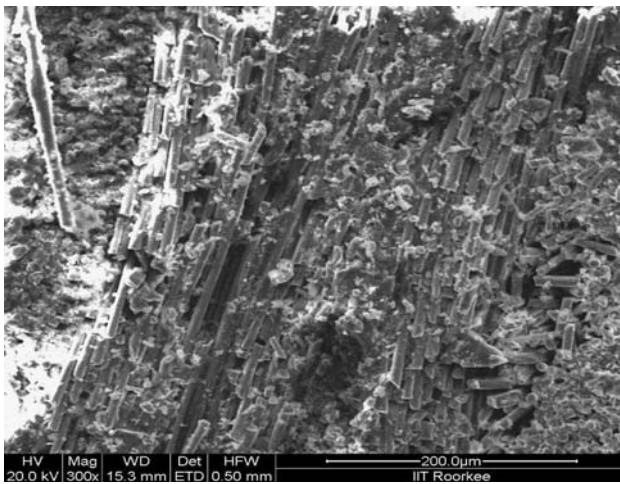


Fig. 12 SEM picture of Sample C at load (40 N) and sliding speed (2 m/s)

particles and this in turn increases the abrasive force, leading to the increased frictional resistance and wear rates. The SEM pictures of G–V Composite A (Figs. 7, 8) at different load and sliding speed conditions show the characteristic features of the worn surface under these conditions. The worn surfaces under similar conditions of normal load and sliding speeds for G–V composite sample B and C are presented in the SEM graphs in Figs. 9, 10 and Figs. 11, 12, respectively. The micrograph Fig. 9 of G–V composite B shows small patches of debris. However, at higher loads of 40 N and sliding speeds of 2 m/s it shows that more fibers are exposed and more debris formation in the form of layers is seen. In SEM graph Fig. 11 for composite C also depicts small patches in small regions only and the small amount of fibers are exposed in the middle of specimen. From the SEM graph Fig. 12 it is evident that at higher load fiber breakage, debris formation and fiber exposure on surface is seen, which has resulted due to increased specific wear rate.

The accumulation of wear debris in the form of patches is seen in some regions of the worn surface of composite specimens. This wear debris plays an important role as third body abrasive in the wear process. Such wear debris leads to high values of wear rates and friction values considerably. Due to the presence of glass fibers in debris which is abrasive in nature may also abrade counterface and hence increases the roughness of surface [5, 10]. This in turn increases the wear rate. The other two possible modes and mechanisms of wear which may occur during the experimentation, firstly the shearing stresses occurred at trailing end of the fibers and this would accelerate wear with a consequential surface failure [11]. However, shearing stresses seem to be of contribution especially for the increase in wear rate for composites A, B, and C with increase in load in Figs. 4 and 5. Second possible

mechanism consists of surface degradation at the interface due to temperature rise. This gives a decrease in the friction coefficient and increase in wear rate. This is true for friction coefficient as shown in Figs. 1, 2, and 3 and for specific wear rates in Figs. 4, 5, and 6. The variation of specific wear rate of G–V composites with sliding velocity shows differing trends. For the G–V composite A the specific wear rate increases with increase in sliding speed. For G–V composites B and C the specific wear rate decreases initially and then increases at higher values of sliding speed. Whereas for G–V composite C the specific wear rate is higher than G–V composite B at lower sliding speeds. This is also evident from the SEM graph Figs. 9, 10, 11, and 12. Under similar applied normal load and sliding velocities the SEM graphs in Figs. 11 and 12 show more of debris. But with increase in sliding velocity the specific wear rate of composite sample C is lower than the sample B. The lowest specific wear rate is also shown by the G–V composite sample C at sliding velocity of 3 m/s in Table 3. However, the effect of sliding speed on specific wear rate is more pronounced for G–V composite A at sliding speed of 2 m/s where as for all other composites the specific wear rate is very small. To clarify the trend of friction coefficient with normal loads and sliding velocities Figs. 1, 2, and 3 show that increasing either normal load or sliding velocity leads to increase in the temperature at the sliding interface. This increase in temperature causes a thermal penetration to occur which results in weakness in the bonds at the fiber–matrix interface [5, 14]. Consequently, the fibers become loose in the matrix and shear easily due to axial thrust. As a result coefficient of friction decreases.

Conclusions

The following conclusion can be drawn from the present study.

1. Friction and sliding wear studies against steel disc counterface under various loads and sliding speeds conducted in this work show that vinyl ester composites manufactured with Styrene as comonomer show the maximum specific wear rate followed by composite manufactured with Methyl acrylate as comonomer and least by composite manufactured with Butyl acrylate. The specific wear rate exhibited by composite C in the order of 10^{-10} mm²/N can be considered as a very good tribo-material.
2. The friction coefficient of all the composites A, B, and C decreases with increase either in load or sliding speed and have low values as that of a good material used for friction applications.

3. For the range of load and sliding velocity in this study it is observed that load has stronger effect on the friction and wear than the sliding velocity.
4. SEM micrographs of the specimen reveal matrix debris formation, fiber breakage, fiber matrix debonding and exposure of fibers due to wear of the matrix.

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