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ORIGINAL ARTICLE

Mechanical, water absorption and tribological behavior of eggshell powder-reinforced bakelite composites

Shakil Danish[1] · **Jitendra Nath Mahto**[1] · **Manoj Kumar Gupta**[2] · **Nitesh Singh Rajput**[3] · **Shweta Kulshreshtha**[4] · **Sandeep Kumar**[2]

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Abstract

Due to its high calcium carbonate concentration, eggshell powder (ESP) has recently attracted a lot of attention in the polymer sector and has the potential to replace traditional mineral fillers. In this paper, ESP-reinforced polymer composites were fabricated. The weight percentage of ESP was varied in Bakelite from 5 to 15%. The mechanical, absorption, and tribological behaviors of fabricated composites were investigated. The result reveals that the hardness of 15% ESP-reinforced composite showed a maximum hardness of 21.64 HV. The composite of 15% reinforced ESP exhibited high water absorption. We investigated the tribological behavior of fabricated composites at different eggshell powder weight percentages (0–15 wt%), speeds (800–1400 rpm), normal loads (5–20N), and sliding distances (1000–2500 m) following the sliding wear test experiments (pin-on disc) by using Taguchi experimental design (L_{16}). The contribution (%) of selected factors was analyzed by analysis of variance (ANOVA), and it showed that the most significant factor influencing the wear properties is eggshell powder reinforcement (46.61%), accompanied by sliding distance (32.68%), speed (18.35%), and, lastly, normal load (2.36%).

Keywords Taguchi's approach · Wear behavior · Eggshell · Bakelite · ANOVA

1 Introduction

The aerospace and automotive industries are under pressure to reduce the weight of vehicles to make them more fuel-efficient and economical. These industries need high-performance materials which have a high strength-to-weight ratio. Nowadays, aerospace and automotive industries use composite materials for making many components to reduce the weight of vehicles and increase the performance of the components or vehicles. In order to meet the requirements of industries, the material processing industries and researchers attract composite materials for the development of new composite materials.

In this context, metal matrix composites (MMCs) and polymer matrix materials (PMCs) are categories of lightweight, high-performance materials. In the category of MMCs, magnesium metal matrix composites (Mg-MMCs) have an advantage over aluminum metal matrix composites (Al-MMCs) in that they reduce 15–20% weight of components without compromising structural integrity [1–10]. The cost and fabrication process are important considerations when producing MMCs. The costs involved should be as low as possible. The development of new compound materials with minimal waste is one of the most significant research areas in the current context [11]. Nowadays, many researchers around the world are focusing on the green production process [12]. Green production is the rehabilitation of a production line that provides a way to keep off a production line that provides a clean environment or provides a way to keep the environment green by reducing waste [13]. The use of waste products in the production process is like green production [14]. Waste materials like peanut shells, bagasse, pine needles, coconut shells, human bones or animal bones, fly ash, waste hen eggshells, bamboo leaves, and sawdust

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✉ Manoj Kumar Gupta
gupta_291@rediffmail.com

[1] Department of Mechanical Engineering, B.I.T. Sindri, Dhanbad, Jharkhand, India

[2] Department of Mechanical Engineering, Hemvati Nandan Bahuguna Garhwal University, Srinagar, Garhwal, Uttarakhand, India

[3] Department of Mechanical Engineering, Amity University Rajasthan, Jaipur, India

[4] Amity Institute of Biotechnology, Amity University Rajasthan, Jaipur, India

are commonly used as reinforcement or filler materials for developing cost-effective polymer matrix composites [15]. Polymer matrix composites (PMCs) have many applications, from the automotive industry to construction materials [16]. The reinforcement or filler material is one of the important ingredients of polymer matrix composites, and it plays a vital role in composite properties [8, 17]. Fillers or reinforcements can improve the properties of polymer matrix materials and help lower the overall cost of composites [18, 19]. The use of organic fillers or reinforcement as eggshell powder has become a new trend among the research fraternity due to their advantageous properties such as renewable, low density, and cost-effective composite [20, 21].

