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Physicomechanical and Taguchi optimized abrasive wear behaviour of KOH/KMnO₄/NaHCO₃ treated Himalayan Agave fiber reinforced polyester composite

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Abstract

This manuscript investigates the effect of various chemical treatments (potassium hydroxide, potassium permanganate and eco-friendly sodium bicarbonate) of Himalayan agave fibers (HAF) of size 7 mm on its physical, mechanical (tensile, flexural, impact, hardness and water absorption) and abrasive wear performance of HAF/polyester composites. The fibers were chemically treated at different concentration of 5, 10 and 15% of these chemicals for 24 h and reinforced at 10 wt% of fiber loading into polyester resin. Sodium bicarbonate fiber treated composite showed maximum tensile strength (145 MPa), flexural strength (214.5 MPa), impact strength (3.65 J cm⁻²), and hardness (30.33 Hₗ) with least water absorption as compared to other chemically treated HAF reinforced polyester composites. The optimum concentration of KOH and KMnO₄ treatment were arrived at 10 and 15% concentration respectively with both exhibiting comparable results. The dry abrasive specific wear rate of composites was studied at three different factors i.e. chemical concentration (5–15 wt%), normal load (10–30 N), and speed (50–150 RPM) using Taguchi technique L₀ orthogonal array. The sodium bicarbonate composite treated HAF based composites exhibited least specific abrasive wear rate. The scanning electron microscope (SEM) analysis of worn surfaces revealed the fiber breakage, debonding, debris, crack surface and micro-ploughing as the prominent wear mechanism.

1. Introduction

Now days, with increasing environmental concerns, the interest towards the utilization of natural fiber as reinforcement in polymer composites keeps on increasing for many engineering applications. There are significant efforts made in recent years to manufacture plant fiber reinforced polymer composites [1–3]. The use of natural fibers is exceptionally enhanced in the field of automotive industries such as in fabricating interior and exterior parts owing to eco-friendly and high specific strength [4–7]. The major reasons trailing for the use of natural fibers (NF’s) as a reinforcement are low cost, medium or high strength, great adaptability, high stiffness, recyclability, high degree of flexibility, less energy expenditure, low health risk and low abrasiveness as compared to synthetic fibers [8]. Natural fibers suffer some limitations such as being hydrophilic in nature which tends to increase the water uptake while the matrices are hydrophobic (water resistant) that decreases the adhesion between the fiber and the matrix resulting in reduced mechanical properties. The pre-treatments of natural fibers are aimed in improving the adhesion between the fiber and the matrix. In pre-treatments, either hydroxyl groups get activated or new parts are added that can effectively interlock the fiber with matrix [9]. An alkali treated composite showed an increase in the mechanical properties, because of better adhesion between fiber and matrix [10]. Mechanical properties of alkaline treated sisal fibrils reinforced epoxy composite was studied by
Nimanpure et al.[11], where it was revealed that the reduction in hemicellulose and lignin content with alkali treatment resulted in good interfacial bonding between fiber and matrix. Batara et al.[12] studied the influence of chemical treatment of alkaline and permanganate treatment on mechanical properties of abaca fiber. The result indicated that the tensile strength of 0.125% potassium permanganate for 3 min treated abaca fiber expressed maximum tensile strength which was again correlated to the fewer hemicellulose and lignin content as compared to the untreated fiber. Srinivasa et al.[13] reported that the areca fibers after alkali treatment with potassium hydroxide delivered better interfacial bonding between fiber and matrix.

