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https://doi.org/10.5109/7160922

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出版情報: Evergreen. 10 (4), pp.2645-2653, 2023-12. 九州大学グリーンテクノロジー研究教育セン
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Experimental Investigation on the Mechanical and Abrasive Properties of Ceramic Particulate Filled Basalt Fiber Reinforced Epoxy Polymer Composites

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(Received March 20, 2023; Revised December 19, 2023; accepted December 19, 2023).

Abstract: The properties of Composite Materials can be consequentially influenced by addition of particulate fillers, which can lead to tailor made specific uses of these composites. Composites materials are widely used materials in automobiles, aviation, defense as well as sport goods industries, due to their properties being at par with traditional engineering materials and sometimes superior as well. In this study, basalt fiber laminates were fabricated by addition of a particulate filler namely Marble Dust, by conventional hand lay-up method. Further, these composite laminates were analyzed for their mechanical (tensile, flexural, impact strength, hardness and density) and three body abrasive wear performance. The analysis indicated that strong impact on properties of composites after the addition of particulate fillers. Moreover, this study also focuses on use of waste Marble dust and stone dust such as Kota Stone Dust for use as particulate filler in composites.

Keywords: Composites, Basalt fiber, Marble Dust, Three body abrasive wear, Flexural strength

1. Introduction

A composite material is an interaction of mediums, i.e. a fiber and a matrix; and its properties can be impacted by inclusion of one more medium in the interphase in form of a particulate filler. When combined, they derive a conglomeration of properties of all the elements which they don't possess when acting alone¹⁾.

A vibrant attribute of composite materials that makes them very pivotal and optimistic engineering material is their trait to tailor make their characteristic properties. This can be achieved via changing of fiber orientation, stacking and layering, hybridization, use of various matrices or by introduction of particulate filler materials in the interphase^{2, 3)}. With the possibility of various changes of properties as well as functionalities, composite materials create a very particular niche among available engineering materials. All these traits opens up a wide future scope and material science applications for composite materials as well as opens up a chance to replace orthodox materials currently under distinct/specific use⁴⁻⁷).

Basalt fiber is one of the most basic yet very functional fiber material used in composites. It provides a wide range of modifications to a composite simply by virtue of change in fiber orientation, weave matting type and weight implications. All these factors play a very vast role in characteristic properties of Basalt fiber composites. Furthermore, various recycled Basalt fiber types are making an entry into already available materials making path for more renewable and environment friendly options for future use⁸⁻¹¹.

The matrix material influences the properties of a material manufactured by composites. It not only secures the fiber reinforcement, transfers load between fibers, but also provides the composite with its penultimate shape and determines its surface grade and finish¹²⁻¹³⁾. In accordance with this, epoxy resin, which is a polymer matrix provides composite materials with a vast variety of properties; i.e. superior mechanical, thermal and electrical properties¹⁴⁻¹⁵⁾. Thus epoxy resin creates a very specific niche in high performance applications such as aviation parts, sports equipment's, body amours etc.¹⁶⁻¹⁷⁾.

Particulate filler particles are also introduced in the composite interphase to elevate various properties¹⁸⁾. Inclusion of filler particles improves toughness, stiffness, hardness; thermal properties for a composite material depending upon its individual attribute¹⁹⁾. Solid particulate fillers such as titanium oxide, aluminum oxide, and waste Marble dust such as Kota stone dust are being used these days to consequentially refine mechanical properties, wear resistance, temperature & water

resistance for various polymer matrix composites²⁰⁻²¹). Composites with particulate fillers have also shown notable improvement in real time operational conditions²²⁻²³).

Titanium oxide (TiO2) as a filler material impacts the mechanical properties of a composite²⁴⁾. While, wear resistance, strength, stiffness, temperature & chemical resistance for a composite material also increases when aluminum oxide (Al2O3) is used as filler²⁵⁾.

