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Original Article

Utilization of waste dolomite dust in carbon fiber reinforced vinylester composites

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ABSTRACT

The present study was designed to explore the applicability of dolomite dust (a waste from a dolomite quarry) as filler in polymer composites. Therefore, fixed carbon fiber reinforced vinyl ester composites with varying dolomite dust proportions (0, 5, 10 and 15 wt.%) are manufactured and subsequently evaluated for physicomechanical and sliding wear properties. Experimental results indicate that density, void content, impact energy, hardness, tensile modulus, and flexural modulus increase with dolomite content increase. On the other hand, a continuous decrease in tensile and flexural strength was observed with increased dolomite dust content. The sliding wear tests were performed using L_{16} orthogonal design, considering four parameters: dolomite dust, sliding distance, applied load, and sliding velocity according to the Taguchi method. It was found that dolomite dust was the most noteworthy parameter of wear rate, followed by applied load and sliding distance.

1. Introduction

Carbon is one of the essential elements known to humankind and present everywhere on this earth. One of the most significant features of carbon is that it can be easily structured in a long chain and form almost two million compounds [1]. Therefore, carbon in various shape and size become a suitable material for high-performance applications that have extensive use in life [2]. One of the applications of carbon is in polymer composites, where carbon fiber is utilized as reinforcement. The various beneficial attribute of carbon fiber, such as low density, high strength, good dimensional stability, moisture resistance etc., resulted in corrosion-resistant, lightweight, high strength polymer composites with increased strength-to-weight ratio [3]. These properties make carbon fiber reinforced polymer composites a favourable candidate for a wide range of applications such as aerospace, automotive, electronics, medical and sports equipment components [1–4].

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The global demand for carbon fibre-based polymer composites is nearly 160 ktons in 2020 and is expected to reach 200 ktons by 2022 [5]. However, these composites have some disadvantages like high cost, low breaking elongation, and low wear and impact resistance. It has been reported that the addition of inorganic and organic fillers can improve the drawbacks associated with carbon fiber reinforced polymer composites [6,7].

Nowadays, significant environmental awareness and stringent policies on municipal, agricultural and industrial waste dumping have encouraged material scientists to up cycle these wastes into innovative applications [8,9]. The application of these wastes in polymer composites lowers the manufacturing cost and helps reduce the environmental burden. Along with that, waste included composites are and being used in many applications [10]. Research by H. Liu et al. [11] explores the sliding wear performance of cenosphere-filled carbon fiber reinforced polyether ether ketone composites. They claimed that composite with 10 wt.% cenosphere exhibits superior wear resistance when compared to the other composites. The study of mechanical and wear behaviour of cement by-pass dust particulate filled carbon fiber-vinyl ester established enhancement in hardness and impact strength, whereas the tensile strength and flexural strength are found to deteriorate. The study also suggested that 12 wt.% cement by-pass dust reinforced vinyl ester gives the best wear resistance performance [12]. Y. Luna-Galiano et al. [13] designed geopolymer composites using blast furnace slag and carbon fiber waste. Authors claimed higher thermal resistance, increased porosity, and reduced compressive strength for increased blast furnace slag to carbon fiber waste concentration. B.M. Reddy et al. [14] developed granite powder-filled *Cordia dichotoma* fiber reinforced hybrid polyester composites. Results concluded that hybrid polyester composites exhibited better mechanical properties (flexural, tensile and impact strengths) when granite powder was 15 wt.%. G. Kalusuraman et al. [15] revealed that the erosive wear resistance of jute fiber reinforced polyester composites was increased with copper slag waste. V.A. Prabu et al. [16] extensively studied the impact of various industrial wastes on the mechanical properties of polymer composites. The authors concluded that not only the type and amount of waste but also the types of polymer matrix have a significant impact on the evaluated properties of composite. The conch shell waste addition was also reported to increase the impact energy, tensile strength and glass transition temperature of glass fiber reinforced epoxy composites by K.V. Kumar et al. [17]. Similarly, fruit waste [18], decayed wood waste [19], *Limonia acidissima* shell waste [20], mollusk shell-waste [21], waste tea leaf fiber [22], *Moringa oleifera* discarded waste fiber [23], cocous nucifera sheath waste [24], were also used as reinforcement and reported to demonstrate beneficial impact on various performance properties of polymer composites.

Dolomite quarry's waste product, dolomite dust, is produced in large quantities and easy to get at a low price. Compositionally dolomite dust contains a substantial amount of calcium oxide, aluminium oxide, and silicon dioxide, which is already being used to develop various polymer composites [25]. It is used in multiple applications, including the catalyst for biomass gasification, clinker composites, tiles and glass manufacturing, magnesium production, construction, and sorbents for metal cations [25–28]. Therefore, the effective utilization of dolomite dust as filler in polymer composites makes it profitable and helps minimize our dependency on traditionally used materials. S.S. Md. Saleh et al. [29] revealed that micro-hardness and thermal conductivity of phenolic composites increased with the addition of hybrid carbon nanotube and dolomite fillers. S. Yan et al. [30] developed dolomite filled geopolymer matrix based composite foams. The developed composite foams showed high open porosity with good mechanical strength. The impact of dolomite concentration on the mechanical properties of natural fiber based low-density polyethylene composites was investigated by A.S. Ahmed et al. [31]. The authors concluded that with dolomite inclusion, the composites' impact strength, tensile strength, and tensile modulus improved considerably. Besides, improvement in mechanical properties of polyether-based polyurethane composite with the addition of dolomite was reported by A. Vazid et al. [32]. The influence of dolomite on physicomechanical properties of natural-synthetic hybrid fiber-based epoxy composites was investigated by S.K. Verma et al. [33]. Results concluded that hybrid epoxy composites exhibited increased hardness and impact energy while composite strength deteriorated with increased dolomite amount. The outcomes of the studies above suggested that waste dolomite dust can be utilized as filler to create fiber reinforced polymer composites with unrivalled mechanical and wear properties. Hence, in this work, carbon fiber reinforced vinyl ester composites with varying proportions of dolomite dust have been fabricated and assessed for physicomechanical and sliding wear properties. The sliding wear tests were performed on a pin on disc machine using ASTM G99 standard. The wear study was designed using Taguchi methodology to find the impact of dolomite dust concentration (0, 5, 10 and 15 wt.%), applied load (15, 30, 45 and 60 N), sliding velocity (1, 2, 3 and 4 m/s) and sliding distance (1000, 2000, 3000 and 4000 m) on the specific wear rate of manufactured composites.

