Research Article

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Effects of nanofillers on the physical, mechanical, and tribological behavior of carbon/kenaf fiber–reinforced phenolic composites

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Abstract: This research investigates the effect of kenaf/carbon fiber reinforcement with the addition of nanofillers such as multi-walled carbon nanotubes (MWCNT) and cellulose nanocrystal (CNC) in the phenolic matrix composite. Three different types of samples were made using short carbon fiber, kenaf fiber (KF), $BaSO_4$, Cashew dust, MWCNT, and CNC. Sample 1 contains 40 wt% of carbon fiber with binders reinforced in the phenolic matrix, whereas, in sample 2, carbon fiber has been replaced with KF in the MWCNT-modified phenolic matrix. On the other hand, in sample 3, carbon fiber has been replaced with KF in a CNC-modified phenolic matrix. The physical, mechanical, and tribological properties were investigated and compared. The results showed that sample 1 exhibited higher mechanical performance compared to other samples. On the other hand, the least wear loss and a high coefficient of friction were observed for Sample 2 compared to Samples 1 and 3. Even though KF-based composites exhibited slightly lower mechanical performance, they showed excellent tribological behavior.

Keywords: carbon fiber, kenaf, MWCNT, CNC, phenolic and polymer

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Abbreviations

COF	coefficient of friction
PAN	polyacrylonitrile
MWCNT	multi-walled carbon nanotubes
PKF	palm-kernel fiber
CNSL	cashew nut shell liquid
CNC	cellulose nanocrystal
MS	mild steel
CR	cyrtostachys renda
KF	kenaf fiber
MH	magnesium hydroxide

1 Introduction

Friction materials for automotive applications must be low wear, thermally stable, have an optimal friction coefficient, be free of hazardous emissions, and be reasonably priced [1]. Carbon fiber composite materials used in automotive applications offer exceptional mechanical qualities and a superior strength-to-weight ratio, resulting in less fuel consumption and lower carbon emissions [2]. The distinctive qualities of carbon fiber, such as inherent self-lubrication, high strength, high modulus, and appropriate thermal stability, make it a charming candidate to be used as a reinforcement in different kinds of composites [3,4]. The cost and brittleness of carbon fiber are higher than those of natural fibers, which offer the benefits of being biodegradable, renewable, and less expensive than carbon fiber [5]. Natural fibers have the following limitations is there such as higher moisture uptake, quality variations, low thermal stability, and poor compatibility with the polymeric matrix [6]. Monolithic materials cannot have all these properties. However, hybridizing different fibers in a nanofiller-modified polymer matrix can provide superior properties compared to monolithic materials [7]. Using natural fibers is a good alternative, but due to their hydrophilic nature, it leads to weak bonding in a matrix. So, investigating different weight percentages and different constituents is needed for this study [1]. One approach that shows promise for toughening composite materials is fiber hybridization. Compared to non-hybrid composites, these hybrid composites offer a superior balance of mechanical characteristics thanks to the combination of two or more fiber types [8].

Kenaf fiber (KF) composites have demonstrated higher flexural properties than other natural fiber composites. The high cellulose content and excellent microfibril orientation in kenaf are the key reasons for the high flexural modulus of KF/polypropylene composites. In addition, KF/polypropylene composites show an increase in crystallization during working, the kinetic energy. This is because KF has a high crystalline concentration. Due to the excellent energy absorption ability of kenaf, the composites also displayed increased storage modulus (E′) as a function of temperature [9]. Natural fiber-based composites exhibit slightly lower mechanical strength compared to synthetic fiber-reinforced composites. However, matrix modification with nanofillers would improve the mechanical strength and stiffness. Therefore, a minimal amount of multi-walled carbon nanotubes (MWCNT) as an inorganic nanofiller is used to enhance the mechanical strength and stiffness of KF-reinforced polymeric composites. The use of carbon nanotubes (CNTs), graphite nano-graphite, and short carbon fibers was because of the interconnecting network between carbon and carbon bonds of these fillers. This often improves the mechanical, thermal, and tribological ability of the composites. A hybrid of CNTs and short carbon fibers in the polymer matrix has a synergistic effect, which in turn contributes to the enhancement of material properties [10–12].

Satapathy and Bijwe studied the influence of organic fibers in improving the wear resistance of the composite. The PAN and carbon fiber inclusion are responsible for lowering the of the composite. Cellulose fibers though offered higher coefficient of friction (COF), proved detrimental to wear resistance and fade [13]. In the work of Chandradass, experimental work results revealed that a sample with 10 wt% of carbon fiber has high flexural strength, Rockwell hardness, and COF. This is due to the interfacial bond strength between the fiber and the matrix Also, the wear test showed a low value for the sample containing 10 wt% of carbon content [14].

Barium sulphate is a low-cost filler material that has a high density that helps to fill the voids in the composite. Also, it has a high melting point, thus it imparts heat stability to the tribological material [15]. Nano-scale fabrication has recently set off in nano-material synthesis for the development of lightweight tribological friction materials. The inclusion of nanoparticles in the base material is done for the requirement of tribe and wear requirements. CNTs have a distinct topological hollow tubular structure; due to this property, it has a high L/D ratio, characteristic electronics properties, mechanical ability, and high thermal stability. CNT is used to produce nanocomposites with excellent trio performance [3].