Chemical Properties of natural fibers extracted from Typha Australis leaves were investigated by Moghaddam et al.[14]. The result showed that the alkali treatment removed most of the non-cellulosic components like pectin and hemicelluloses, and the chemical treatment using potassium hydroxide (KOH) exhibited higher tensile strength than those of sodium hydroxide (NaOH) treated ones. The potassium permanganate (KMnO₄) treatment of many fibers imparted improved mechanical properties [15–18]. Zegaoui et al.[19] demonstrated an enhancement in mechanical properties with sodium bicarbonate (NaHCO₃) treatment of hemp fiber due to removal of non-cellulosic components such as hemicellulose, the pectin and wax, and other impurities attached at the outer surface of the plant fibers with NaHCO₃ treatment. Natural fibers are chosen as reinforcements because they can reduce the tool wear at manufacturing process, respiratory irritation and serve as substitute for artificial/synthetic fiber composites in the increasing global energy disaster and environmental risks [20]. However, the main disadvantages of natural fibers in composites are the poor compatibility between fiber and matrix and the relative high moisture sorption. Therefore, the chemical treatments are recognized as basic methods in modifying the fiber surface properties.

In present study, the comparative evaluation of mechanical and abrasive wear properties of different chemical (KOH, KMnO₄ and NaHCO₃) treated Himalayan Agave fiber (HAF) based polyester composite materials were conducted in order to arrive best chemical treatment method of desired concentration. Himalayan agave fiber reinforced polyester composites were manufactured by hand lay-up process followed by compression molding. In the present work, the reinforcing HAF was treated by KOH, KMnO₄ and NaHCO₃ at different concentration (5, 10, and 15 wt%) for 24 h and the treated HAF was loaded at 10 wt% into polyester resin. The influence of different chemical treatments of HAF on physical and mechanical (tensile, flexural, impact and hardness) properties of HAF/polyester composites were evaluated. The secondary objective was to apply the design of expert and arrive the optimal parameter setting by Taguchi analysis to assess the damage to the different chemically treated HAF reinforced polyester composites due to three body dry abrasive wear by using the software MINITAB 16. Moreover, the significance of each selected parameter on the specific wear rate of the composite was also discussed in this work by analysis of variance (ANOVA).

2. Materials and fabrication

2.1. Materials and composites preparation

Himalayan Agave Fiber (Agave cantula Robx.) of density 1.2 g cm⁻³ was extracted from the plant by hand decortication process. These extracted fibers were chopped of length 7 mm and pretreated with respective chemical before reinforcing into unsaturated polyester GP resin. The compositional testing of HAF was conducted at SIGMA Test and Research Centre New Delhi, India which revealed its chemical compositions as cellulose (30 wt%), hemicellulose (33.59 wt%), lignin (28.27 wt%), pectin (0.204 wt%), moisture content (7.20 wt%), inorganic salt (0.42 wt%) and waxes (0.32 wt%).

The unsaturated polyester resin with density 1.35 g cm⁻³, methyl ethyl ketone peroxide (MEKP) with density 1.17 g cm⁻³ as hardener and cobalt naphthanate as accelerator were used. Further, three different chemicals i.e. KOH, KMnO₄ and NaHCO₃ of varying concentrations (5%, 10%, and 15 wt%) were used for chemical modification of HAF. The fibers were kept in the respective solution for 24 h at room temperature and was then thoroughly washed in distilled water to remove the non-cellulosic component from the fiber to reduce the hydrophilicity. Washing was continued until the fibers were free from chemical reagent and other impurities, before introducing into polymer matrix. The chemical reaction between fiber and the chemical reagent of KOH, KMnO₄ and NaHCO₃ is depicted in equation (1), equations (2)–(5) respectively as reported by researchers [21–23].

Potassium hydroxide treatment (KOH)

\[
\text{Fiber} \rightarrow \text{OH} + \text{KOH} \rightarrow \text{AF} \rightarrow \text{Fiber} \rightarrow \text{O} + \text{K}^+ + \text{H}_2\text{O}
\] (1)
Potassium permanganate treatment (KMnO₄)

\[
\text{Fiber – OH} + \text{KMnO}_4 \rightarrow \text{Fiber – H}_2\text{O – Mn} – \text{OK}^+ \\
\text{Fiber – H – O – Mn – OK}^+ \rightarrow \text{Fiber + H – O – Mn – OK}
\]

(a) Sodium bicarbonate treatment (NaHCO₃)

\[
\text{Fiber} + \text{NaHCO}_3 \rightarrow \text{Fiber} + \text{Na}^+ + \text{HCO}_3^- \\
\text{HCO}_3^- + \text{H}_2\text{O} \rightarrow \text{H}_2\text{CO}_3 + \text{OH}^- (5)
\]