2. Experimental details

2.1. *Materials and composite fabrication*

Vinyl ester (VE 700; density = 1.05 g/cm^3) was supplied by Excellent Resin Meerut, India. Woven carbon fiber was provided by New Era Composite Chennai (density = 1.39 g/cm^3). Dolomite dust was collected from Bharat Crusher Mahoba U.P. India, and particles passed through 80 mesh sized sieves were used to develop composites. The scanning electron microscope (SEM) image and energy-dispersive spectroscopy (EDS) spectrum of dolomite dust are presented in Fig. 1.

The composites were developed using the hand lay-up technique by varying dolomite dust as 0, 5, 10 and 15 wt.% in fixed (10 wt.%) carbon fiber in vinyl ester resin as presented in Table 1. The hand lay-up moulding technique was used for

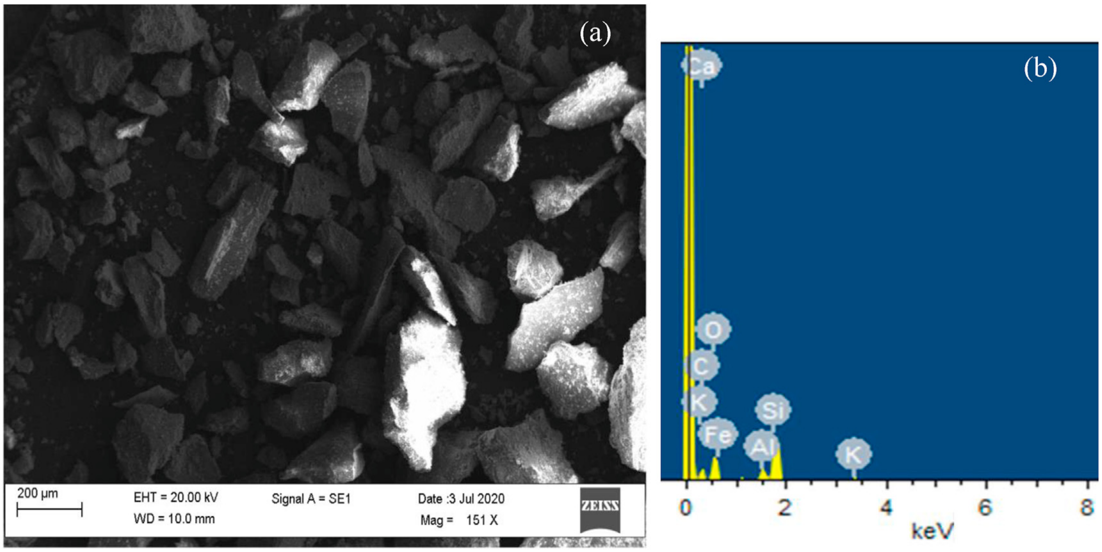

Fig. 1 – (a) SEM image, (b) EDS of dolomite dust.

composite fabrication, as presented in Fig. 2. First, a mixture of vinyl ester, cobalt Naphthenate (accelerator, 1.5 wt.%) and methyl-ethyl-ketone-peroxide (hardener, 1 wt.%) was prepared. The mixture is then stirred mechanically for 5 min to obtain the required homogeneity.

The prepared mixture was then filled with dolomite dust and stirred using a magnetic stirrer for 10 min. After that, the prepared mixture of resin and dolomite dust was poured inside the mould size of 200 × 200 mm^2 before impregnating the first layer of fiber. The impregnation settles down the fibrous mat at the bottom of the mould. The process followed with pouring the mixture of resin and hardener on the first layer of the fibrous mat. The mixture was spread uniformly over the mat with the help of a roller and brush. The mat of carbon fiber was then placed over the surface, followed by the pouring of the mixture. Care was taken to avoid sticking the composite to the mould surface by spraying the releasing agent. A static load of 25 kg was applied over the stacking at ambient temperature and left for curing for 24 h. After that, the composites were machined in required dimensions to study physical, mechanical and wear properties.

2.2. *Physicomechanical characterization*

Experimental and theoretical densities were measured using the water immersion technique and rule of the mixture, while void content was determined using ASTM D 2734 protocol. The tensile properties were in a universal testing machine (UTM, HEICO India) according to ASTM D 368 for sample size of 165 mm × 12 mm × 10 mm, with a crosshead speed of 10 mm/min. Flexural properties were measured as per ASTM D790 protocol for sample size 100 × 10 mm × 10 mm with a crosshead speed of 2 mm/min. For hardness measurement, tests were performed on the Rockwell hardness tester (Fine Manufacturing Industries, India) according to ASTM D 785. Impact energy was assessed using an impact testing machine (TINIUS OLSEN, USA) for a sample size of 60 mm × 10 mm × 5 mm according to ASTM D 256. The results reported are the average of five measurements carried out at room temperature.