The amount of additives (MWCNT) weight percentage is kept low since the efficiency of carbon-based additives is often significant on polymer matrix when a low amount of these additives is added and depleted when the additive content is more. The investigation revealed that 0.3% MWCNT obtained a higher COF value (0.413) than 0.1 and 0.2% (0.319) [16]. The COF depends on the materials used, most dry materials in combination have friction coefficient values between 0.3 and 0.6 [17]. Phenolic resin is extensively utilized because of its ease of obtaining raw ingredients, low cost, superior heat resistance, high mechanical strength, and consistent performance [18].

MWCNT and cellulose nanocrystals (CNC) are used as nanofillers to enhance the properties of the composite. The inclusion of MWCNTs, which have high microstructural properties, contributes by increasing the hardness of the epoxy-polyamide nanocomposites. With the high-strength nanoparticles incorporated with the phenolic matrix composite, the microhardness value has been increased. It is also found that the values of wear decrease with the increase of the wt% of CNTs but it increases with wt% higher than 0.5 wt%, due to the collection of nanomaterials [19]. MWCNT of 0.5% included with 50% fiber-reinforced phenolic composites improved the flexural, tensile, and impact strength than pure phenolic composites by creating interlocking between fiber and the matrix. These bio-based composites when reinforced with nanofillers provide desirable properties with low cost [10].

Timur *et al.* studied the effect of various percentages of cashew dust (5, 10, and 15%) on the tribological properties of tribological friction materials using a brake dynamometer. It was shown that 10% of cashew friction dust-based friction materials were beneficiary by showing improved stability and fade resistance [20]. Cashew dusts are less expensive than other tribological materials, which inspired the notion of investigating their potential use in friction materials. The results show that the addition of cashew dust enhances the hardness by 2.3% and the ignition loss by 3%. Due to limited porosity and the fact that cashew dust deforms to prevent thickness increases in the composite material, it is evident that adding cashew dust reduces automotive component composite thickness [21–24].

Carbon fibers are made mostly of carbon atoms and are about 5–10 micrometers wide. Carbon fibers have many benefits, such as being stiff, strong, light, resistant to chemicals, able to handle high temperatures, and not expanding much when heated [25]. Kenaf is made up of 60–80% cellulose, 5–20% lignin, and up to 20% moisture. The cell wall is a hollow tube with four layers: one main cell wall, three subsidiary cell walls, and a lumen, a microfibril's open channel. Similar to an artificial fiber-reinforced composite, each layer is cellulose embedded in hemicellulose and lignin. Hemicellulose is responsible for fiber biodegradation, moisture absorption, and heat degradation. Although lignin is thermally stable, it destroys fibers when exposed to UV light [26].

Lee *et al.* studied the mechanical and thermal properties of KF-reinforced polypropylene (PP) composites and compared them with pure PP composites and KF/magnesium hydroxide composites. The KF/PP composites showed better flexural strength than other composites. The samples were fabricated with different compositions from 10 wt% KF to 25 wt% KF with PP and MH composites. Tensile tests were also carried out with all samples. It was found that the tensile strength of the composites dropped by 35% as the KF content increased from 10 to 25 wt%. As KF wt% increased, poor compacting in the composite caused weaker bonding strength between the matrix and the fiber. Thus, resulting in a decline in tensile strength. Also, high fiber fillings in the samples resulted in inadequate fiber wetting, impacting load transfer structure in the composite negatively, and leading to low strength [9].

There is very little literature on the combination of carbon and KF supplemented with nanofillers. This article evaluates the hardness, impact strength, compressive strength, coefficient of friction, and wear rate experimentally. Then, the optimum wt% of reinforcements were found for automotive components.

2 Materials and experimental details

2.1 Materials

The phenolic composite consists of different constituents to achieve wear performance. Novolak phenolic resin is used as the matrix for this polymer-based phenolic composite sample. A powdered form of phenolic resin is used for achieving homogeneous mixtures in fabrication. Cashew dust is used as functional filler material, barium sulphate is used as a filler agent, and short carbon fiber, and KF which were chopped to 10–15 mm are used as reinforcements. Table 1 shows the details of the selected

Table 1: Details of materials used in this study

Constituents	Density (g/cm^3)	Young's modulus (GPa)	Tensile strength (MPa)	Melting point temperature (°C)	Suppliers	Ref.
Carbon fiber	1.8	200–500	3,000–7,000	280	Go green, Chennai	[27,28]
KF	1.34–1.45	33.8 ± 3.9	577 ± 71	160	Go green, Chennai	[29,30]
Phenol-formaldehyde Novolak powder resin	0.3–0.6	8	55	110–116	Ankur Rasayan, Chennai	[31,32]
Barium sulphate	4.5	—	—	1,580	Surya Color Chem, Bengaluru	[33]
Cashew dust functional filler	0.590	—	—	280	Satya cashew chemicals, Chennai	
MWCNT	0.04	—	—	—	BTCORP Generique Nano Pvt Ltd. Bengaluru	
CNC	0.3	—	—	—	Nanografi, Turkey	