Waste marble and stone dust such as Kota stone powder, when used as particulate filler have also shown a notable impact on properties of composites. Moreover, composites have shown greater temperature and chemical resistance when Kota Stone Dust was used as filler²⁶. Moreover, the particulate fillers generated as a water byproduct of various marble and stone industries can be used to replace many conventional particulate types of filler currently under practical use; simply because of the fact that this process is more ergonomically. As well as this practice can have a positive environmental effect and can be a solution to the waste problems related with stone and marble industries²⁷⁾. Various industries have already demonstrated the noteworthiness of particulate fillers in composite materials vastly due to the strong impact it depicts on material properties. Furthermore, they can be effectively utilized to tailor make the composite properties as per the user and practical requirement²⁸⁻³¹). The implementation of composite materials in aerospace & aviation, automobile, sports and biomedical applications have been on the rise since past few years, and with continuous research and experimentation, this trend of using composites in place of traditional materials is only going to escalate in the fore-coming years³²⁻³⁴⁾. The objective of this analysis is to scrutinize the impact of particulate fillers i.e. TiO2, Al2O3 and especially Marble Dust on Basalt fiber polymer composites³⁵⁻³⁶⁾. For this purpose, hand lay-up technique was used to fabricate composites³⁷⁻³⁸⁾. The particulate fillers were added as 8%, 16% & 24% of fiber weight ratio. The samples were then analyzed for tensile, compressive and hardness inspection while their toughness and density were depicted using standard theoretical approach³⁹⁻⁴¹⁾. Despite all the advancements and research work in composite's material science, the impact of particulate filler generated as a byproduct from stone and marble industry is yet to be researched rigorously. Therefore, this study and research work is mainly focused on role of Marble Dust as particulate filler in composite materials.

2. Experimental Procedure

2.1 Materials

Basalt fiber composites of 4 variations were put together for this research work. Plain weave basalt fiber of 600GSM was procured from Carbon Black Composites. Epoxy laminating resin and hardener combination of 2:1 mixture ratio was also supplied by Carbon Black Composites and is used as the matrix material. Particulate filler were used for composite fabrication, i.e. Marble dust which was sieved to the particle size of 420 microns prior to use. Marble Stone dust was collected from Shree Ram Industries. The 4 composites prepared for this study are identified as EBM 0, EBM 8, EBM 16, and EBM 24. The exact compositions and designations are mentioned in Table 1.

2.2 Fabrication of composite

Basalt fiber composite laminates were prepared and were isolated for 48 hours for proper curing at room temperature. Fiber and epoxy matrix were kept in equal parts i.e. fiber loading was kept at 50% for all samples. No particulate filler was added in the EBM 0 composite. Thereafter, other composite samples contained particulate fillers in 8%, 16% & 24% epoxy weight ratios respectively, thus making up a total of 4 composite laminates. The laminates EBM 8, EBM 16 & EBM 24 contained marble dust as filler in 8%, 16% & 24% proportions respectively. Diamond cutter trimmer is used for identification and testing of samples and specimens.

3. Physical and Mechanical Testing

3.1 Hardness

The laminates were analyzed for surface toughness using Rockwell Hardness Scale. This test wedges a conical shaped perforator of steel or diamond against a test specimen and assesses the hardness as indexed by developed indentation depth. Higher HR value represents hard material. In this test, a smaller load of 10 Kgf is first executed, and the test dial is set to zero. Then to create a complete indention, a load of 60 Kgf is executed. The higher load is reverted to the smaller load, and the change in depth is measured. The perforator is generally 1.588 mm in caliber, and for composite materials, this test was processed on the HRF scale of the Rockwell Hardness Test.

3.2 Density and Voids Content

The manufactured composite comprises of Matrix material, including fibre and embedded with particulate filler material.

Therefore, the theoretical density (ρ_{ct}) is analysed via Eq. 1.1 (Agarwal and Broutman):

$$\rho_{ct} = \frac{1}{\frac{P_{fr}}{\rho_{fr}} + \frac{P_{mm}}{\rho_{mm}} + \frac{P_{pfm}}{\rho_{pfm}}}$$
(1.1)

Where,

P = weight fraction ρ = density of the ingredients pfm = particulate filler materials fr = fiber reinforcement mm = matrix material

Simple water immersion method is used to analyze the experimental density (ρex) of the sample and voids fraction (Δv) present is normalized by using Eq. 1.2 [19].

$$\Delta \mathbf{v} = \left[\frac{\rho_{ct} - \rho_{ex}}{\rho_{ct}}\right] \tag{1.2}$$

3.3 Tensile and Flexural Strength

The tensile strength (T.S.) of composite specimen is calculated by Universal Testing Machine (UTM). The tensile test is carried on specimen of specific sizes as per ASTM standard D3039. A uniaxial load is provided on both the edges of sample with cross head velocity of 2 mm/min during analysis. The tensile strength is calculated by using the achieved results.

Flexural test standard follows ASTM D7264. Flexural test sample's dimension is $80 \text{ mm} \times 10 \text{ mm} \times 4 \text{ mm}$. Eqs. 1.3 And 1.4 are used to determine Flexural strength and flexural modulus respectively.

$$FS = \frac{3WD}{2bt^2}$$
(1.3)

$$F M = \frac{D^3 m}{4bt^3}$$
(1.4)

Where,

W = Maximum applied load

D = Span length

m = Slope of tangent

b = sample width

t = sample thickness

3.4 Impact Strength

Pendulum type impact tester is utilised according to ASTM D256 specification to calculate Impact energy.