2.3. *Sliding wear study*

The sliding wear study of the manufactured dolomite dust-filled carbon fiber-based composites was performed using a pin-on-disc test rig from Ducom, India, using ASTM G 99 protocol. The schematic of the pin-on-disc machine is presented in Fig. 3, while the detailed working was discussed elsewhere [34]. The specimen sized 60 mm × 10 mm × 10 mm machined from the manufactured composites and held stationary inside the fixture, which was normal to the disc. The sliding wear tests were designed for selected parameters of sliding distance, applied load, dolomite dust content and sliding velocity. After that, the weight of the composite specimen was measured after and before the sliding wear experiment using an electronic balance (Wensar Weighing Scales Ltd., India). Each wear trial was repeated three times to get the statistical deviation. The wear was presented in terms of specific wear rate (SWR) using the following equation [34]:

$$\mathrm{SWR} = \frac{W_I - W_2}{\rho \times S \times \omega} \quad (\mathrm{cm}^3/\mathrm{Nm}) \qquad (1)$$

where, W_1=Specimen weight before test (g); W_2=Specimen weight after test (g); ρ = Specimen density (g/cm^3); S=Sliding distance (m); ω = Applied load (N).

Table 1 – Detailed description of composites.

Sample	Composition (wt.%)		
	Vinylester	Carbon fiber	Dolomite dust
S0	90	10	0
S1	85	10	5
S2	80	10	10
S3	75	10	15

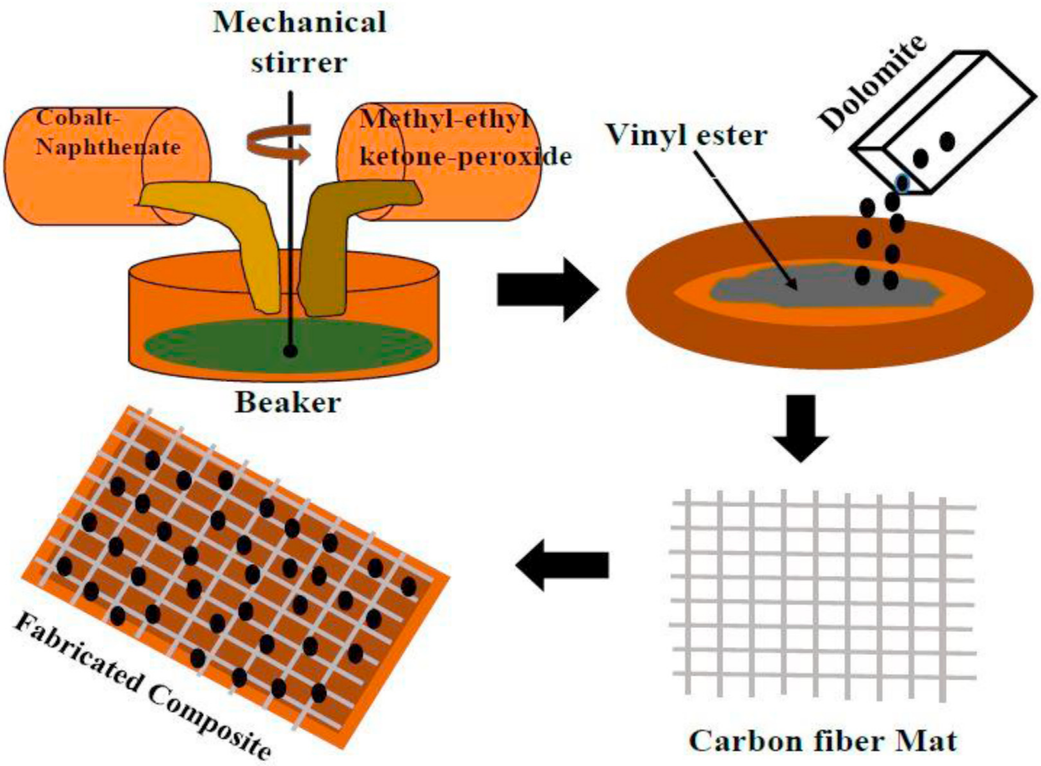

Fig. 2 – Details of fabrication process.

2.4. *Experimental design*

The experimental design was structured based on L_{16} orthogonal array using the Taguchi method to assess the influence of parameters such as dolomite dust (0, 5, 10 and 15 wt.%), sliding velocity (1, 2, 3 and 4 m/s), applied load (15, 30, 45 and 60 N) and sliding distance (1000, 2000, 3000 and 4000 m) on sliding wear performance of the manufactured composites

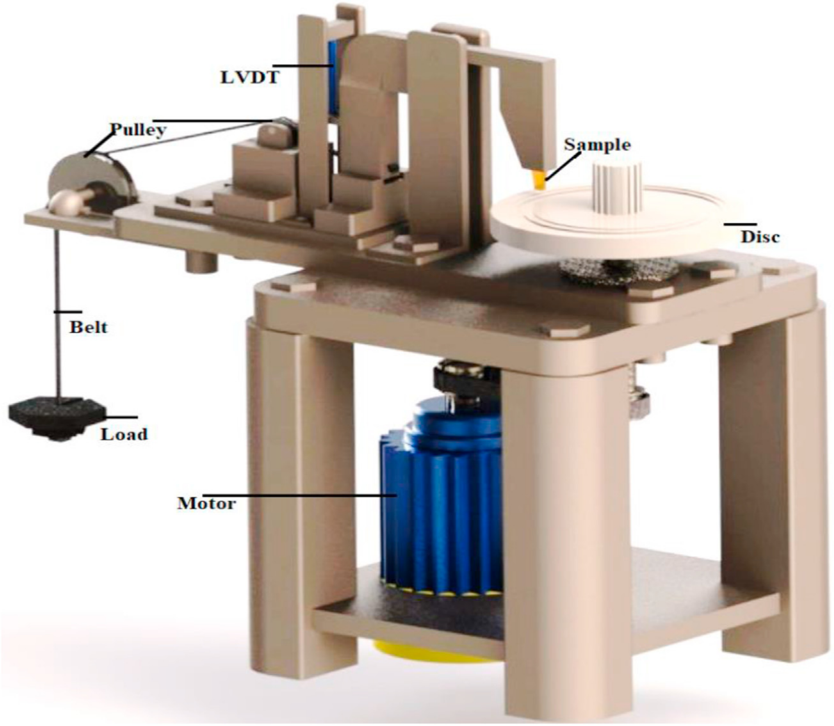

Fig. 3 – Schematic illustration of a pin-on-disc machine.