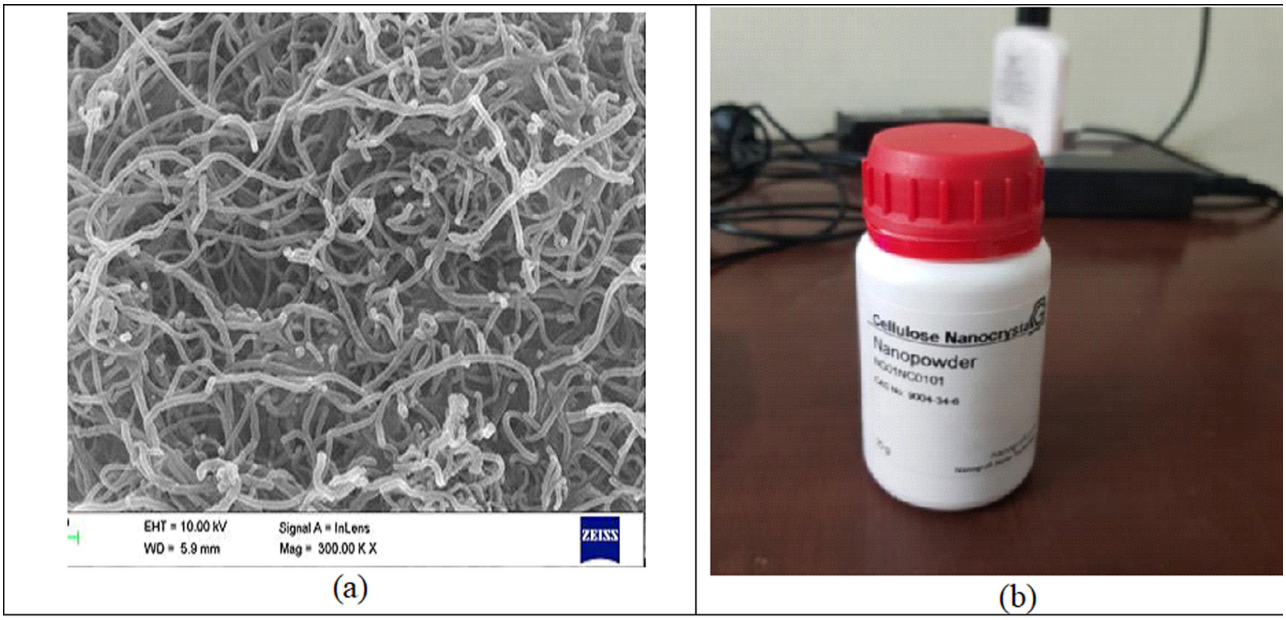

Figure 1: (a) SEM image for MWCNT; (b) procured CNCs.

constituents for the phenolic composite. Figure 1 shows the (a) SEM image for MWCNT, and (b) the procured material of CNC.

2.1.1 Formulation of phenolic composites

The phenolic composite specimens are fabricated by a hot press compression molding process. Three phenolic composites were fabricated by hot press molding by varying the weight percent of the reinforcements and with the inclusion of Nanofillers. From the literature, 30 wt% phenolic resin + 65 wt% periwinkle shell particles were applied and investigated the phenolic composite in automobile applications [34]. The wear behavior was investigated for up to 60 wt% of phenolic resin-incorporated composite material using organic-based composite materials [35].

Table 2: Formulation of the fabricated samples

	Sample 1 (wt%)	Sample 2 (wt%)	Sample 3 (wt%)
Phenolic resin	40	40	40
Carbon fiber	40	20	20
KF	—	20	20
$BaSO_4$	10	9.5	9.5
Cashew dust	10	10	10
MWCNT	—	0.5	—
CNC	—	—	0.5

Table 2 shows the formulation for the prepared samples named Samples 1, 2, and 3.

Before fabrication, mixing was carried out by the ball milling method. First, the powdery materials (phenolic resin, $BaSO_4$, Cashew dust, MWCNT, and CNC are mixed for 1 h at 400 rpm. Then, the chopped fiber is added and mixed for 1 h to get a homogeneous blend [36]. A mild steel mold is prepared to fill this homogeneous mixture and compress it in the hot press. Figure 2 shows the hot press machine with the operated parameters. A 30-ton hot press machine is used in this study.

After the mixture is filled, the mold is pressed at 100 bar at 150°C for 10 min [37]. An aluminum foil is placed above and below the substance to prevent sticking of material. Then curing is done in a hot air oven for 6 h [38]. Figure 3 shows the schematic diagram of the fabrication process.

The above procedure is done for the other two samples and labeled as Samples 1, 2, and 3. The samples are prepared for dimensions of 150 × 150 × 5 mm. Figure 4 shows the fabricated sample after taking out from the mold. Now, this sample is cut into test specimens according to the ASTM standards.