2.2. Preparation of composites

In order to fabricate the chopped-HAF/polyester composites, hand lay-up technique and compression molding were applied. A wooden mould with the dimension of 250 mm × 250 mm × 10 mm was used. The randomly distributed chemically modified fibers were reinforced in polyester resin with fixed amount of 10 wt% to prepare the composites. Primarily, the silicon spray as a mould releasing agent was applied on the inner surface of the mould to facilitate easy removal of composite from the mould after curing. The curing of the polyester resin was done by the incorporation of 3/4 wt% of hardener (MEKP) and 3/2 wt% of cobalt naphthanate as accelerator, respectively. The proper mixing was done by mechanical stirrer before the mixture was poured into the mould. The as-casted mixture was pressed by compression molding machine at a room temperature with an applied load of 75 kN for 30 min. Finally, the composite was post cured in the oven at 70 °C for 7 h. The pictorial view of as-extracted Himalayan agave fiber, chopped fiber of length 7 mm, KOH treated HAF reinforced composite, KMnO₄ treated HAF reinforced and NaHCO₃ treated HAF reinforced composite are shown in Figure1 (a), (b), (c), (d) and (e) respectively. The designation of different chemicals treated composite is mentioned in Table 1.

2.3. Void fraction and water absorption testing

Voids fraction \(V_e\) of the composites was determined by using equation (6), where \(\rho_1\) and \(\rho_2\) represent the theoretical and experimental density of the composite. Water absorption test was done according to the ASTM D570 standard. The size of the specimen is cut in circular section of 50 mm diameter as shown in figure 2.

\[
V_e = \frac{\rho_1 - \rho_2}{\rho_1}
\]

The sample was removed at specific time interval from water and immediately weighed after dried. The difference between the mass after a given time of immersion and the initial mass was used for the determination of the water absorption. The percentage (%) of water absorption is calculated by equation (7) [24].

\[
\text{Water absorption} (%) = \frac{(W_f - W_i)}{W_i} \times 100
\]

Where \(W_f\) is the weight of sample at time (t), and \(W_i\) is the initially weight of sample (at t = 0).

2.4. Mechanical testing

Tensile (ASTM D 638) and flexural (ASTM D 790) test of composite were performed on universal testing machine (Make: INSTRON-3382) with the dimensions of 165 mm × 13 mm × 7 mm (figure 3(a)) and 125 mm × 12.7 mm × 5 mm (figure 3(b)) respectively. The Izod impact testing (ASTM D256) with dimensions of 63.5 mm × 12.7 mm × 10 mm was used to measure the impact strength of composites. Hardness test is conducted on computerized Vickers hardness testing machine (VM-50 PC). A pyramid diamond shape intender is allowed to intend on the surface of the specimen with a minimum load of 5 kgf for 10 s.
2.5. Three body sand abrasion test

Three body sand abrasion wear test was conducted on Dry Sand Abrasion Tester (Make: Ducom TR-50). The schematic diagram is shown in figure 4. The size of specimen was kept 76 mm × 25 mm × 78 mm as per ASTM G65 standard as shown in figure 5. Test specimen was fixed in tool holder and the required normal load was applied in the way that the face of the abraded specimen was in contact with the rotating wheel. The rotating wheel with a chlorobutyl rubber tire, freely flowing quartz sand (constant flowing rate 365 g min⁻¹) and the specimen created the scenario of three body abrasion. Wear rate was determined by measuring the loss of weight of the specimen. Volume loss and the specific wear rate is determined by the equations (8) and (9) as reported by Agarwal et al [25].

\[ \Delta V = \frac{\Delta w_1 - \Delta w_2}{\rho} \times 1000 \]  

(8)

Where,
\[ \Delta w_1 = \text{Initial weight before test.} \]
\[ \Delta w_2 = \text{Final weight after test.} \]
\[ \Delta V = \text{Volume loss of specimen.} \]
\[ \rho = \text{Density of the specimen.} \]
Specific wear rate of the specimen is determined by following equation (9).