Table 2 – Design parameters and level range.

Parameters	Levels				Units
	I	II	III	IV	
A: Dolomite dust	0	5	10	15	wt.%
B: Applied load	15	30	45	60	N
C: Sliding velocity	1	2	3	4	m/s
D: Sliding distance	1000	2000	3000	4000	m

as listed in Table 2. As per the L_{16} orthogonal array, in total, sixteen experiments were performed with the selected parameters and levels as listed in Table 3.

In order to reduce the experimental error rate, three trials were executed for every experiment. After that, a specific wear rate was calculated for each experiment using Eq. (1). The best wear result was obtained by determining the signal-to-noise (SN) ratio for each experimental output where signal refer to desired output while noise refers to undesired factors. In order to obtain the best results, Taguchi methodology has three types of SN ratio, namely the lower-the-better, nominal-the-better and higher-the-better. In this study, the target is to reduce the wear of the manufactured composites; hence 'lower-the-better' characteristic was used as [34]:

$$SN\ ratio = -10\log\left[\frac{1}{n}\left(\sum \alpha^2\right)\right] \tag{2}$$

where, α = specific wear rate; n = number of experiments.

3. Results and discussion

3.1. Physicomechanical properties

The measurements of the manufactured composites' densities (experimental and theoretical) and void content are presented in Fig. 4.

Both void content and densities were found to increase with increased dolomite dust concentration. This increase in densities is expected as low, dense vinyl ester resin (density = 1.05 g/cm^3) was replaced with high dense dolomite dust (density = 2.84 g/cm^3). As seen in Table 4, there was an increase in the void content of the composites upon increasing the amount of dolomite dust from 0 to 15 wt.%. The lowest (1.02%) and highest (3.86%) void content was observed for S0 and S3 composites. This trend in void content may be ascribed to the increased concentration of dolomite dust which might bring structural inhomogeneities due to its agglomeration and improper distribution at a higher proportion in the vinyl ester matrix. The results for the hardness of the tested composites are shown in Fig. 5.

The composites' hardness fluctuated between a small range of 110.75–114.30 HRL and was found to increase with increased dolomite dust content. With the addition of 5–15 wt.% dolomite dust, i.e. S1/S2/S3 composites, the hardness value was observed to increase by ~1–3% compared to the S0 composite. Similar results for increased hardness with increased filler content were reported [32]. H. Liu et al. [11] have said that the hardness of carbon fiber reinforced polymer composites increased gradually when the introduced cenosphere filler rose from 5 to 20 wt.%. The influence of dolomite dust on the impact energy performance of carbon fiber composite is depicted in Fig. 5. Significant improvement in composite impact energy was noticed with the addition of dolomite dust particles. The impact energy of the S0 composite was found to increase from 1.92 kJ to 2.25 ± 0.1 kJ for ≤10 wt.%, and ~2.50 kJ for 15 wt.% dolomite dust-filled composites, respectively. The decreased inter-particle spacing with the presence of stiffer dolomite dust particles resulted in increased hardness impact energy of the composites [35]. S.K. Verma et al. [33] reported that the impact energy of synthetic-natural hybrid fiber reinforced polymeric composites increased gradually when the amount of introduced dolomite filler rose from 5 to 15 wt.%. The tensile strength and the tensile modulus of unfilled and the dolomite dust-filled carbon fiber reinforced composites are shown in Fig. 6.

The inclusion of ≤10 wt.% dolomite dust caused a decrease of ~21 ± 4% in tensile strength compared to the S0 composite and decreased by ~39% with a further increase in dolomite dust to 15 wt.%. 15 wt.% dolomite dust-filled carbon fiber-based composite (i.e. S3) exhibits a smallest tensile strength of ~162.54 MPa. The trend of decreased tensile strength with

Table 3 – Experimental design.

Exp. no.	Parameters				Exp. no.	Parameters			
	A	B	C	D		A	B	C	D
1	0	15	1	1000	9	10	15	3	4000
2	0	30	2	2000	10	10	30	4	3000
3	0	45	3	3000	11	10	45	1	2000
4	0	60	4	4000	12	10	60	2	1000
5	5	15	2	3000	13	15	15	4	2000
6	5	30	1	4000	14	15	30	3	1000
7	5	45	4	1000	15	15	45	2	4000
8	5	60	3	2000	16	15	60	1	3000