2.2 Characterization

The physical, mechanical, and tribological properties are investigated for Samples 1, 2, and 3.

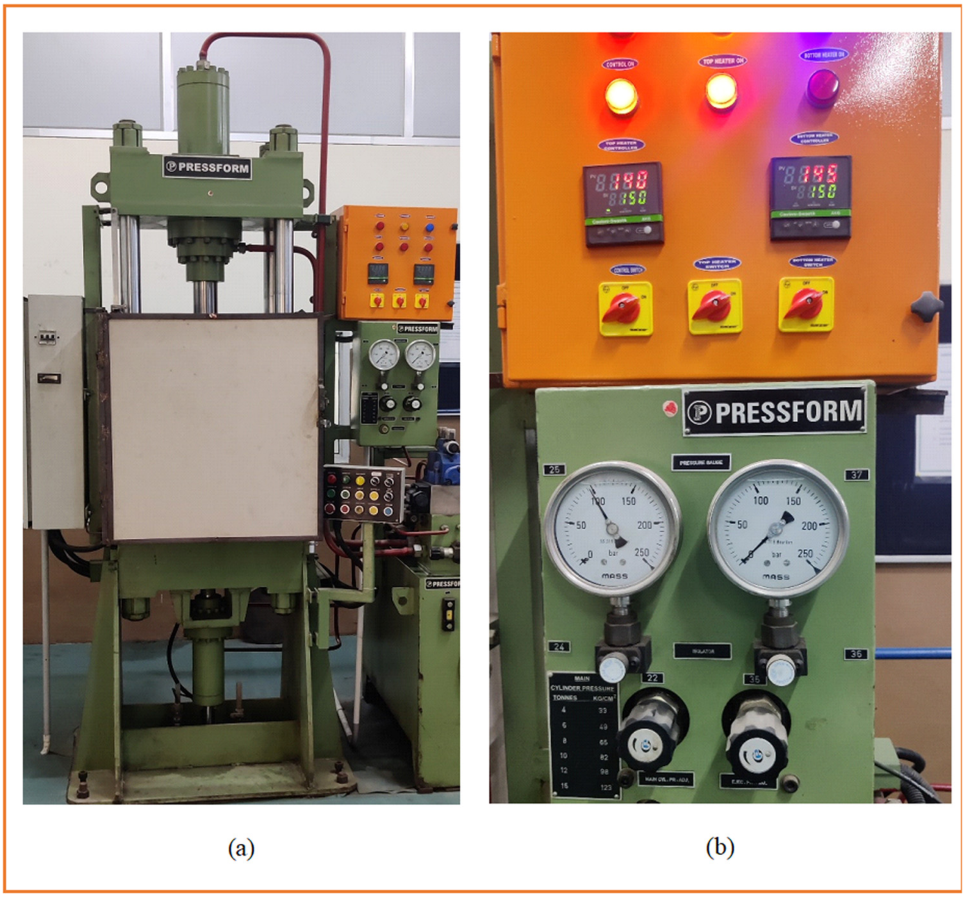

Figure 2: (a) Hot press machine; (b) control setup with operating parameters in a hot press machine.

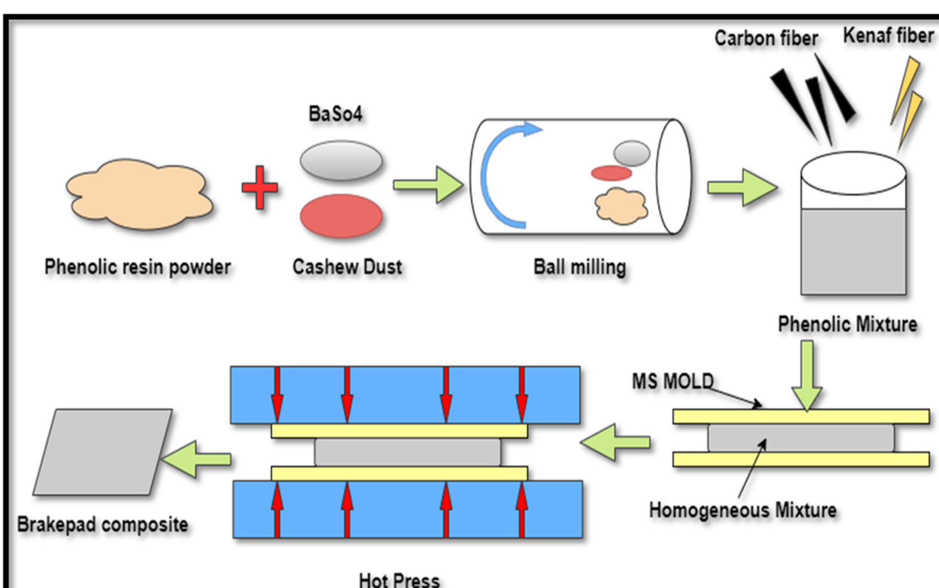

Figure 3: Schematic diagram of the fabrication of carbon/KF-reinforced phenolic composites.