\[ W_s = \frac{\Delta V}{F_s \times S_s} \tag{9} \]

Where, \( W_s \) = specific wear rate (mm\(^3\)/Nm), \( F_s \) = normal load (N) and \( S_s \) = sliding distance (m).
2.6. Experimental design
The Taguchi method is generally used to minimize the large number of repetition to the fewer steps. Rather than the all desirable solutions, Taguchi method gives optimum result for all the desirable solution. This method minimizes the number of experiments and time \[25\]. The specific wear rate of different chemically treated HAF/polyester composite was evaluated by using the combination of three factor with three levels individually as shown in tables 2–5. Using experimental point of view, the impact of three factor and three variables can be studied by using L9 orthogonal array for each chemical treatment. The S/N ratio for three factors and three variables are determined by selecting smaller-is-better characteristic since our purpose is to reduce the specific wear rate to be minimized as given in equation (10). Later, ANOVA (analysis of variance) was used to examine the significant contribution of each selected parameter on dry abrasive wear rate.

\[
\frac{S}{N} = -10 \times \log \left( \frac{1}{n} \sum y^2 \right)
\]  

(10)

Where \(S/N\) = signal to noise ratio, \(n\) = number of experiment, and \(y\) = data observed.

2.7. Surface morphology
Scanning electron microscope (SEM) analysis was taken on ZEISS EVO 18 on worn surface of different mechanical test specimen.
Table 5. Experimental design using L₉ orthogonal array for fabricated composite.

| Taguchi runs | Concentration (%) | Load (N) | Speed (rpm) | \( \text{Specific wear rate (SWR) (mm}^3/\text{Nm)} \) | \( \text{S/N ratio (db)} \) |
|--------------|-------------------|----------|-------------|-------------------------------------------------|-----------------------------|
|              |                   |          |             | \( SWR_{KOH} \) | \( SWR_{KMnO_4} \) | \( SWR_{NaHCO_3} \) | K-composites | P-composites | C-composites |
| 1            | 5                 | 10       | 50          | 0.0518           | 0.0660           | 0.0215           | 25.7134      | 23.6091      | 33.3512      |
| 2            | 5                 | 20       | 100         | 0.0639           | 0.0586           | 0.0314           | 23.8900      | 24.6420      | 30.0614      |
| 3            | 5                 | 30       | 150         | 0.0592           | 0.0612           | 0.0479           | 24.5536      | 24.2650      | 26.3933      |
| 4            | 10                | 10       | 100         | 0.0319           | 0.0361           | 0.0111           | 29.9242      | 28.8499      | 39.0935      |
| 5            | 10                | 20       | 150         | 0.0467           | 0.0596           | 0.0322           | 26.6137      | 24.4951      | 29.8429      |
| 6            | 10                | 30       | 50          | 0.0644           | 0.0639           | 0.0416           | 23.8223      | 23.8900      | 27.6181      |
| 7            | 15                | 10       | 150         | 0.0587           | 0.0482           | 0.0501           | 24.6272      | 26.3391      | 26.0032      |
| 8            | 15                | 20       | 50          | 0.0456           | 0.0402           | 0.0443           | 26.8207      | 27.9155      | 27.0719      |
| 9            | 15                | 30       | 100         | 0.0548           | 0.0709           | 0.0554           | 25.2244      | 22.9871      | 25.1298      |
3. Results and discussion

3.1. Physical properties of HAF/polyester composite

The void fraction and water uptake capacity of different composites is shown in Figure 6. Theoretical and experimental density was calculated for determining the voids fraction in the composite, and the water absorption of HAF/polyester composite with different concentration of chemical treatment was studied. The compression molding at a high magnitude of loading (70–80 kN) may be envisaged to reduce the creeping and promotes the fiber bridging [26–28] of the composite during curing which may lead to reduce the overall void content and water absorption of the fabricated composites as reported earlier.