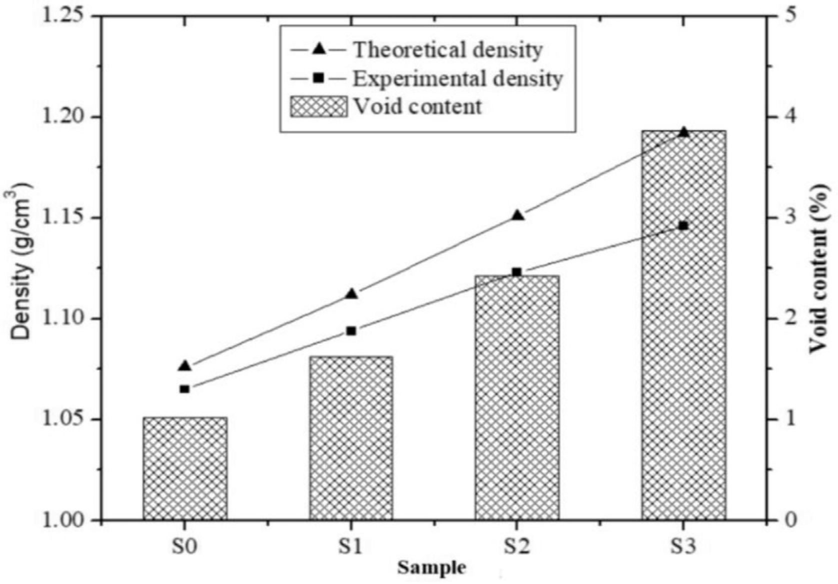

Fig. 4 – Density and void content measurements.

increased filler loading was consistent with the literature. V. Ali and co-workers [32] reported that the tensile strength of polyurethane composite decreased gradually with increased dolomite loading. Patnaik and co-workers [36,37] reported that the tensile strength of fiber-reinforced polymeric composites decreased with an increased concentration of marble dust. Flexural properties (flexural strength and flexural modulus) of the manufactured dolomite dust-filled carbon fiber reinforced vinyl ester composites were measured using a three-point bending test, and the results are depicted in Fig. 7.

For S0 composite (without dolomite dust), the flexural strength was 132.30 MPa and decreased with increased dolomite dust. With the addition of 5 wt.% dolomite dust (S1 composite), flexural strength decreased by ~9% and remained 120.10 MPa. However, beyond 5 wt.% (S2 and S3 composites), dolomite dust inclusion resulted in a 22–32% reduction in flexural strength and remained ~95 ± 7 MPa. The increased concentration of dolomite dust results in resin mobility, resulting in decreased adhesion between the filler and matrix. This decreased binding and increased void content may cause a weak load transfer mechanism between filler and matrix, resulting in decreased flexural strength of the composites [38]. Similar results for reduced flexural strength of fiber-reinforced polymeric composites with increased dolomite (5–15 wt.%) and granite dust (8–24 wt.%) loadings were reported by S.K. Verma et al. [33] and M. J. Pawar et al. [39], respectively.

Unlike tensile strength, the tensile modulus was observed to enhance with the inclusion of increased dolomite dust concentration. It can be observed that the unfilled carbon fiber composite has a tensile modulus of 3.8 GPa, and the value increases to 5.6 GPa, 7.9 GPa and 8.8 GPa with an increase in the dolomite dust weight percentage of 5%, 10% and 15%, respectively. Incorporating 15 wt.% dolomite dust into the S0 composite caused a nearly two and half-fold increase in the tensile modulus from 3.8 GPa to 8.8 GPa. Like tensile modulus, the flexural modulus of the composites is also found to increase with increased dolomite dust loading. It can be observed that the S0 composite (without dolomite dust) has a flexural modulus of 12.26 GPa. An increase of ~6%, ~23% and ~44% in flexural modulus was observed with 5 wt.%, 10 wt.%, and 15 wt.% dolomite dust added composites, respectively. This increase in tensile and flexural modulus with dolomite dust was attributed to its higher stiffness that enhances the modulus value of the composites [32]. Similar observations for increased modulus were reported by S.K. Verma et al. [33] for dolomite (5–15 wt.%) filled and fiber-reinforced polymeric composites. The authors concluded that tensile and flexural modulus of the composites increased gradually when the amount of introduced dolomite filler rose from 5 to 15 wt.%. While studying the influence of marble dust (10–30 wt.%), Patnaik and co-workers [36,37] reported that the tensile and flexural modulus of natural/synthetic fiber-reinforced polymeric composites increased gradually with increased marble dust loading.

3.2. *Taguchi analysis for wear*

Polymer composites have many applications where wear remains a critical issue. Thus, the current study was designed to determine the essential parameters and their combination, which results in the lowest wear rate. The SWR results obtained from the pin-on-disc experiments are used to compute

Table 4 – Response table for SN ratio and contribution of SWR.

Level	A	B	C	D
I	147.2	143.3	146.9	143.7
II	148.2	143.6	145.9	145.3
III	144.8	148.3	144.7	146.7
IV	142.4	147.2	145.1	146.8
Delta	5.8	5	2.2	3.1
Rank	1	2	4	3