Figure 4: The prepared phenolic composite specimen.

2.2.1 Physical properties

2.2.1.1 Hardness test

The hardness of the phenolic composite samples is found using the Rockwell Hardness testing machine. The test was carried out according to the standard ASTM E18.'R' scale hardness is used for testing, as the samples are polymer-based. A load of 60 kgf is applied with a 12.7 mm steel ball intender. The samples were cut into 20 × 20 mm, and hardness was measured at five different points of the specimen.

2.2.2 Mechanical properties

2.2.2.1 Impact strength

Charpy impact testing was done on the phenolic composite samples using the Pendulum impact testing apparatus. The test was carried out on the standard ASTM D256 with dimensions of 120 × 12 × 10 mm^3. The test aims to assess a material's capacity to endure a high-rate load. The dial on the machine shows the energy absorbed by the specimen at the point of breaking. Five samples were tested for each composition, and the average impact strength is calculated using equation (1).

$$\text{Impact strength} = \frac{\text{Impact energy(kJ)}}{\text{Area(m}^2\text{)}}. \qquad (1)$$

2.2.2.2 Compressive strength

The compressive strength test was conducted on UTM (Universal Testing Machine) INSTRON 8801. The test was conducted by ASTM D6641 with rectangular specimen dimensions of 145 × 15 × 5 mm. The sample gauge length was maintained at 40 mm, and the crosshead speed was given as 1 mm/min. The mean compressive strength for the samples was determined.

2.2.3 Tribological properties

2.2.3.1 Pin-on-disc wear test

The wear loss and Coefficient of friction in the phenolic composite samples are measured using a Pin-on-disc tribometer. The test was performed according to the ASTM G99 standard. The samples were cut in the shape of a disc with a diameter of 55 mm and a counter shank hole of 6 mm for fixing it. The pin used is a standard material of hardened bearing steel (AISI 52100) with a diameter of 6 mm and a height of up to 40 mm.

The load applied on the sample was 60 N with an rpm of 480 rpm. A time period of 500 s was taken to calculate the wear rate and coefficient of friction.

3 Results and discussion

3.1 Rockwell hardness test

In this test, a steel ball intender is forced into the material's surface under specific conditions. The type of intender material also depends upon the specimen. For thermoplastics, the 'R' scale is used with a load of 60 kgf and 1/12″ in the indenter [39]. The apparatus measures the difference in depth of indentation as the force on the intender is increased from the initial load to a specified applied load and then returned to the initial load. Table 3 shows the Rockwell hardness for the prepared phenolic composite samples.

Table 3 shows that hardness increased with the addition of KF and nanofillers. Samples 2 and 3 show an increase in hardness value by 20 and 18%, respectively. The Rockwell harness at a point on the part of the sample does not represent the physical characteristics of the whole

Table 3: Hardness test values of different wt% of samples

Sample label	wt%	Hardness (HRR)
Sample 1	C 40%	54.33
Sample 2	C20/K20%/MWCNT	65
Sample 3	C20/K20%/CNC	64

sample. Hence, five specimens of each sample were taken to evaluate the hardness and the mean value was taken. Thus, it is observed that natural KF shows good physical properties and can be a good replacement for carbon fiber with Sample 2 having the highest hardness value.

3.2 Impact strength test

The test determines the resistance of materials to breakage by flexural shock as indicated by energy extracted from the standard pendulum-type hammers mounted in the Charpy testing apparatus. The impact energy obtained in Joules for all the samples is shown in Table 4.

From Figure 4, it is visible that impact strength is higher for increased carbon fiber content. Sample 1 with C 40% has a higher impact strength of 51% than Sample 2. From Figure 5, Sample 2 and Sample 3 do not show much difference in their impact strength. Thus, it can be inferred that this reduction in impact strength might be due to the replacement of carbon fiber with natural fiber.

The pictures of shattered samples under impact load obtained from FE-SEM fracture analysis are presented in Figure 6. Sample 1 (C 40%) phenolic composite including carbon fiber, phenolic resin, $BaSO_4$, and cashew sawdust is shown in Figure 6(a). In Figure 6(b), there is Sample 2 (C20/K20%/MWCNT) phenolic composite with the observed, following components: carbon fiber, KF, phenolic resin, $BaSO_4$, and cashew sawdust. Sample 3 (C20/K20%/CNC) of the phenolic composite is shown in Figure 6(c). It contains the following compositions: cashew sawdust, CNC, phenolic resin, KF, carbon fiber, and $BaSO_4$. However, Sample 1 exhibited the highest impact strength when compared to Samples 2 and 3 because of the strong bonding between the carbon fiber, phenolic resin $BaSO_4$, and cashew sawdust. $BaSO_4$ and phenolic resin stopped the fiber from debonding. Compared to synthetic fiber reinforced composites, Samples 2 and 3's natural fiber (kenaf)-based composites show a somewhat reduced mechanical strength. However, matrix modification using nanofillers such as MWCNT and CNC would increase the mechanical strength and stiffness in Samples 2 and 3.