Eggshells are a waste of food processing industries, restaurants, and consumption at home. India produces around 20,000 tons of egg waste every year. In spite of eggshells being a biodegradable material, their decomposition rate is very slow. Eggshell waste produces an odder smell and creates air and soil pollution around settlements until decomposition [22–24]. The application of waste eggshells in the fabrication of composite may resolve the problem of environmental pollution up to some extent. Various biodegradable materials such as LLDPE/MPP and HDPE composites have been shown to have good electrical and flame properties [25, 26]. Eggshells have a network of protein fibers associated with $CaCO_3$ (96%), $MgCO_3$ (1%), and calcium phosphate (1%) of the eggshell weight [27, 28]. It has an ordered structure consisting of polycrystalline calcium carbonate ceramics, which is only a polymorphic form of calcite [29, 30]. The carbonized eggshell powder (ESP) was successfully used as a reinforcement material for the fabrication of metal matrix and polymer matrix composites [31–34]. Aluminum matrix with ESP particles can lead to the production of inexpensive and lightweight aluminum composites with improved weight. The applications of Al/ESP composite materials in automotive components may be such as pistons, connecting rods, and other applications where lightweight materials are required [35–37]. Ononiwu [38] investigated the effect of carbonized egg shells (CES) and flying ash on the mechanical properties and corrosion characteristics of the fabricated composite. The result revealed that tensile strength and compressive strength were the highest at 2.5 wt% carbonized egg shells reinforced composite. Furthermore, a corrosion rate of 2.5 wt% fly ash-reinforced composite showed the lowest corrosion rate. Dwivedi et al. [39] investigated the effects of eggshell reinforcement in AA 2014 on the hardness of Al/eggshell composite. It was reported that the hardness and tensile strength of composite containing 12.5 wt% eggshell improved by 33.33% and 37.83%, respectively. Omah et al. [36] analyzed the dielectric properties of composites made of carbonized and uncarbonized cow bone. The result shows that the carbonized cow bone particle-reinforced composite has a lower dielectric strength and a higher dielectric constant compared to the uncarbonized cow bone particle-reinforced composite. Ji et al. [40] prepared epoxy compounds from eggshell powder (ESP) with a content of 1–10 wt%. They found that the impact strength of epoxy compounds of ESP content of 5 wt% of material was 16.7 kJ/m^2 compared to 9.7 kJ/m^2 of pure epoxy resin. Asuke et al. [41] fabricated a polymer matrix composite by reinforcing carbonized bone particle ranges from 5 to 20 wt% in the polypropylene matrix. The wear rates of the composite increase with the increasing applied load. However, it decreases with the increasing carbonized bone from 0 to 15% particles. Shuhadah et al. [42] studied the effect of chemical reactions on isophthalic acid and ESP content in 5–25% in the ESP/LDPE composite mechanical properties due to chemical conversion and fillings of ESP showing higher durability compared to the LDPE matrix. Isophthalic acid makes the compounds stronger. Toro et al. [43] studied Young's modulus comparison of ESP/PP compound and $CaCO_3$/PP compound. Young's modulus of ESP with a particle size of 8.4 µm was found to be higher than the compound of $CaCO_3$ with a particle size of 17.1.2.0 microns and 0.7 microns. This is because the ESP/PP compound has a better continuous phase than the $CaCO_3$/PP compound. Supri et al. [44] prepared the composite of ESP/LDPE from different ESP contents with the addition of PE-g-MAH. They found robust strength and thermal stability of the ESP/LDPE compounds containing PE-g-MAH, which were greater than that of the ESP/LDPE compounds. Boronat et al. [29] developed a polymer composite by reinforcing eggshells. It was reported that the degradation temperature and mechanical properties of the fabricated composite improved. Iyer et al. [45] reported that Young's modulus and yield strength of polypropylene/eggshell composite increased due to reinforcement of eggshell in matrix material.

The main idea behind this research was to develop a cost-effective polymer matrix composite. The novelty of this research is that most of the researchers used uncarbonized eggshells for the fabrication of polymer matrix composite. Very few researches have been carried out for the development of composite using carbonized eggshells. In this research, the carbonized eggshell powder was used for the development of a new polymer matrix composite. The rationale for the selection of Bakelite materials as a matrix material was that it is widely used for making various parts of an automobile, electrical switches, switchboards, and the outer body of electronic goods. The Bakelite polymer is cheaper in comparison to other polymers. Calcium (Ca) is the main element of eggshell, which plays a vital role in improving the mechanical and triological properties of eggshell powder-reinforced composites. The idea for selecting carbonized eggshell powder is that the weight percentage of calcium (Ca) in carbonized eggshell is higher in comparison to uncarbonized eggshell. Eggshells have $CaCO_3$

(96%), $MgCO_3$ (1%), and calcium phosphate (1%) of the eggshell weight. During the carbonization process of eggshells, the $CaCO_3$ thermally decomposes into calcium oxide (CaO) and releases carbon dioxide (CO_2) gas. Hence, the weight percentage of CaO in carbonized eggshell powder increased because thermal decomposition is approximately 40% of thermal loss.

In this research article, the waste eggshell was processed to convert into carbonized eggshell powder and used as reinforcement for the fabrication of polymer matrix composite by reinforcing it into Bakelite matrix material. The weight percentage of ESP was varied in Bakelite from 5 to 15%. The mechanical, wear, and water absorption properties of fabricated composites were examined and reported. The optimal factor setting is done by Taguchi analysis to evaluate the harm due to sliding wear by generating the design of the experiment (DOE) using the popular statistical software MINITAB 16. Moreover, the contribution (%) of each selected factor to the specific wear rate of the fabricated composites is also discussed by analysis of variance (ANOVA).