3.5 Three-Body Abrasive Wear Analysis

As per ASTM G65 specifications, sample is tested for three-body abrasive wear behaviour. Figure 1 shows the setup for this and Input variables are shown in Table 2. The rectangular moulds with size of $75 \times 25 \times 12 \text{ mm}^3$ is used. From the nozzle, particles are flown into the hopper at the feed speed of $255 \pm 5 \text{ g/min}^2$. The weights are noted prior and post of the experiment by use of electronic balance and then change is noted. Using Eq. 1.5 Abrasive behaviour is measured¹⁹).

$$Wa = \frac{\Delta V}{\rho \times W_n \times S_d} (mm^3/N-m)$$
(1.5)

Where,

Wa = Abrasive wear rate $(mm^3/N-m)$

 $\rho = \text{Experimental density of composite } (g/cc)$

Wn=Normalload (N)

S_d = Sliding distance (m)

3.6 Practical based on Taguchi's design

Conventional method needs a large number of experiments for various inputs at various levels. So by using Taguchi's design, number of recordings can be reduced for finding optimum value. Table 2 shows the i/p control factors at various stages. From this experiment, parameter can be identified which affects wear characteristics. Simulation using Minitab 17 software was done. There are 4 input variable and 4 stages, so L_{16} (4^A4 = 16) matrix is used and output are changed into signal-to-noise (S/N) value. For minimum wear characteristics, smallest S/N value is used.

$$\frac{S}{N} = -10 \log \frac{1}{n} \sum Y^2$$
 (1.6)

Where, n represent number of observations, and Y represent observed data.

After that, analysis of variance (ANOVA) is used to find optimal value. By using ANOVA and S/N ratio optimal level and its effect can be identified.



Fig. 1: Abrasion experiment setup

4. Results and Discussions

4.1 Density and void content characteristics

The concept behind evaluation of density and void content of composites is that how the composite performs when subjected to mechanical tests. Figure 2 and Table 3 shows the results of experimental density and void content of unfilled and marble dust filled basalt fiber-reinforced epoxy composites. Density of composites found to increase with increase in filler content in the range of 1.3129-1.4226 g/cm³. Marble dust filler have higher density than the epoxy resin; this may be the reason of increasing in density of composites with addition of marble dust filler.

Table 1: Identification & Detailed Composition of Composites				
Designation	Composition			
EBM 0	Epoxy + Basalt Fiber			
EBM 8	Epoxy + Basalt Fiber + 8 wt. % Marble dust			
EBM 16	Epoxy + Basalt Fiber + 16 wt. % Marble dust			
EBM 24	Epoxy + Basalt Fiber + 24 wt. % Marble dust			

Table 2: Abrasion experiment setup variables						
Control factors	Ι	II	III	IV	Units	
A: Filler loading	0	8	16	24	wt.%	
B: Normal load	20	40	60	80	Ν	
C: Sliding distance	500	1000	1500	2000	m	
D: Abrasive size	100	200	300	400	μm	

Table 3: Theoretical and actual density of the composites							
Characteristics	Composite						
	EBM 0 EBM 8 EBM 16 EBM 24						
Experimental density (g/cc)	1.3129	1.3475	1.3875	1.4226			
Theoretical density (g/cc)	1.3374	1.3865	1.4394	1.4961			
Voids content (%)	1.70	2.62	3.37	4.60			
Micro-hardness (Hv)	30	33	37	43			



Fig. 2: Theoretical, actual density & void content of composites

4.2 Hardness

Hardness of composites is the ability to resist indentation. Harder surface of composite indicates the better wear resistance of its. Generally, the fiber reinforcement improves the hardness of composites, but particulate filler improves the hardness of composites higher than fiber reinforcement. The variation in hardness with marble dust filler of sample are shown in Figure 3 & Table 3. Hardness of sample rises as marble dust percentage rises from 0 to 24 wt. %. Sample with 24 wt. % marble dust filler has hardness value of 43 which is maximum.



Fig. 3: Hardness of filled and unfilled sample

4.3 Tensile strength

The ratio of the maximum weight and cross section area of composite indicates ultimate tensile strength of material. Tensile strength of polymer matrix can be enhanced by fibre reinforcement. The inclusion of particulate filler enhances the tensile load bearing capacity of composite as well as tensile strength and modulus of composites. Graph shows the variation of tensile strength with marble dust content. Composite with 16 wt. % marble dust shows highest tensile strength (56.33 MPa) which is 39.31% higher than virgin (EBM 0) composite, respectively. Further, addition of marble dust content (24 wt. %) decrease the value of tensile strength.



