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친환경 공학 응용을 위한 *Cissus Quadrangularis* 줄기 및 바나나 섬유 기반 고분자 복합재 연구

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Investigation on *Cissus Quadrangularis* Stem Fiber Reinforced Novel Polymeric Composite with Banana Chopped Fiber for Green Engineering Applications

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Abstract: This study investigates the potential of a novel hybrid composite material made from *Cissus quadrangularis* fibers (CQF) and banana fibers (BF) for use in sustainable engineering and construction applications. Using the hand layup technique, the composite was fabricated with varying CQF content (0 wt% to 100 wt%) in an epoxy resin matrix. Analytical techniques, including Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM), revealed significant structural modifications in the treated fibers. Mechanical and tribological tests assessed the hardness, wear rate, and coefficient of friction. Results showed that the composite with 20 wt% CQF had an 11.32% increase in hardness, while the 100 wt% CQF composite achieved a 1.4% reduction in wear rate. The composite with 60 wt% CQF notably improved the coefficient of friction. This research demonstrates the potential for using CQF and BF to create sustainable composite materials with enhanced performance properties.

Keywords: natural fibres, epoxy resin, hand layup, morphology, tribology characteristics.

Introduction

Natural fibers have gained significant attention in recent years as reinforcing materials in composite structures due to their eco-friendliness, cost-effectiveness, and excellent mechanical properties.[1-8] *Cissus quadrangularis*, commonly known as the Veldt Grape, is a perennial plant widely used in traditional medicine. The fibers extracted from this plant are known for their high strength and durability, making them suitable for reinforcement in composite materials. Banana Fiber (BF), derived from the pseudostem of the banana plant, is another natural fiber with significant potential in composite materials.[9-14] This study focuses on a new composite made from stem fiber (CQSF) and chopped banana fiber. Unlike other studies that mainly use fibers like jute, sisal, or flax, we combine CQSF and banana fibers to create a stronger, more durable material. Most past research has looked at single fiber composites, but this work examines the synergistic effect of using two fibers together. We

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also treat the fibers with alkali, which helps them bond better with the polymer, improving the composite's performance. This study offers a greener, more sustainable alternative for applications in areas like automotive, aerospace, and construction.[15] X-ray diffraction (XRD) analysis of CQF reveals its crystal-line nature, which contributes to its high mechanical strength. The characteristic peaks in the XRD pattern indicate the presence of cellulose, hemicellulose, and lignin in the fiber structure.[16-21] Fourier-transform infrared (FTIR) spectroscopy provides insight into the functional groups present in CQF, confirming the pres-ence of hydroxyl groups, which are essential for bonding with polymer matrices in composites FTIR spectra of BF reveal the presence of functional groups such as hydroxyl, carboxyl, and acetyl, which facilitate interaction with polymer matrices.[22-23] Scanning electron microscopy (SEM) images of CQF show a rough surface morphology, which enhances the interfacial adhesion between the fiber and the polymer matrix.[24-26] SEM analysis of Banana Fibers demonstrates a relatively smooth sur-face with some fibrillation, which can contribute to good inter-facial bonding in composites.[27] Mechanical properties such as hardness, tensile strength, flex-ural strength, and impact resistance are critical for evaluating the performance of composite materials and their studies have shown that the inclusion of CQF with banana composites enhances hardness, tensile strength and modulus due to its high cellulose content and crystalline nature.[28-31] Tribological studies, which focus on the friction, wear, and lubrication properties of materials, are essential for evaluating the performance of composites in dynamic applications.[32-33] The inclusion of fibre composites has been shown to reduce the coefficient of friction and wear rate due to its high hardness and surface roughness with its relatively smooth surface, con-tributes to reduced wear by forming a protective layer during sliding contact.[34-40] The implication of natural fibers offer substantial benefits as reinforcement materials in composite applications. and their favorable characteristics, including high tensile strength, hard-ness, good interfacial bonding with matrices, and excellent wear resistance, make them viable alternatives to synthetic fibers in various engineering applications.[41-50]

Experimental

Materials and Methods. Materials: Natural fibers were sourced from the mature stems of *Cissus quadrangularis* and banana plants. The *Cissus quadrangularis* fibers, which do not contain pectin, were collected from Trichy, India. The extraction process involved harvesting the stems, retting, and subsequent processing. In contrast, the banana stems, containing pectins ranging from 0.15% to 1%, were collected from Lagudi, India. These banana stems were washed, dried, and mechanically processed using a hammering technique to extract the fibers, as shown in Figure 1. Epoxy resin, selected for its excellent adhesive properties and mechanical strength, was used as the matrix material for the composite.