2 Materials and methods

2.1 Reinforcement material

The process carried out in this research project is shown in Fig. 1. In this study, carbonized eggshell powder was selected as reinforcement material for the fabrication of polymer matrix composites. The eggshells were collected locally from food stores and restaurants near Sinagar and Chauras cities of Uttarakhand, India. A cleaning process was performed on the eggshells to remove all organisms, including membranes, lipids, and proteins. The cleaned eggshells were dried in an open atmosphere in the presence of sunlight for 3 days. The carbonization of the eggshell was carried out by placing the sun-dried eggshell in an electric oven and dried at 80 °C for 4 h to remove moisture [29, 43]. In the next step, the oven-dried egg shells were ground. The grind eggshells were placed in an electric muffle furnace and heated at the temperature of 800 °C for 2 h. Furthermore, the eggshell powder was grind and sieved by using a sieve shaker of 75 microns in size. The average particle size of 75 microns of carbonized eggshell powder is used as reinforcement material for the fabrication of composites. The flow chart of the fabrication of carbonized eggshell powder (ESP) is shown in Fig. 2, and the scanning electron micrograph (SEM) image is depicted in Fig. 3.

2.2 Matrix material

Bakelite powder is used as a matrix material for the fabrication of composites. Bakelite is the trade name of a polymer material obtained by polymerizing phenol and formaldehyde. Bakelite is a polymer containing phenol and formaldehyde monomers. This phenol-formaldehyde is a thermosetting polymer. They are the oldest polymers synthesized by humans.

Bakelite is widely used in car parts, wire cases, and kitchen appliances to withstand heat.

2.3 Composites fabrication

The polymer matrix composites were formed by mixing varying weight percentages (wt%) of carbonized eggshell powder (ESP) reinforcement in Bakelite. The ESP was mixed in

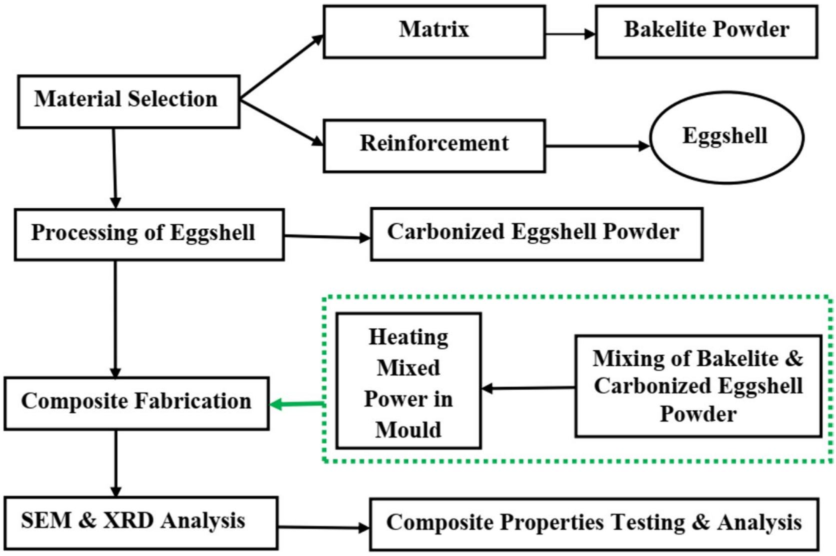

Fig. 1 Research work methodology

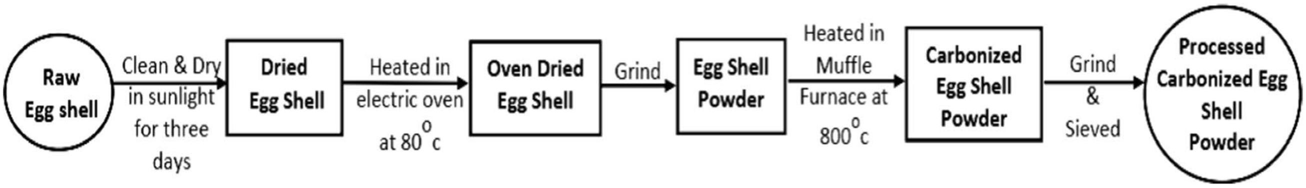

Fig. 2 Processing of carbonized eggshell powder

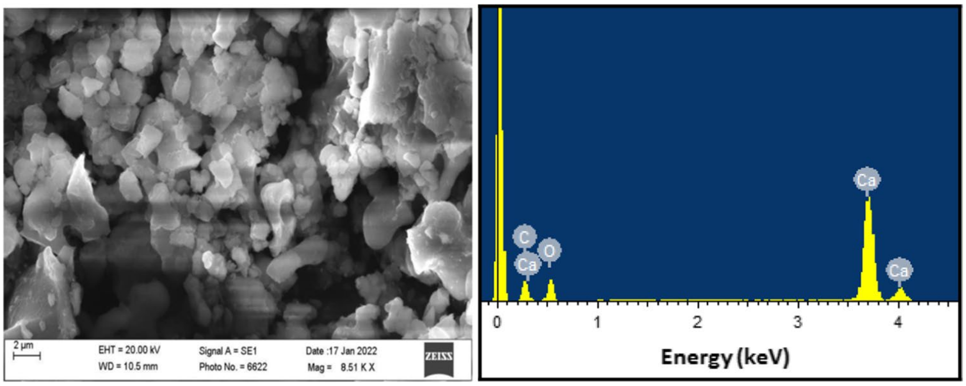

Fig. 3 SEM image of carbonized eggshell powder

Table 1 Composition of composites

S. no.	Composite composition
1	Bakelite + 0 wt% ESP
2	Bakelite + 5 wt% ESP
3	Bakelite + 10 wt% ESP
4	Bakelite + 15 wt% ESP

Bakelite by varying percentages of 5%, 10%, and 15%. The composites' composition is shown in Table 1.