It was observed that the extent of moisture/water absorption of surface-treated fibers was reduced significantly which might had happened due to creation of hydrophobicity over the fiber surface via long chain hydrocarbon attachment. The different surface modification showed an improvement in the interfacial adhesion between the hydrophilic fiber and the hydrophobic polymer matrices. Natural fibers are chemically treated to remove lignin, waxy substances, and natural oils covering the external surface of the fiber cell wall. The chemical treatment was reported to remove the hemicellulose and lignin and the fiber surface became coarser than the untreated fiber [29, 30]. The maximum weight gain of HAF/polyester composite was observed in 5% potassium permanganate treatment due to excess hole and air bubble present in the composite. Least void fraction and water uptake was observed in 10% sodium bicarbonate treated HAF based composite due to maximum reduction of non-cellulosic component.

3.2. Tensile properties of composite

The mechanical properties of fiber reinforced composites depend on the intrinsic property of reinforcing fiber and matrix, fiber–matrix adhesion, aspect ratio, length distribution and orientation of fiber in the fabricated composites. The tensile strength and tensile modulus of different chemically treated HAF fiber (at 10 wt% of fiber loading) reinforced polyester composite is demonstrated in figures 7(a) and (b) respectively. It is clear from figure 7 that the tensile strength and modulus of KOH and NaHCO₃ treated HAF reinforced polyester composites increase with the increase in the concentration from 5 to 10%, which further decrease at concentration of 15% which may be due to better fiber–matrix adhesion and so better distribution of stress from fiber to matrix. Beyond 10% concentration in case of KOH and NaHCO₃, it is observed that the tensile strength and modulus are slightly decreased due to excess removal of hemicellulose content, which makes the fiber more brittle and stiff leading to decreased strength and modulus of composite. As reported by Rokbi et al [31], sisal fiber–reinforced polyester composite exhibited superior tensile strength at 10% alkali treated fiber based composite which showed excess delignification of natural fiber beyond 10% alkali treatment. Potassium hydroxide (KOH) treated fiber 10% (K10/10), showed the maximum strength and modulus of 134.5 MPa and 1.6 GPa respectively. While with KMnO₄ treated fiber, the tensile strength and tensile modulus are found to increase with increase in the concentration of KMnO₄ showing maximum tensile strength i.e. 135.2 MPa at 15% concentration, which may be correlated to the fact that the KOH has more aggressive etching or surface modifying tendency than that of KMnO₄ and therefore KMnO₄ enables better result at higher concentration i.e. at 15% than the KOH treatment. Overall, the sodium bicarbonate treated composite imparts best tensile properties at 10% concentration expressing highest tensile strength of 145 MPa and modulus of 1.72 GPa.
delivering the tensile strength about 5.7 times and tensile modulus 1.17 times higher than without treated 7 mm fiber at 9 wt% loaded in polyester resin in earlier work [27].

3.3. Flexural strength of composite

Figures 8(a), (b) represent the flexural strength and modulus of polyester composites fabricated by reinforcing different chemically treated HAF fiber. A similar trend is shown in flexural properties as tensile properties. The concentration of chemical treatment significantly affects the flexural properties of the composite. It is clear from the figure 8 that, from 5% (K5/10) to 10% (K10/10) concentration of KOH, the flexural strength and modulus are increasing due to fiber capability to fill up the apparent flaws resulting in better sharing of flexural load [32]. But after 10% concentration i.e. 15% KOH treated fiber (K15/10), it slightly decreases due to failure of specimen’s cross-bonding by breakage and pull out of the fiber from the resin matrix [33]. In case of KMnO₄ treated HAF composite, as the concentration of the KMnO₄ increases from 5% (P5/10) to 15% P1(5/10), the flexural strength and modulus reveal an increasing trend. This indicates that as the concentration of KMnO₄ increases the bond strength between the fiber and the matrix due to the formation of cellulose radical through MnO⁻ ion formation [34]. As carbonate (NaHCO₃) treated composite, the similar trend is observed as shown by KOH treated composite. The 10% (C10/10) NaHCO₃ treated composite has delivered best flexural strength and modulus i.e. 214.5 MPa and 4.46 GPa. This indicates that the 10% sodium bicarbonate treated composite has better interfacial bonding between fiber and matrices, better load bearing capacity and higher stiffness than the others.