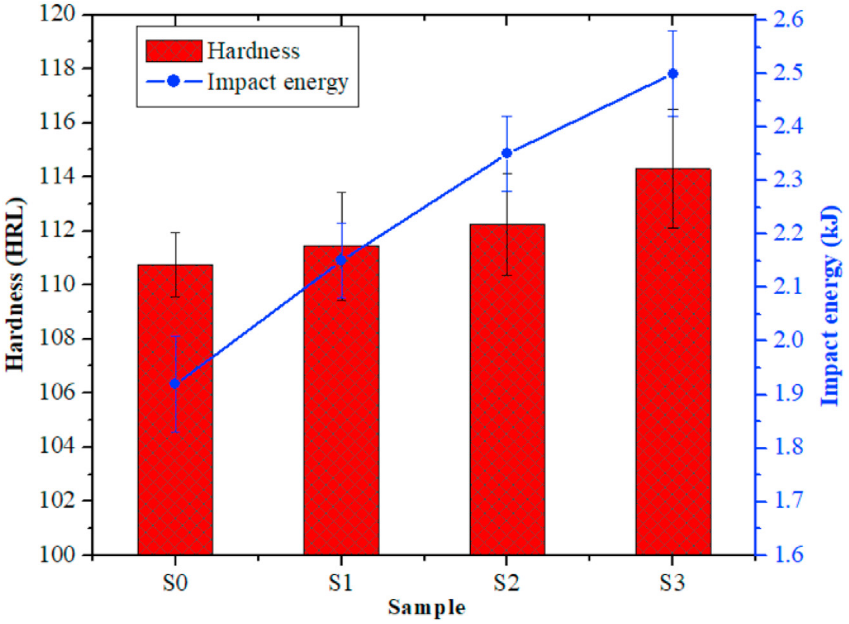

Fig. 5 – **Impact energy and hardness measurements.**

the SN ratio for each experiment using MINITAB 17 statistical tool. The SWR and corresponding SN ratio is presented in Fig. 8. From Fig. 8, it can be seen that 15 wt.% dolomite dust-filled composites exhibited the highest SWR with a value of 1.323×10^{-7} cm^3/Nm for exp. no. 13, while the lowest SWR was 2.899×10^{-8} cm^3/Nm observed for S0 (without dolomite dust) composite when tested under exp. no. 4 conditions. The main effect for SN ratio is plotted in Fig. 9 and presented in Table 4.

As seen in Fig. 9, the SN ratio observes to increase from A1 (0 wt.% dolomite dust) to A2 (5 wt.% dolomite dust) level and to decrease after that (≥10 wt.% dolomite dust). It suggests that the SWR decreases with 5 wt.% dolomite dust and increases with a further increase in dolomite dust. This trend in wear rate may be ascribed to the agglomeration of dolomite dust with increased concentration because of decreased interparticle spacing. The aggregation hampered the wettability and resulted in a weak structure easily damaged during sliding. Moreover, the poor wettability results in easy removal of dolomite dust particles from the composite during sliding and contribute to wear enhancement as found experimentally. The obtained wear results were consistent with the literature. H. Liu et al. [11] investigated the influence of cenosphere filler content (5–20 wt.%) on the tribological properties of carbon

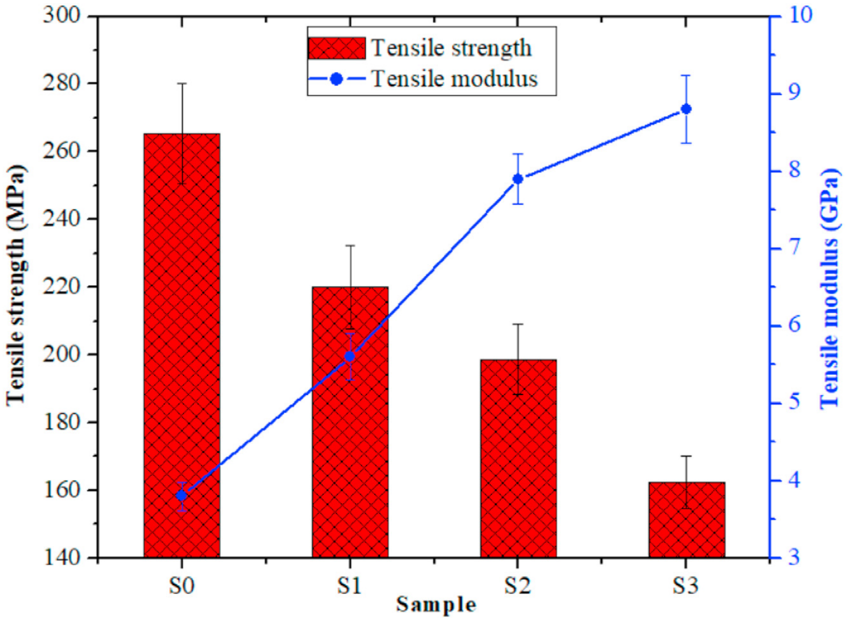

Fig. 6 – **Tensile strength and modulus measurements.**

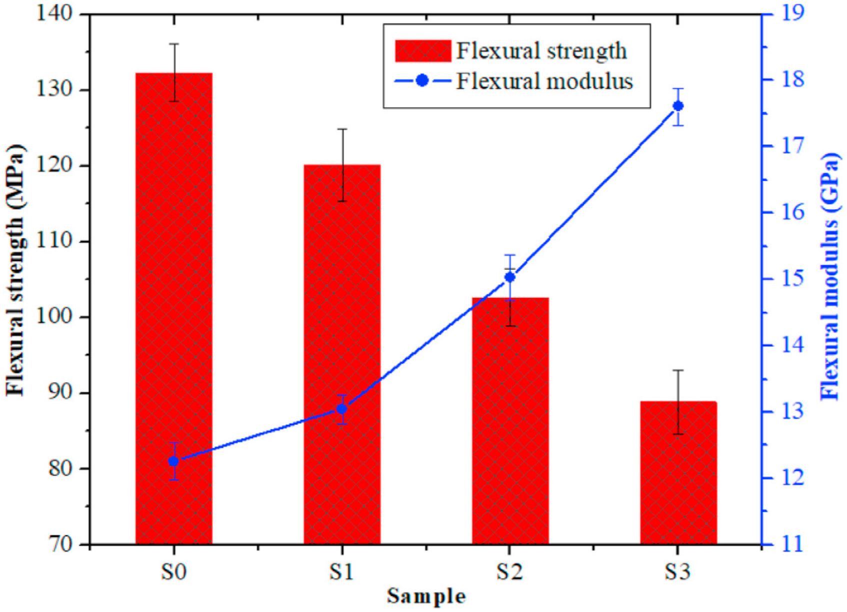

Fig. 7 – Flexural strength and modulus measurements.

fiber reinforced polymeric composites. The author concluded that the specific wear rate remains optimal for lower 10 wt.% cenospheres added composites and increased with further addition of cenosphere content.