The composites were fabricated by using a sample mounting machine. The mixture of Bakelite and ESP powder was heated at 120 °C for 30 min in a sample mounting machine mold for the fabrication composites. The process of fabrication of the composite is shown in Fig. 4. The fabricated composites are shown in Fig. 5.

3 Result and discussion

3.1 Density analysis

The density of carbonized eggshell powder (ESP) was measured by using the free flow method. The density of ESP was found at 1.3448 gm/cm^3. The density of Bakelite powder is 1.344 g/cm^3.

The density (ρ) of the composite was measured by using the following formula:

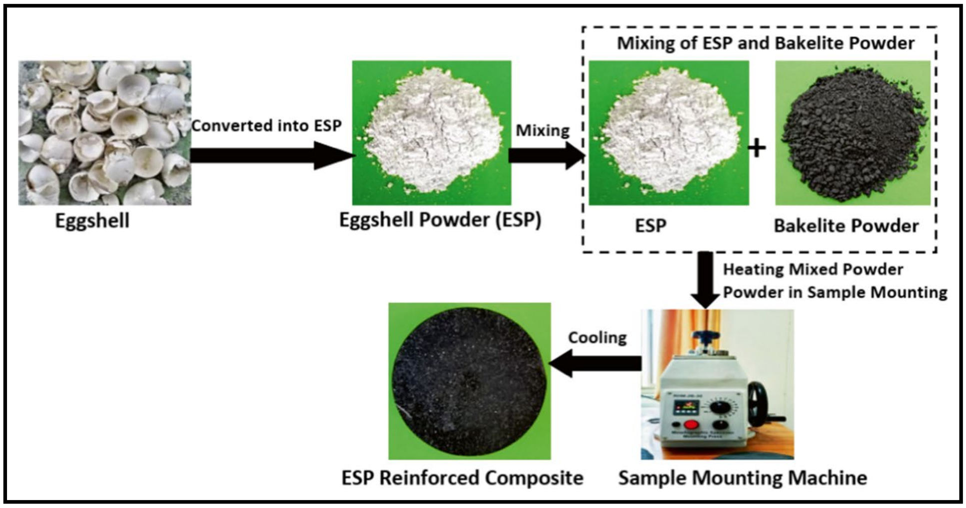

Fig. 4 Composite fabrication process

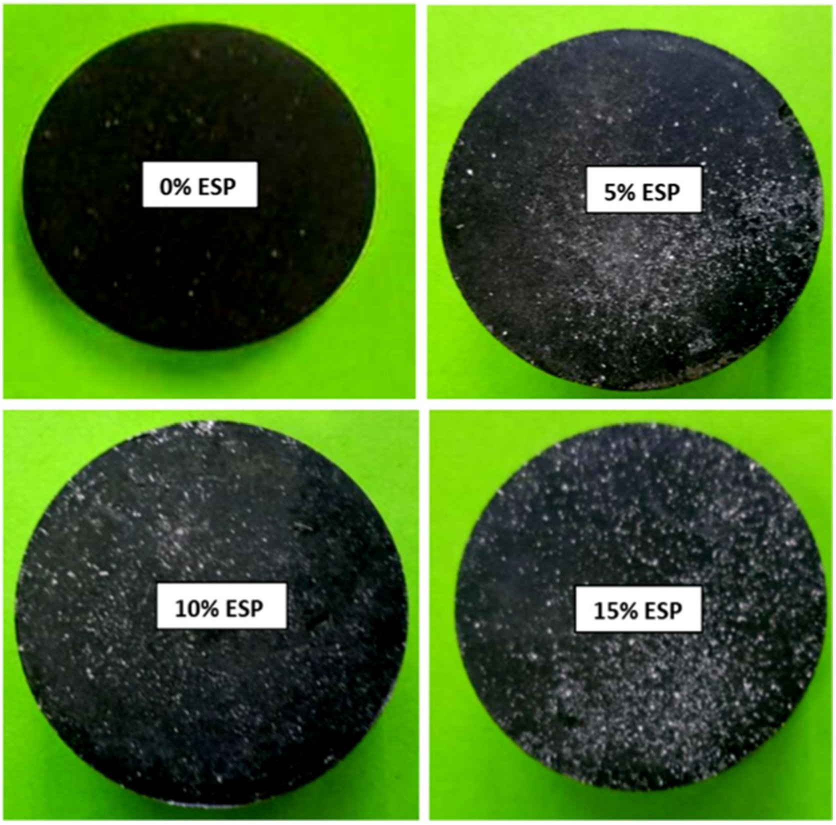

Fig. 5 ESP-reinforced Bakelite composites

Table 2 Densities of composites

S. no.	Composite	Density (ρ), g/cm^3
1	Bakelite + 0 wt% ESP	1.404
2	Bakelite + 5 wt% ESP	1.474
3	Bakelite + 10 wt% ESP	1.493
4	Bakelite + 15 wt% ESP	1.568