3.4. Impact strength

The impact strength of a composite is governed mainly by two factors: first, the capability of the filler to absorb energy that can stop crack propagation and second, poor interfacial bonding which induces micro-spaces
between the filler and the matrix resulting in easy crack propagation [17]. From figure 9, It is interpreted that the impact strength of 10% (K10/10) KOH and 15% (P15/10) KMnO4 treated HAF based composite exhibit good impact strength i.e. 2.4 and 1.3 J cm$^{-2}$ respectively as compared to other treated composite due to high energy absorption capacity with low fiber pull out and with good interfacial bond between fiber and matrix [13, 17]. But whereas the sodium bicarbonate (NaHCO$_3$) treated fiber at 10% (C10/10) concentration shows highest value i.e. 3.65 J cm$^{-2}$. This indicates that NaHCO$_3$ treatment has enhanced the surface roughness of the fiber, which has enhanced the adhesion between reinforced agave fiber and matrix [19]. From figures 7 and 8, the tensile and flexural strength of 10% (C10/10) have maximum strength which conclude that composite with superior mechanical properties also possesses the highest impact strength.

3.5. Hardness

Hardness is basically a function of the respective fiber volume and modulus. The graph of hardness of different chemically treated HAF reinforced composite is shown in figure 10. As the concentration of KOH treatment of HAF increases from 5 to 10%, composite gains the hardness to a maximum value of 28.4 H$_{v}$ which further drops with higher concentration. Basically the treated fiber that increase the moduli of fabricated composites has a tendency to increase the hardness of the composite [35]. While KMnO$_4$ treatment of HAF at all concentration of fiber does not change the hardness of respective composite. The sodium bicarbonate treatment of HAF improves the hardness up to 10% which decreases further as similar to the pattern of KOH treatment. The 10%
sodium bicarbonate (NaHCO₃) treated HAF based composite expresses greatest harness of 30.33 Hv among all composites.

3.6. Analysis of three body abrasive wear result of HAF/polyester composite using Taguchi technique

The experimental wear output was analysed by Taguchi method and the control factor affecting the material abrasion was identified. L₉ orthogonal array was chosen according to the assigned experiment as presented in table 6. The ranking of the control factor according to their contribution on specific wear rate was found out by delta value (subtraction of the maximum and minimum value of S/N ratio). The highest value of delta had a greater effect on the specific wear rate as tabulated in table 7. The plot of the mean of S/N ratio versus considered control factor is depicted in figures 11–13. The factor combination of A₂ B₁ C₂ (10% of concentration, 10 N of normal load, and 100 rpm of speed) delivered minimum specific wear rates for KOH, KMnO₄, and NaHCO₃ treated HAF/polyester composites. It was concluded that the abrasion wear rate decreased with increase in the concentration from 5% to 10% which further increased from 10% to 15%. Conversely, the inferior wear properties were obtained as the normal load and abrading distance increased from 10 to 30 N and 100 to 150 rpm. For similar test conditions, NaHCO₃ treated HAF/polyester composites exhibited higher wear resistance among all treated composites. This work establishes that the wear resistance property of the HAF/polyester composites depends upon the fiber treatment methods and level of concentrations.

| Table 6. Response table for S/N ratios (smaller is better) for K, P, and C-composites. |
|----------------------------------------|--------|--------|--------|
| Level | Concentration (%) | Load (N) | Speed (rpm) |
|-------|--------------------|----------|-------------|
| A      | B                  | C        | D           |

| Average SN ratios for K- composites (db) | 1 | 24.72 | 26.75 | 25.45 |
|-----------------------------------------|---|-------|-------|-------|
| 2 | 26.79 | 25.77 | 26.35 |
| 3 | 25.56 | 24.53 | 25.26 |
| Delta | 2.07 | 2.22 | 1.08 |
| Rank | 2 | 1 | 3 |