Figure 1. Fibre Extraction: (a) cisuss quadrangularis stem; (b) *cissus quadrangularis* fiber; (c) banana stem; (d) banana fiber.

Fiber Treatment: To enhance the bonding of natural fibers with the epoxy matrix, an alkaline treatment was applied to both types of fibers. This process involved preparing a 5% calcium hydroxide ($Ca(OH)_2$) solution by dissolving $Ca(OH)_2$ in distilled water. The fibers were immersed in this solution for 2-3 days to remove impurities and increase surface roughness. After immersion, the fibers were thoroughly rinsed with distilled water to eliminate any residual $Ca(OH)_2$, as shown in Figure 2. Finally, the cleaned fibers were dried at laboratory oven controlled temperature (36-38 ℃) for 1-2 days to ensure complete moisture removal.

Analysis of Fibres. FTIR Analysis: Fourier Transform Infrared (FTIR) analysis is a powerful technique is used to identify and characterize chemical compounds based on their

Figure 2. Fibre treatment.

absorption of infrared light. By measuring the specific wavelengths of light absorbed by sample molecules, FTIR spectroscopy provides valuable information about molecular structure and functional groups present as shown in Table 1.

The FTIR analysis of *Cissus quadrangularis* fiber and banana fibre before and after treatment reveals significant modifications in its chemical structure are shown in Figure 3.

Before treatment, the fiber shows characteristic peaks indicating the presence of functional groups such as O-H stretching (3328.15 cm^{-1}), C-H stretching (2916.28 cm^{-1}, 2848.48 cm^{-1}), C=C stretching from alkenes or aromatic rings (1654.32 cm^{-1}, 1637.24 cm^{-1}, 1617.85 cm^{-1}), N-H bending (1541.57 cm^{-1}), and C-O stretching (1241.81 cm^{-1}, 1028.25 cm^{-1}). Post-treatment, there are noticeable shifts and new peaks, such as O-H stretching at 3328.37 cm^{-1}, C≡C or C≡N stretching at 2037. 90 cm^{-1} and 1800.45 cm^{-1}, and altered C-O stretching peaks, indicating the introduction of new functional groups and changes in hydrogen bonding and the bonding environment.

Similarly, the FTIR analysis of banana fiber shows significant structural changes after treatment. Before treatment, the fiber's spectrum displays peaks associated with O-H stretching (3328.80 cm^{-1}), C-H stretching (2916.91 cm^{-1}), C=C stretching (1601.75 cm^{-1}), and various C-O stretching peaks (1242.20 cm^{-1}, 1159.41 cm^{-1}, 1032.01 cm^{-1}). Post-treatment, the spectrum indicates major shifts and new peaks, such as O-H stretching at 3349.11 cm^{-1}, C≡C or C≡N stretching at 2181.02 cm^{-1}, 2019.81 cm^{-1}, and 1978.01 cm^{-1}, and changes in C-O stretching peaks, signifying the incorporation of new functional groups and modifications in the existing ones. These observations highlight the chemical alterations induced by the treatment process, which can affect the fibers' properties and potential applications.

X-Ray Diffraction Analysis: X-ray diffraction (XRD) is a powerful analytical technique used to determine the crystallographic structure, chemical composition, and physical properties of materials. In XRD, a sample is bombarded with X-rays generated by a cathode ray tube, typically with a copper anode. The X-rays diffract upon interacting with the crystal lattice of the material, and these diffracted rays are detected using a detector, usually a scintillation or charge-coupled device (CCD) detector. The resulting diffraction pattern, which shows intensity versus angle (2θ), is unique to the material's structure and is analyzed using software to identify phases and crystal structures. The primary equipment components include the X-ray source (usually a Cu-Kα radiation source), a sample holder, a goniometer for precise angle measurements, and the detector. Sample preparation involves finely grinding the material to ensure homogeneity.The XRD analysis of *Cissus quadrangularis* fiber and banana fibre before and after treatment reveals significant modifications in molecular arrangements are shown in Figure 4.