Table 3 Water absorption

S. no.	Composites	Water absorption (%Wa)
1	Bakelite + 0 wt% ESP	9.2%
2	Bakelite + 5 wt% ESP	14.8%
3	Bakelite + 10 wt% ESP	18.3%
4	Bakelite + 15 wt% ESP	31.6%

$$\rho = m/v\left(gm/cm_3\right) \tag{1}$$

where m is the mass of the composite and v is the volume of the fabricated composite. The densities of composites calculated are listed in Table 2. Table 2 shows that the density of composite increases with the percentage increase of ESP. The higher density of ESP than the Bakelite powder increases the density of the composite [44].

3.2 Water absorption

The water absorption by composites was calculated by putting the composites in water for 48 h. The percentage water absorption of composites was calculated by using the following formula:

$$\text{Percentage of water absorption (\%Wa)} = \left(\mathrm{w}_1 - \mathrm{w}_2\right)/\mathrm{w}_2 * 100 \tag{2}$$

where w_1 is the weight of the wet composite, and w_2 is the weight of the dry composite.

The calculated percentage of water absorption obtained is listed in Table 3. Table 3 reveals that the percentage increase of ESP increases the water absorption of composites. The cause of the increase in water absorption of composites may be due to the hydrophilic nature of ESP.

3.3 Mechanical properties

The hardness of the fabricated composite depends on the matrix and reinforcement material and their hardness and weight percentage in the composite. Hardness tests were performed on composites using Vickers hardness testing machine. The test was performed at three different locations, and the average value was taken as the hardness of the composite, as reported in Table 4. Table 4 shows that the presence of ESP in composites improved the hardness of composites.

The relationship between the weight percentage of ESP reinforcement in composite and their effects on hardness is shown in Fig. 6. The result shows that the hardness improved with the increase in the weight percentage of ESP in composite [39, 40]. Furthermore, the figure reveals that Bakelite with 0% ESP reinforcement has a hardness of 13.47 Hv. The hardness of 15% ESP-reinforced composite shows the highest hardness, which is 21.64 Hv and 60.65% higher than the matrix material (Bakelite).

Compressive strength is the property of the material to sustain the maximum amount of load without breaking or bending [23, 27]. The compressive strength of composites was examined on a universal testing machine. Three samples of each category of composites were examined, and the average value is reported in Table 5 and Fig. 7. Figure 7 reveals that the compressive strength of composite increases with the increase of weight percentage of ESP in composite up to 10 wt%. Further increase of wt% ESP in the composite decreased the compressive strength. In the composite, matrix material supports the reinforcement materials. The higher weight percentage of reinforcement in the matrix decreases the weight percentage of matrix material in the composite, which affects the bonding strength between matrix and reinforcement materials, affecting composite properties. The increase of wt% ESP in the composite decreased the compressive strength, which might be due to higher wt% ESP in the composite, weakening the bonding properties between matrix and reinforcement in fabricated composites. The maximum compressive strength is exhibited by 10% ESP-reinforced composite, which is 81.26 MPa, and it is 32.80% higher than that without ESP-reinforced composite. The tested samples are shown in Fig. 8. Figure 8 reveals that the amount of fracture of 10% ESP-reinforced composite is less in comparison to other composites.

Table 4 Hardness of composites

S. no.	Composites	Hardness (Hv)
1	Bakelite + 0 wt% ESP	13.47
2	Bakelite + 5 wt% ESP	16.68
3	Bakelite + 10 wt% ESP	18.56
4	Bakelite + 15 wt% ESP	21.64

Table 5 Compressive strength of composites

S. no.	Composites	Compressive strength (MPa)
1	Bakelite + 0 wt% ESP	61.19
2	Bakelite + 5 wt% ESP	70.98
3	Bakelite + 10 wt% ESP	81.26
4	Bakelite + 15 wt% ESP	75.19

3.4 Tribological behavior

The pin-on disc apparatus and ASTM G99 [31, 33] are used for evaluating the specific wear rate of composites. The electronic balance machine with an accuracy of 0.1 mg is used to carry out the mass loss of the composite surface, and Eq. (iii) is used to determine the specific wear rate of composites (SWR).