Table 4: Charpy test values of the prepared phenolic composite samples

Energy absorbed (in Joules)			
	Sample 1	**Sample 2**	**Sample 3**
Trials	C 40%	C20/K20%/MWCNT	C20/K20%/CNC
1	8	5.5	6
2	8	5	6.5
3	9	6	6
Mean	8.333	5.5	6.167

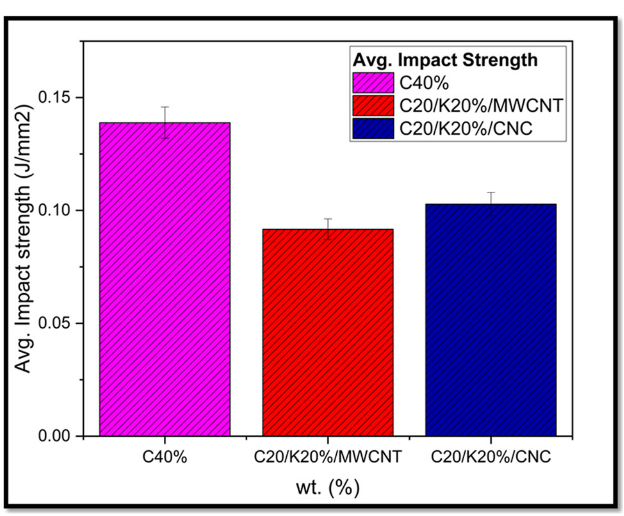

Figure 5: Effect of the addition of KF and nanofillers on impact strength on Samples 1, 2, and 3.

3.3 Compression test

In this test method, compressive properties like compressive strength and modulus of elasticity are found [40]. A combined loading fixture is used in the UTM for this test method. The compressive force is introduced by end loading and shear loading. Table 5 shows all the obtained compressive strength values for the samples. Figure 7 shows the fractured sample after the test.

The compressive strength of Sample 1 was measured to be 43.86044 MPa, which is found to be four times greater than the compressive strength values observed for Samples 2 and 3. The strength of the material is diminished as a result of incorporating kenaf, a natural fiber with relatively limited mechanical properties. Hence, the effective observation of Nanofillers' impact is also limited. Figure 8 shows the compressive strength values in MPa for the samples. In this analysis, 40% carbon fiber was used in Sample 1, which is a synthetic fiber material. The carbon fiber has a tensile strength of 3,000–7,000 MPa. Moreover, Samples 2 and 3 contain 20% carbon fiber and 20% KF, where the kenaf has a tensile strength of 571–640 MPa. However, the nanofillers are MWCNT and CNC-influenced composites to improve the compressive strength, which declined to the expected level. Due to these analyses, sample 1 has a high compressive strength compared to other samples. In a similar vein, it is

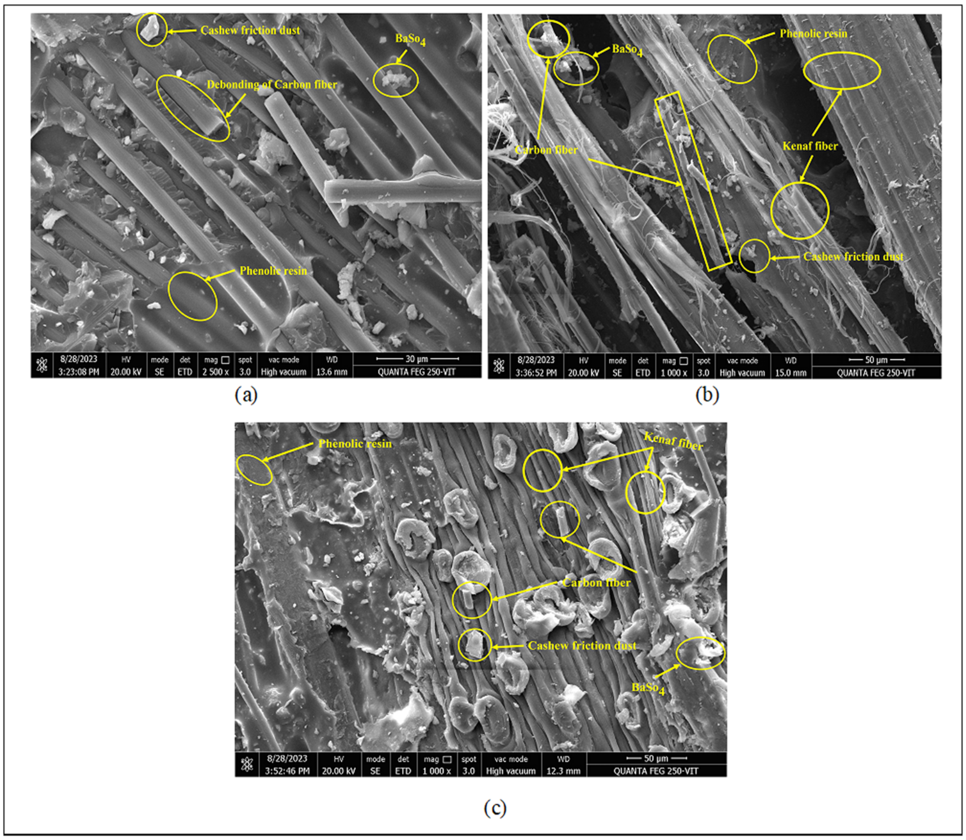

Figure 6: FESEM microstructure analysis of fractured specimens under impact loading: (a) C 40% (Sample 1), (b) C20/K20%/MWCNT (Sample 2), and (c) C20/K20%/CNC (Sample 3).