| Average SN ratios for P-composites (db) | 1 | 24.17 | 26.27 | 25.14 |
|----------------------------------------|---|-------|-------|-------|
| 2 | 25.74 | 25.68 | 25.49 |
| 3 | 25.75 | 23.71 | 25.03 |
| Delta | 1.58 | 2.55 | 0.46 |
| Rank | 2 | 1 | 3 |

| Average SN ratios for C- composites (db) | 1 | 29.94 | 32.82 | 29.35 |
|----------------------------------------|---|-------|-------|-------|
| 2 | 32.18 | 28.99 | 31.43 |
| 3 | 26.07 | 26.38 | 27.41 |
| Delta | 6.12 | 6.44 | 4.02 |
| Rank | 2 | 1 | 3 |

| Table 7. ANOVA results for K, P, and C-composites. |
|----------------------------------------|--------|--------|--------|
| Source | DF | Adj SS | Adj MS | F-Value | P-Value | Rank |
|---------|----|--------|--------|---------|---------|-------|

Panel 1: ANOVA results for K-composites

| Factor | DF | Adj SS | Adj MS | F-Value | P-Value | Rank |
|--------|----|--------|--------|---------|---------|-------|
| Concentration (%) | 2 | 6.490 | 3.245 | 0.48 | 0.675 | 2 |
| Normal load (N) | 2 | 7.437 | 3.718 | 0.55 | 0.645 | 1 |
| Speed (rpm) | 2 | 2.004 | 1.002 | 0.15 | 0.871 | 3 |
| Error | 2 | 13.496 | 6.748 | | | |
| Total | 8 | 29.427 | | | | |

Panel 2: ANOVA results for P-composites

| Factor | DF | Adj SS | Adj MS | F-Value | P-Value | Rank |
|--------|----|--------|--------|---------|---------|-------|
| Concentration (%) | 2 | 4.955 | 2.478 | 0.30 | 0.772 | 2 |
| Normal load (N) | 2 | 10.733 | 5.366 | 0.64 | 0.610 | 1 |
| Speed (rpm) | 2 | 0.349 | 0.174 | 0.02 | 0.980 | 3 |
| Error | 2 | 16.779 | 8.389 | | | |
| Total | 8 | 32.815 | | | | |

Panel 3: ANOVA results for C- composites

| Factor | DF | Adj SS | Adj MS | F-Value | P-Value | Rank |
|--------|----|--------|--------|---------|---------|-------|
| Concentration (%) | 2 | 57.426 | 28.713 | 4.37 | 0.186 | 2 |
| Normal load (N) | 2 | 62.860 | 31.430 | 4.78 | 0.173 | 1 |
| Speed (rpm) | 2 | 24.193 | 12.096 | 1.84 | 0.352 | 3 |
| Error | 2 | 13.140 | 6.570 | | | |
| Total | 8 | 157.618 | | | | |
3.7. ANOVA and the effects of factors

ANOVA was accomplished on experimentally evaluated wear result to find out the percentage of contribution of various selected control factors such as chemical concentration, normal load, and speed under dry sand abrasion condition. The ANOVA results for both fabricated composites taken under this research is shown in table 7 (panel 1), panel 2, and panel 3. The Sixth column of the table explains the contribution of selected control factors.
Figure 12. Contribution of control factors on the specific wear rate of K, P, and C-composites.

Figure 13. SEM micrograph of K-composite (a), P-composites (b), and C-composites (c) at 10% concentration and 10 N load.
on the specific wear rate (abrasive). From table 6, for K-composites, normal load shows the highest contribution 46.68%, concentration exhibits moderate contribution of 40.74%, and speed shows less contribution of 12.58%. Likewise, in the situation of P-composites, the contribution arrives as normal load (66.93%), concentration of 30.9%, and speed of 2.18%. Moreover, for C-composites, the contribution is evaluated as normal load (43.51%), concentration (39.75%), and speed (16.74%). The ANOVA results indicate that the normal load is most significant factor influencing the specific wear rate (abrasive) of composites are presented in figure 12.