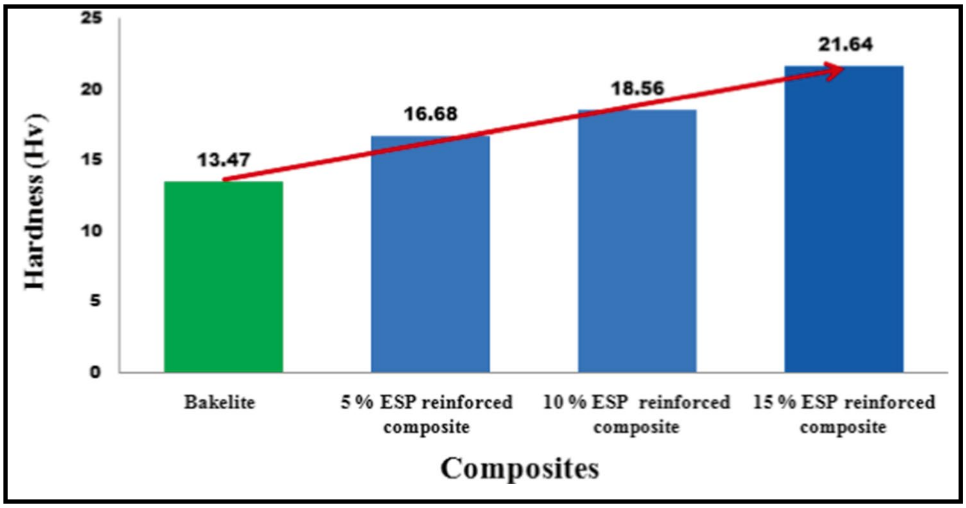

Fig. 6 Effect of ESP on hardness

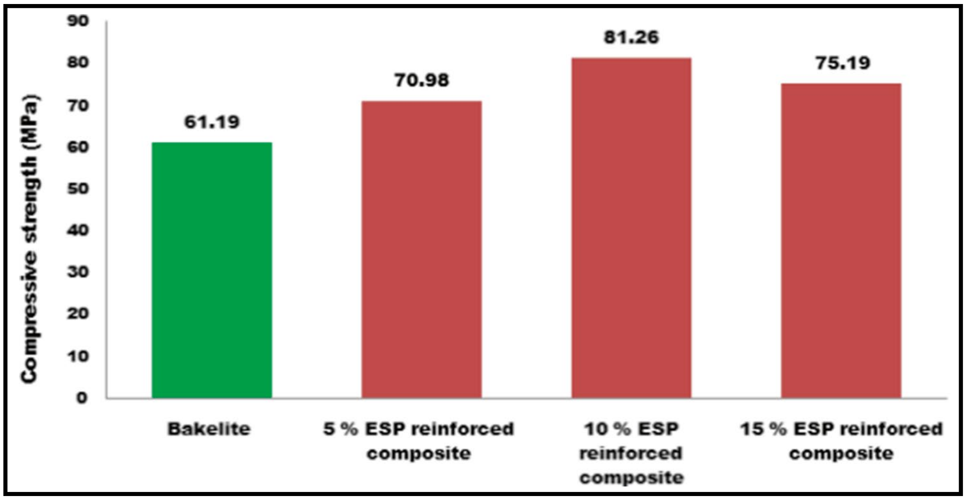

Fig. 7 Effect of ESP on compressive strength

Fig. 8 Compressive strength in tested samples

$$SWR = \frac{m_i - m_f}{\rho l f_n} \tag{3}$$

where m_i and m_f are the initial and final mass (g), ρ is density, l is the sliding distance (m), and fn is normal load (N).

The response of the input control factor on output performance is analyzed by Taguchi's method. The authors already investigated the influence of control factors on the sliding wear rate of composites and revealed that reinforcement content, speed, sliding distance, normal load, etc. greatly affect the sliding wear rate of the fiber-reinforced composites [31–33, 37, 38, 41]. In the present research work, four control factors (reinforcement, speed, normal load, and sliding distance) are used, and each control factor has four levels, as shown in Table 6.

The operating conditions by Taguchi's approach (L_{16}) under which sliding wear tests are done are illustrated in Table 7. Furthermore, the evaluated specific wear rate is

Table 6 Control factors and levels used in the experiment

Control factor	Level				Unit
	I	II	III	IV	
Reinforcement (R)	0	5	10	15	wt%
Speed	800	1000	1200	1400	rpm
Normal load	5	10	15	20	N
Sliding distance	1000	1500	2000	2500	m

Table 7 Experimental design using L16 orthogonal array

S. no.	% R	Speed (rpm)	Load(N)	Sliding distance(m)
1	0	800	5	1000
2	0	1000	10	1500
3	0	1200	15	2000
4	0	1400	20	2500
5	5	800	10	2000
6	5	1000	5	2500
7	5	1200	20	1000
8	5	1400	15	1500
9	10	800	15	2500
10	10	1000	20	2000
11	10	1200	5	1500
12	10	1400	10	1000
13	15	800	20	1500
14	15	1000	15	1000
15	15	1200	10	2500
16	15	1400	5	2000