Table 5: The compression test results for the phenolic composite specimens

Specimen label	wt%	Maximum comp. load (N)	Compressive strength (MPa)	Modulus (MPa)
Sample 1	C 40%	3289.53271	43.86044	1761.21896
Sample 2	C20/K20%/MWCNT	646.46008	8.61947	1649.26148
Sample 3	C20/K20%/CNC	713.74176	9.51656	1201.56469

worth noting that Samples 2 and 3, which incorporate KF, exhibit significantly lower compressive strength when compared to the entirely synthetic carbon reinforcement. It can be posited that the bonding between KF and the phenolic resin matrix is comparatively weaker.

3.4 Pin-on-disc wear test

The amount of wear is determined by respectively weighing both before and after the test. The coefficient of friction is also measured using the frictional force obtained in the wear results. Table 6 shows the given parameters and the obtained results, while Figure 9 shows the worn specimen in the apparatus.

It has been observed that Sample 2 exhibits the least wear loss and high coefficient of friction. The higher hardness value in Sample 2 can also be correlated to the low wear loss [14,41]. This lower wear loss may be attributed to the homogeneous dispersion of carbon/kenaf MWCNT nanofiller to produce a more stable structure [16]. Moreover, MWCNT may occupy the pores in the composite structure due to which the coefficient of friction has been improved. A similar trend was observed by Rangaswamy *et al.*, whereas the addition of MWCNT in hybrid epoxy composites improved the wear resistance compared to the pure composite samples [42].

Figure 7: Effect of the addition of KF and nanofillers on impact strength on Samples 1, 2, and 3.

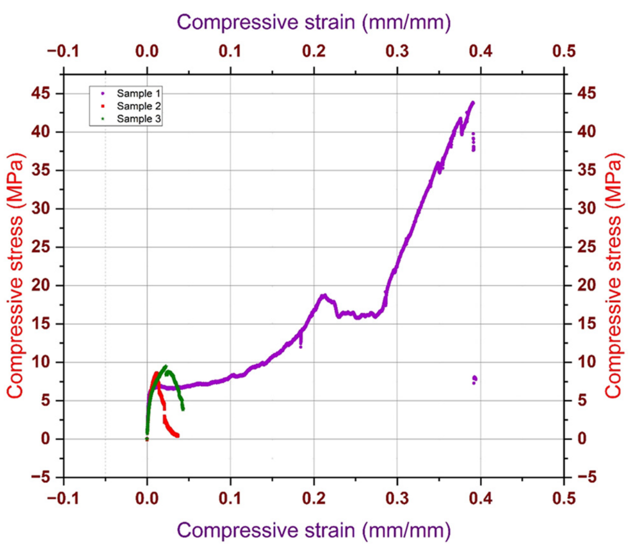

Figure 8: Comparison of compressive stress curve for all three samples.

Venkategowda *et al.* have investigated the wear behavior of only KF-reinforced epoxy composites with different wt% [43]. However, the present research has focused on replacing carbon fiber with KF in MWCNT-modified epoxy composites. It has been observed that replacing 20 wt% carbon fiber with KF in MWCNT-modified composites exhibited higher COF (Figure 10) and has paved the way toward sustainability and Net Zero. Figure 11 shows the wear in micrometers for the pure carbon and hybrid composites.

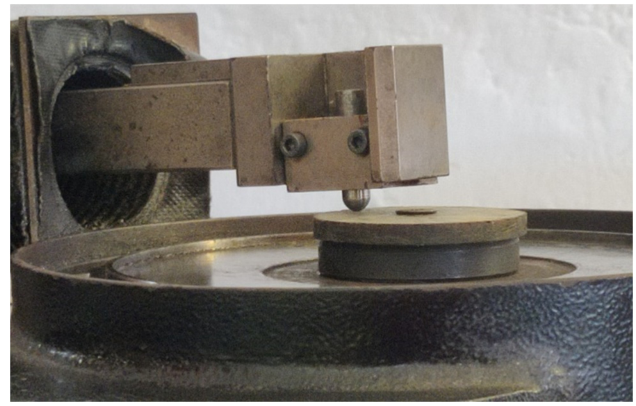

Figure 9: Pin-on-disc apparatus.

4 Conclusion

In this study, carbon/KF-reinforced phenolic-based composites were fabricated by hot compression molding. The effect of adding nanofillers (MWCNT and CNC) on the physical, mechanical, and tribological properties was investigated.