3.8. SEM analysis

This conclusion is further supported by the SEM (scanning electron microscopy) micrographs of worn samples at different conditions for finding the predominant wear mechanism. Basically, dry sand abrasive wear mechanism of composites depends on different mechanisms such as ploughing, micro-cutting, cracking, fiber pull out and debonding [36]. It should be noted that in three-body abrasion experiment, the abrasive particles which act as third body come in contact with composite material mainly by any of the following three ways: (1) Freely falling sand particles gain energy when come in contact with the rubber wheel, and hit the material, result in the formation of pits, plastic deformation either of the matrix or fiber; (2) sand particles become ingrained in the softer rubber wheel which slide over the material surface and (3) sand particles roll between the rubber wheel and material resulting in plastic composite material deformation [37]. The optimum wear rate of values 0.0319 mm³ N⁻¹ m⁻¹, 0.0361 mm³ N⁻¹ m⁻¹, and 0.0111 mm³ N⁻¹ m⁻¹ are obtained at 10% chemical concentration and 10 N load (Exp. 4, table No. 5) for K, P and C-composites respectively. Figures 13(a)–(c). shows the worn surface of the composites with 10% of chemical treatment subjected to normal load of 10 N with three variations like potassium hydroxide, potassium permanganate and sodium bicarbonate treated respectively. Figure 13(a) reveals a higher wear resistance as compared to the potassium permanganate treated HAF composites because of possible lower cellulosic content presence in K-composites as compared to P-composite. The better adhesion is manifested with less fiber breakage and crack in figure 13(a) as compared to figure 13(b). The inclusion of sodium bicarbonate treated fiber in figure 13(c) greatly minimizes the breaking of fiber and ploughing in worn abraded surface encountering a more uniform stress distribution at interface with the lower magnitude which leads to create a more protective surface against abrasive wear. As per the result, figure 13(c) shows minimum wear as compared to figure 13(b) and 13(c). With the load increment from 10 to 30 N, the fracture of the fiber or damage of the matrix occurs which results in more surface damage as shown in
Based on the results and discussion, the following major conclusions are drawn.

1. The physical properties such as void fraction and water absorption were observed to be least with 10% sodium bicarbonate treated HAF composite due to higher decrement in the hydrophilicity in the fiber and better adhesion between fiber and matrix.

2. The optimal values of tensile strength, flexural strength, impact strength of composite were achieved at 10 wt% concentration of both KOH and NaHCO₃ treatment, and 15 wt% concentration for KMnO₄ treatment. The 10% sodium bicarbonate treated HAF-polyester composite is emerged as best treatment delivering highest magnitude of tensile strength of 145 MPa, tensile modulus of 1.72 GPa, flexural strength of 214.5 MPa, flexural modulus of 4.46 GPa and impact strength of 3.65 J cm⁻².

3. Hardness of sodium bicarbonate treated HAF-polyester composite achieved greatest magnitude at 10% concentration however the potassium paramagnet treated HAF-polyester exhibited no changes in hardness.

4. Taguchi analysis revealed that the abrasion wear rate decreased with increase in the concentration from 5 to 10% which further increased from 10 to 15%. Conversely, the inferior wear properties were obtained as the normal load and abrading distance increased from 10–30 N and 100–150 rpm. NaHCO₃ treated HAF/polyester composites exhibited highest wear resistance among all treated composites.

5. SEM analysis of the worn surface of different treated HAF-polyester composite revealed the fiber breakage, debonding, debris, crack surface and micro-ploughing as the prominent wear mechanism.

6. The contribution ratio of control factors for concentration of treatment was achieved in K-composite (46.68% of concentration, 40.74% of load and 12.58% of speed), P-composite (66.93% of concentration, 30.9% of load and 2.18% of speed) and C-composite (43.51% of concentration, 39.75% of load and 16.74% of speed).

7. This work establishes that the eco-friendly sodium bicarbonate treatment is most effective chemical treatment among all. KOH treatment and KMnO₄ treatment exhibited comparable Physicomechanical and abrasive wear properties with optimum results obtained at 10% KOH treatment and 15% KMnO₄ treatment.

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