From response Table 4, one can see that dolomite dust remains the most critical parameter affecting SWR of the manufacture composites followed by applied load, sliding distance and sliding velocity. The optimum combination of parameters that yield the lowest SWR was obtained as 5 wt.% dolomite dust, 45 N applied load, 1 m/s sliding velocity and 4000 m sliding distance. In Table 4, delta is the range of four-level average SN ratios for each parameter and determined as the difference between the maximum and minimum SN ratio. The effectiveness of parameters was determined in terms of percentage contribution (%–C) using Eq. (3) [40] and presented in Fig. 10.

$$\% - C = \frac{Delta}{\sum_{i=1}^{k} (Delta_i)} \times 100 \tag{3}$$

where, i = parameter; k = total number of parameters.

It was observed that dolomite dust (A) have dominant influence on SWR with %–C of 36.02% closely followed by applied load 31.06%. Whereas, sliding distance (D) and sliding velocity (C) have least influence with %–C of 19.26% and 13.66% respectively.

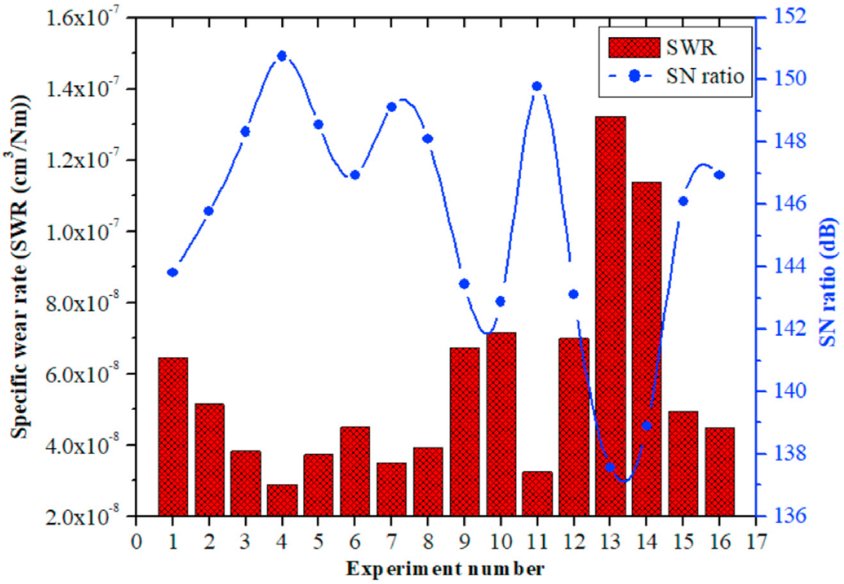

Fig. 8 – Wear results and SN ratio of the composites.

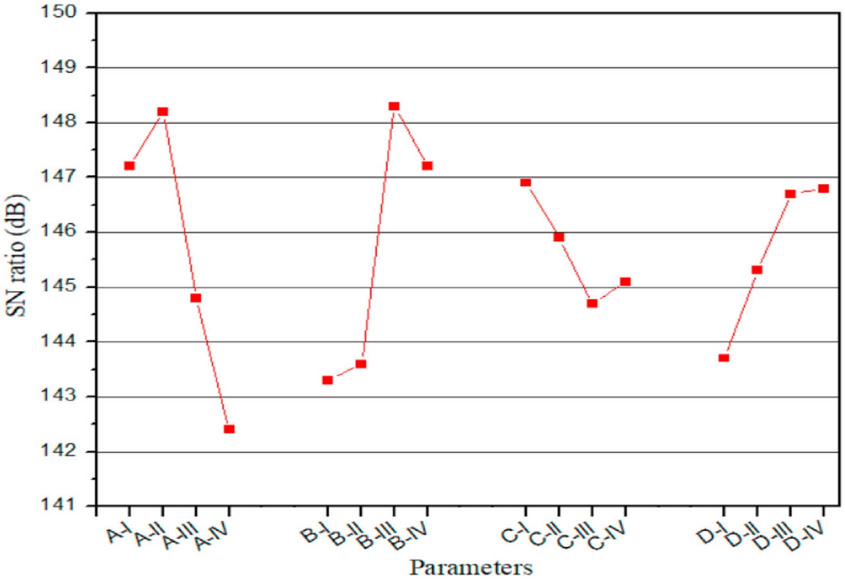

Fig. 9 – Main effect plots for SN ratio of SWR.

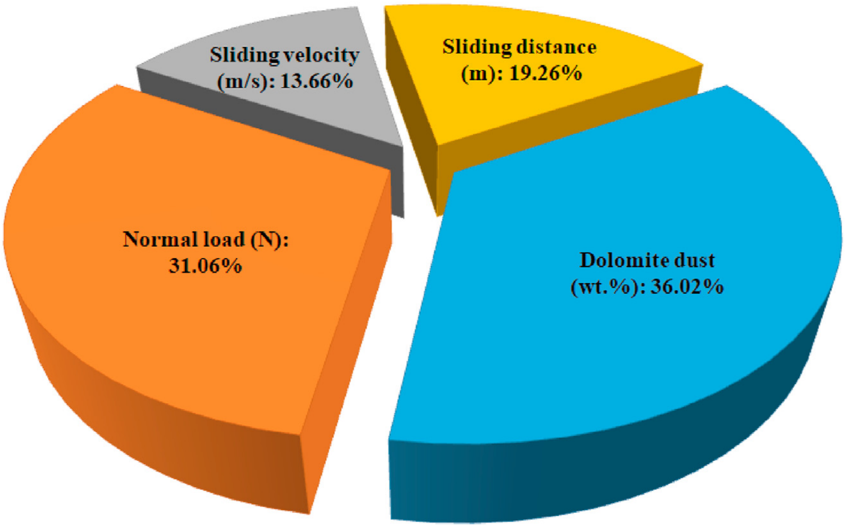

Fig. 10 – Contribution of each parameter for SN ratio of SWR.