Fig. 4: Variation in tensile strength of composites

4.4 Flexural strength

There are many important properties of fiber/filler reinforced polymer composites one of them is flexural strength. Composites are failing during bending due to high ratio value of lateral dimension and thickness. The flexural strength of marble dust-filled basalt-epoxy composites are depicted in Figure 5 and Table 4. Flexural strength of composite increased marble dust reinforcement percentage up to 16 wt. %; more increment in dust percent decreases the flexural strength of composites. Basalt-epoxy composite with 16 wt.% marble dust (EBM 16) shows maximum flexural strength (155.8 MPa) which is 13.31% higher than virgin composite (EBM 0), respectively.

4.5 Impact strength

Figure 6 and Table 4 represents the impact strength of prepared composite samples with rise in the percentage of marble dust filler. Impact strength rises with more addition of filler up to 16 wt. %, and then it starts decreasing till 24 wt. % marble dust.

4.6 Taguchi's design and ANOVA analysis

With the help of L16 Taguchi's orthogonal array abrasive behaviour is analysed and the S/N values of composite samples is shown in Table 5 and Figure 7.

The overall average value for S/N ratio for all experiments is 40.59 dB as shown in Figure 7.



Fig. 5: Variation in flexural strength of composites



Fig. 6: Variation in impact strength of composites

Table 6 represents the ranking order of input variable. From Table 6 it is concluded that the descending order of effect on abrasive behaviour of sample is sliding distance, normal load, filler loading, and abrasive size. The ANOVA analysis results are shown in Table 7. As per ANOVA analysis for various composition of Marble dust filled basalt-epoxy samples, ranking of the control variables in ascending order are abrasive size, filler loading, normal load and Sliding distance.

5. Conclusions

On the basis of work carried out in this study the following important outcomes were made

• When particulate filler content in composite increases, its hardness increases.

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- At 16 wt. % of marble dust filler the basalt epoxy composite shows the maximum tensile strength, flexural strength and impact strength.
- In the Taguchi experimental analysis, the ascending order of effect of input variable are: sliding distance, normal load, filler loading, and abrasive size.

Table 4: Characteristics of unmed and filled basan-epoxy sample						
Characteristics	Unit	Composite				
		EBM 0	EBM 8	EBM 16	EBM 24	
Tensile Strength	MPa	37.61	40.86	56.33	42.1	
Flexural Strength	MPa	137.2	142.8	155.8	148.8	
Impact Strength	J	0.53	0.71	1.01	0.54	

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Table 5: Taguchi's L16 orthogonal array based design

Exp. No.	A: Filler Loading	B: Normal Load	C: Sliding distance	D: Abrasive Size	Abrasive Wear Rate	S/N Ratio (dB)
1	0	20	500	100	0.0107580	39.3654
2	0	40	1000	200	0.0100679	39.9412
3	0	60	1500	300	0.0061733	44.1897
4	0	80	2000	400	0.0062460	44.0879
5	8	20	1000	300	0.0105009	39.5755
6	8	40	500	400	0.0350950	29.0951
7	8	60	2000	100	0.0099539	40.0401
8	8	80	1500	200	0.0062982	44.0156
9	16	20	1500	400	0.0029356	50.6461
10	16	40	2000	300	0.0076723	42.3015
11	16	60	500	200	0.0102409	39.7932
12	16	80	1000	100	0.0089076	41.0048
13	24	20	2000	200	0.0040720	47.8039
14	24	40	1500	100	0.0104427	39.6238
15	24	60	1000	400	0.0199987	33.9800
16	24	80	500	300	0.0199658	33.9942

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Table 6: Input variable ranking order of effect					
Level	A: Filler Loading	B: Normal Load	C: Sliding Distance	D: Abrasive Size	
1	41.90	44.35	35.56	40.01	
2	38.18	37.74	38.63	42.89	
3	43.44	39.50	44.62	40.02	
4	38.85	40.78	43.56	49.45	
Delta	5.25	6.61	9.06	3.44	
Rank	3	2	1	4	



Fig. 7: Effect of input variable on abrasive behaviour of marble dust Filled BFRP samples

Input control factors	DF	Adj. SS	Adj. MS	F-value	p-value	P (%)
A: Filler Loading	3	74.537	24.846	22.97	0.014	17.85
B: Normal Load	3	93.847	31.282	28.92	0.010	22.48
C: Sliding Distance	3	216.733	72.244	66.78	0.003	51.93
D: Abrasive Size	3	28.984	9.661	8.93	0.053	6.94
Error	3	3.245	1.082			
Total	15	417.346				

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