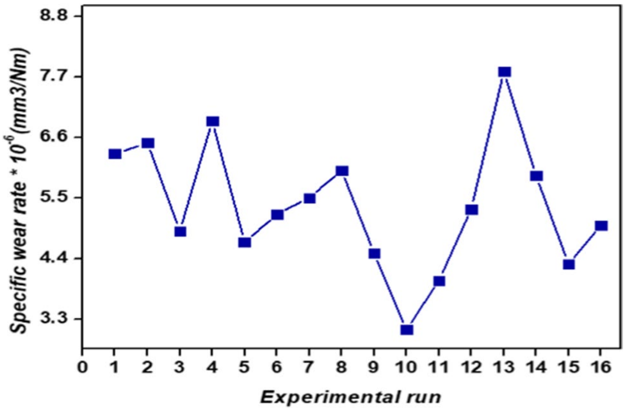

Fig. 9 Variation of specific wear rate of composites with experimental runs

transformed into a signal-to-noise (S/N) ratio by using smaller and better characteristics (Eq. (iv)). The contribution (%) of each selected control factor on sliding wear rate is determined by analysis of variance (ANOVA).

$$S/N = -10 \times \log(1/k)\left[\sum x^2\right] \tag{4}$$

where S/N is the signal-to-noise ratio, k is the number of observations, and x is the observed data.

The sliding wear response is analyzed by Taguchi's approach depicted in Table 8 and Fig. 9. The order of influence of the selected control factor according to their significance on specific wear rate is determined by subtracting the higher and lower value of S/N ratio (higher delta value gives

Table 8 Experimental design using L16 orthogonal array

S. no.	% R	Speed (rpm)	Load (N)	Sliding distance (m)	SWR × 10^{-6} (mm^3/Nm)	S/N ratio (db)
1	0	800	5	1000	6.3	104.013
2	0	1000	10	1500	6.5	103.742
3	0	1200	15	2000	4.9	106.196
4	0	1400	20	2500	6.9	103.223
5	5	800	10	2000	4.7	106.558
6	5	1000	5	2500	5.2	105.680
7	5	1200	20	1000	5.5	105.193
8	5	1400	15	1500	6.0	104.437
9	10	800	15	2500	4.5	106.936
10	10	1000	20	2000	3.1	110.173
11	10	1200	5	1500	4.0	107.959
12	10	1400	10	1000	5.3	105.514
13	15	800	20	1500	7.8	102.158
14	15	1000	15	1000	5.9	104.583
15	15	1200	10	2500	4.3	107.331
16	15	1400	5	2000	5.0	106.021

Table 9 S/N ratio response table for specific wear rate of composites

Control factor					
	Level	R (wt%)	Speed (rpm)	Normal load (N)	S sliding distance (m)
Average S/N ratio (db)	I	104.3	104.9	105.9	104.8
	II	105.5	106.0	105.8	104.6
	III	107.6	106.7	105.5	107.2
	IV	105.0	104.8	105.2	105.8
	Delta	3.4	1.9	0.7	2.7
	Rank	1	3	4	2

higher effect on specific wear rate), as tabulated in Table 9. The graph of the mean of S/N ratio versus the selected control factor is presented in Fig. 10. The factor combination of A3B3C1D3 (10wt% of R, 1200 rpm of speed, 5N of load, and 2000 m of sliding distance) carried optimum specific wear rates for fabricated composites. It is concluded that the specific wear rate decreased with the increase of the reinforcement wt% from 0 to 10 wt%, which further increased from 10 to 15 wt%. On the other hand, the inferior wear properties are obtained for normal load 5N to 20N, speed 1200 to 1400 rpm, and sliding distance 2000 to 2500 m. This research established that the sliding wear properties of the fabricated composites can be enhanced by increasing the reinforcement up to 10 wt% (ESP).

Furthermore, the experimentally evaluated sliding wear results are accomplished by ANOVA, and the contribution of each selected control factor (reinforcement, speed, normal load, and sliding distance) on a specific wear rate is