- The hardness values of Samples 2 and 3 show an increase of 20 and 18%, respectively. Thus, it is observed that even after replacing carbon fiber with natural KF, it is shown to have good physical properties and can be a good replacement for carbon fiber with Sample 2 having the highest hardness value.
- The impact strength is higher for increased carbon fiber content. Sample 1 with C 40% has a higher impact strength of 55% than Sample 2. Samples 2 and 3 do not show much difference in their impact strength. Thus, it can be inferred that this reduction in impact strength might be due to low interfacial strength due to the random distribution of KF and insufficient fiber wetting with the resin.

Table 6: Wear loss and coefficient of friction results of the phenolic composite samples

Samples	wt%	Initial weight (g)	Final weight (g)	Wear loss	Coefficient of friction
1	C 40%	17.79	17.758	0.032	0.259792
2	C20/K20%/MWCNT	15.23	15.207	0.023	0.305673
3	C20/K20%/CNC	19.036	19.002	0.034	0.173646

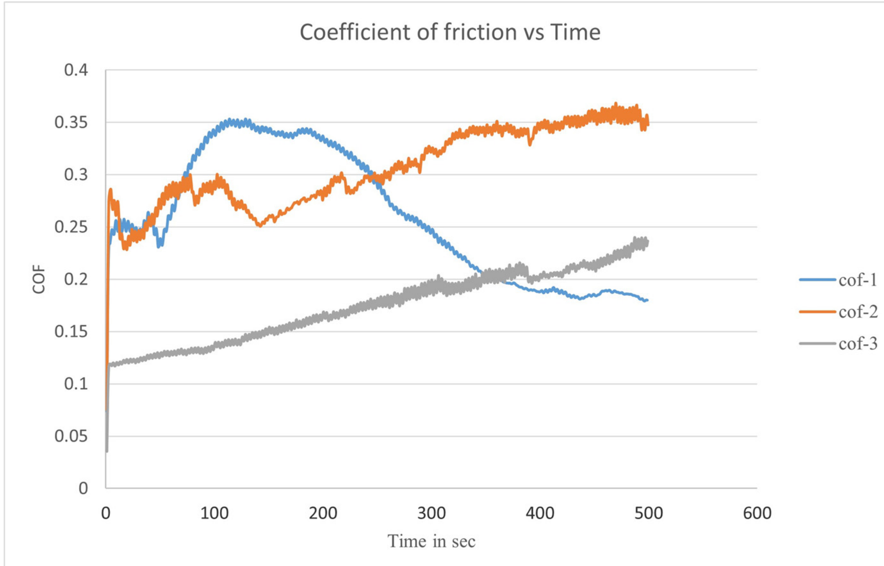

Figure 10: Comparison of coefficient of friction *vs* time graph of all 3 samples.

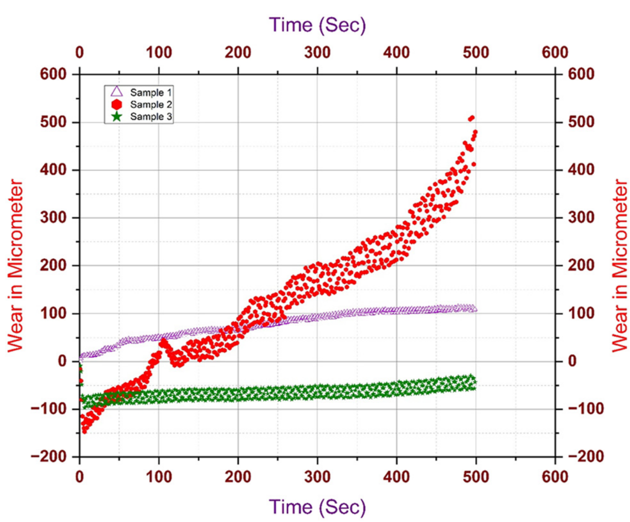

Figure 11: Computer-generated wear data representing wear in micrometer for all three samples.

- The compressive strength of Sample 1 was 43.86044 which has four times higher strength than Samples 2 and 3. The strength in Sample 2 is reduced due to the addition of natural fiber – kenaf, which shows low mechanical properties. Thus, the effect of nanofillers also cannot be observed in KF samples effectively. As KF wt% increased, poor compacting in the composite led to a decrease in compressive strength.
- Wear test results showed that the least wear loss and a higher coefficient of friction are observed in Sample 2 than in Samples 1 and 3. The higher hardness value in Sample 2 can also be correlated to the low wear loss. Sample 3 also produces similar wear loss as sample 1 after the replacement of carbon fiber with 20% KF indicating tribological characteristics of KF. Wear resistance is good in kenaf long fiber-reinforced polymer matrix composites. Automobile components and other tribological components can benefit from these composites.
- Hence, Sample 2 (20%carbon/kenaf) with MWCNT provides better tribological properties. This inference can be used for further studies with also need to study the thermal behavior for fade characteristics.

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