3.3. *Worn surface morphology*

Further, to understand the wear mechanism, the composites' worn surfaces were analyzed using SEM (ZEISS EVO-18) and presented in Fig. 11. During sliding, it was observed that the surface undergoes stretching and twisting of both matrix and fiber, resulting in surface deformation, as shown in Fig. 11a-d. In addition, micro-ploughing, micro-cutting and grooves were also observed, as shown in Fig. 11a. The S1 composite (Fig. 11b) with 5 wt.% dolomite dust showed smooth surface morphology with micro-ploughing, micro-cutting and grooves formation indicating almost the same wear performance as composite S0 (Fig. 11a). Observation revealed a higher wear rate was caused by increased dolomite dust (≥10 wt.%) for S2/S3 composites, as evident from Table 4. Expanding the dolomite dust content greater than ≥10 wt.%, the percentage of vinyl ester reduces to 75%, making the composite structure highly rigid and brittle. Upon loading the specimen, the brittle nature of the composite started showing the extensive surface deformation and micro-ploughing shown in Fig. 11c-d. The micro-ploughing leads to the extensive micro-cutting of the composites, and dolomite dust particles start detaching from the composite surface as wear debris resulting in increased wear of the composites [33]. The detachment of the dolomite particles causes the carbon fiber to come into contact with the sliding interface. The exposed carbon fiber is then impacted and sheared by hard dolomite particles during sliding, losing the reinforcing effect and further accelerating the wear rate [41].

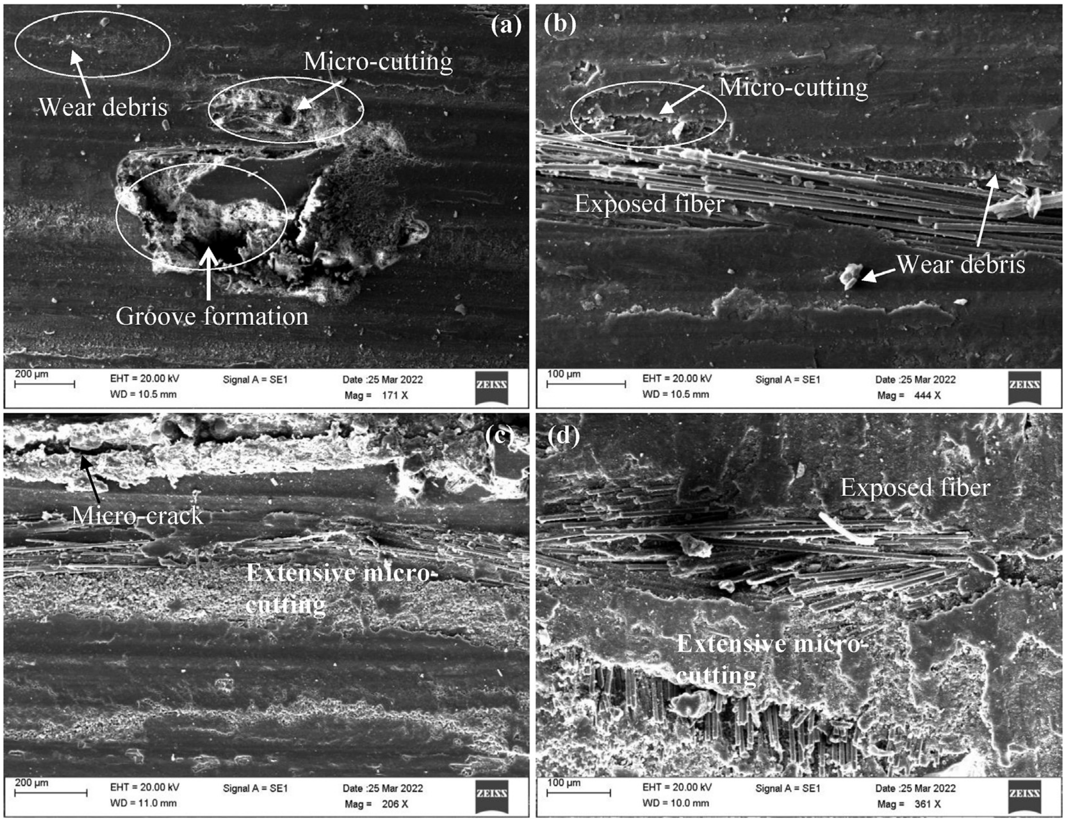

Fig. 11 – Worn surface morphology of (a) S0, (b) S1, (c) S2 and (d) S3 composites sample tested under conditions for trials 3, 8, 10 and 14 of Table 3.

4. Conclusion

Carbon fiber reinforced vinyl ester composites filled with varying amounts of dolomite dust were fabricated and evaluated for physicomechanical and sliding wear properties. Experimental results revealed that the void content, density, hardness, impact energy, tensile modulus and flexural modulus of carbon fiber reinforced vinyl ester composites increased with increased dolomite dust, whereas a constant decrease in tensile strength and flexural strength was observed. The design of the experiment revealed that the specific wear rate was decreased with 5 wt.% dolomite dust and started increasing with a further increase in dolomite dust loading (≥10 wt.%). The SN ratio study demonstrates that dolomite dust is the most significant and influential parameters in the sliding wear with a contribution of 36.02%. As the investigated properties either improved or decreased slightly, thus waste dolomite dust can be recommended as cheap filler for the development of innovative polymer composites.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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