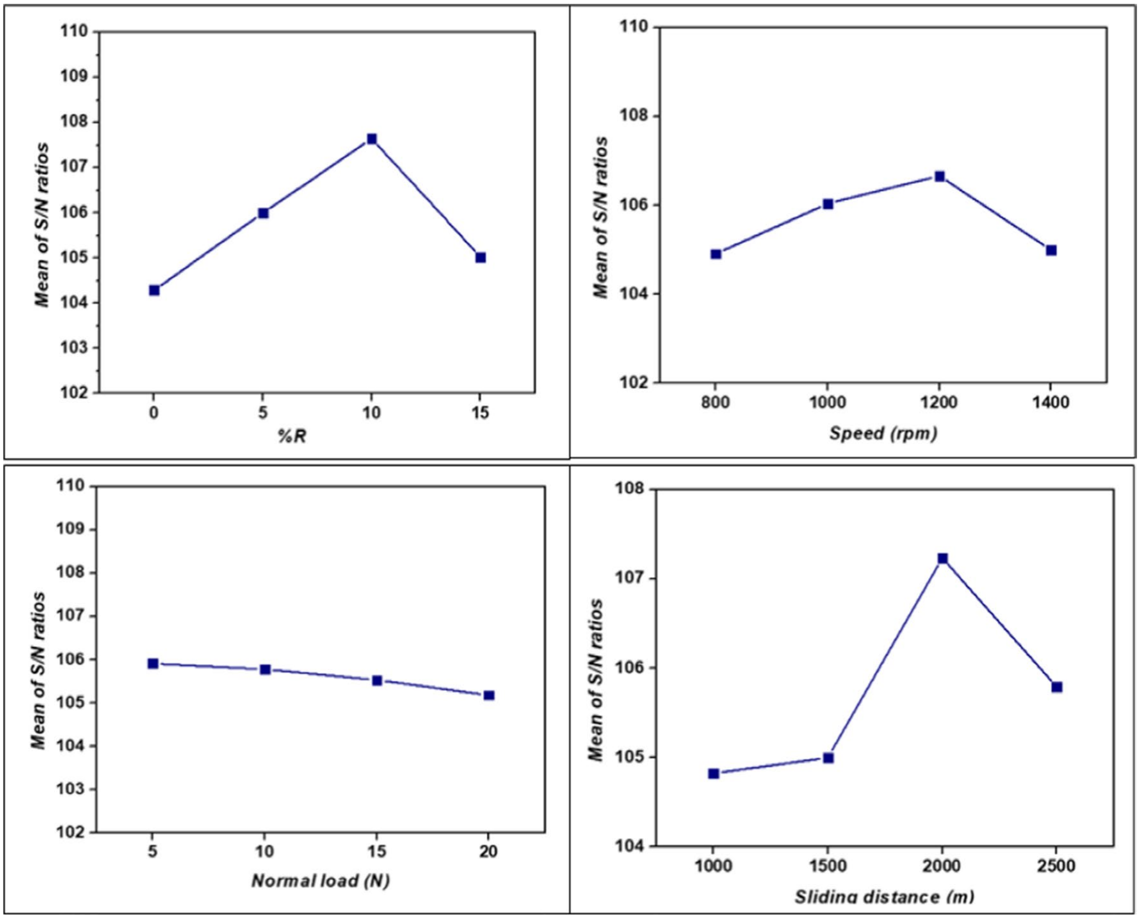

Fig. 10 Effect of control factors on the specific wear rate of composites

analyzed. The contribution of the selected control factor is depicted in Fig. 11, where the reinforcement shows the highest contribution of 46.61%, followed by a sliding distance of 32.68%, moderate contribution of speed of 18.35%, and least contribution of the normal load of 2.36% for composites.

4 Conclusion

The research's most significant findings are as follows.

1. Based on the results, the weight percentage of ESP in composite greatly affects the water absorption of composites. The composite, which has 5% ESP, exhibited low water absorption, whereas the composite 15% ESP-reinforced composite exhibited high water absorption.
2. The experiment showed that the density of the composite increased with the increase in weight percentage of ESP reinforcement in composites.
3. The composites of 10% reinforced ESP showed the highest compressive strength, which is 81.26 Mpa, whereas 5% and 15% ESP-reinforced composites exhibited compressive strengths of 70.98 MPa and 75.19 MPa, respectively.
4. The composite of 15% reinforced ESP exhibited maximum hardness, which is 21.64 Hv, in comparison to other composites.
5. The sliding wear properties of fabricated composites are successfully analyzed by Taguchi's experimental L_{16} orthogonal array, and it is revealed that reinforcement (R wt%), sliding distance, speed, and normal load established the critical control factor in decreasing order affecting sliding wear rate. The combination of reinforcement (10wt%), speed (1200 rpm), normal load (5N), and sliding distance (2000 m) gives the optimal condition for minimization of the sliding wear rate. The contribution ratio for each selected control factor on the specific wear rate of composites is 46.61% for reinforcement, 32.68% for sliding distance, 18.35% for speed, and 2.36% for normal load.

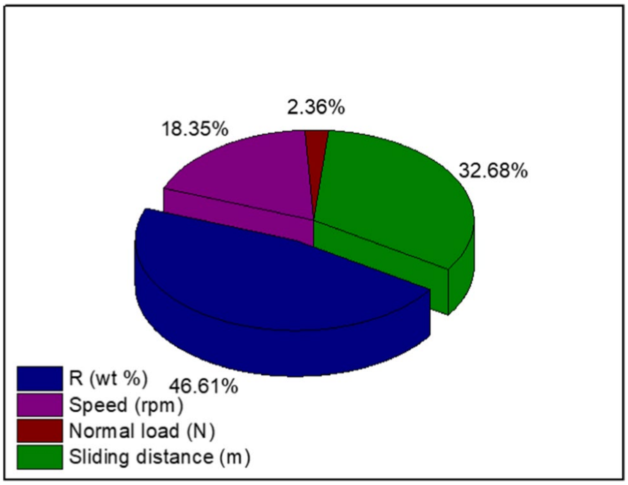

Fig. 11 Contribution of control factor on the specific wear rate of fabricated composites

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Author contribution All authors equally contributed work to this paper.

Data availability All necessary data were reported in the manuscript.

Declarations

Ethical approval All authors agreed with the manuscript's ethical considerations and involvement.

Consent for publication The consent for publication of this work was approved by all authors.

Competing interests The authors declare no competing interests.

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