Before treatment, the XRD analysis of *Cissus quadrangularis* fibre shows characteristic peaks at 14.274°, 15.068°, and 16.65°. These values indicate the crystalline regions within the fibre's natural structure. After treatment, the XRD peaksshift to 15.172° and 15.687°, suggesting alterations in the crystalline structure due to the treatment process. The changes in peak positions reflect modifications in the molecular arrangement and possible improvements in the fibre's crystallinity, indicating a successful alteration of its structural properties through the treatment process.

Table 1. FTIR Peak Assignment Chart

Wavenumber (cm^{-1})	Functional Group / Bond Assignment	Possible Component
3500–3200	O-H Stretch (Hydroxyl)	Alcohols, Phenols, Water
3300–3100	N-H Stretch	Amines, Amides
3100–3000	C-H Stretch (Aromatic)	Aromatic Rings (Epoxy)
3000–2800	C-H Stretch (Aliphatic)	Alkanes, CH_2, CH_3
1750–1700	C=O Stretch (Carbonyl)	Esters, Aldehydes, Ketones
1650–1600	C=C Stretch (Aromatic)	Aromatic Rings (Lignin, Resin)
1550–1500	N-O Asymmetric Stretch	Nitro Compounds
1450–1400	C-H Bending (Alkanes)	CH_3, CH_2 Groups
1300–1200	C-O Stretch	Ethers, Esters, Alcohols
1150–1000	C-O-C Stretch	Epoxy, Cellulose
900–700	C-H Bending (Aromatic, Out-of-plane)	Aromatic Rings
600–500	Metal-Oxygen Vibrations	Inorganic Fillers

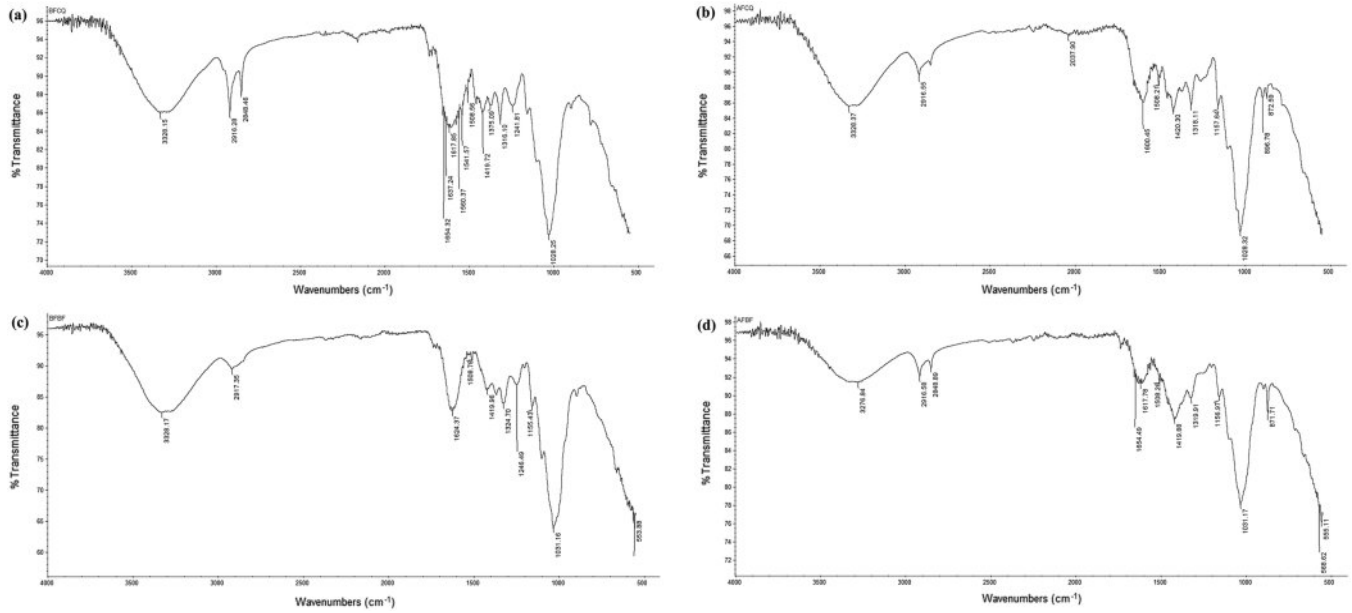

Figure 3. FTIR results of fibre treatments CQF: (a) before; (b) after BF; (c) before; (d) after.

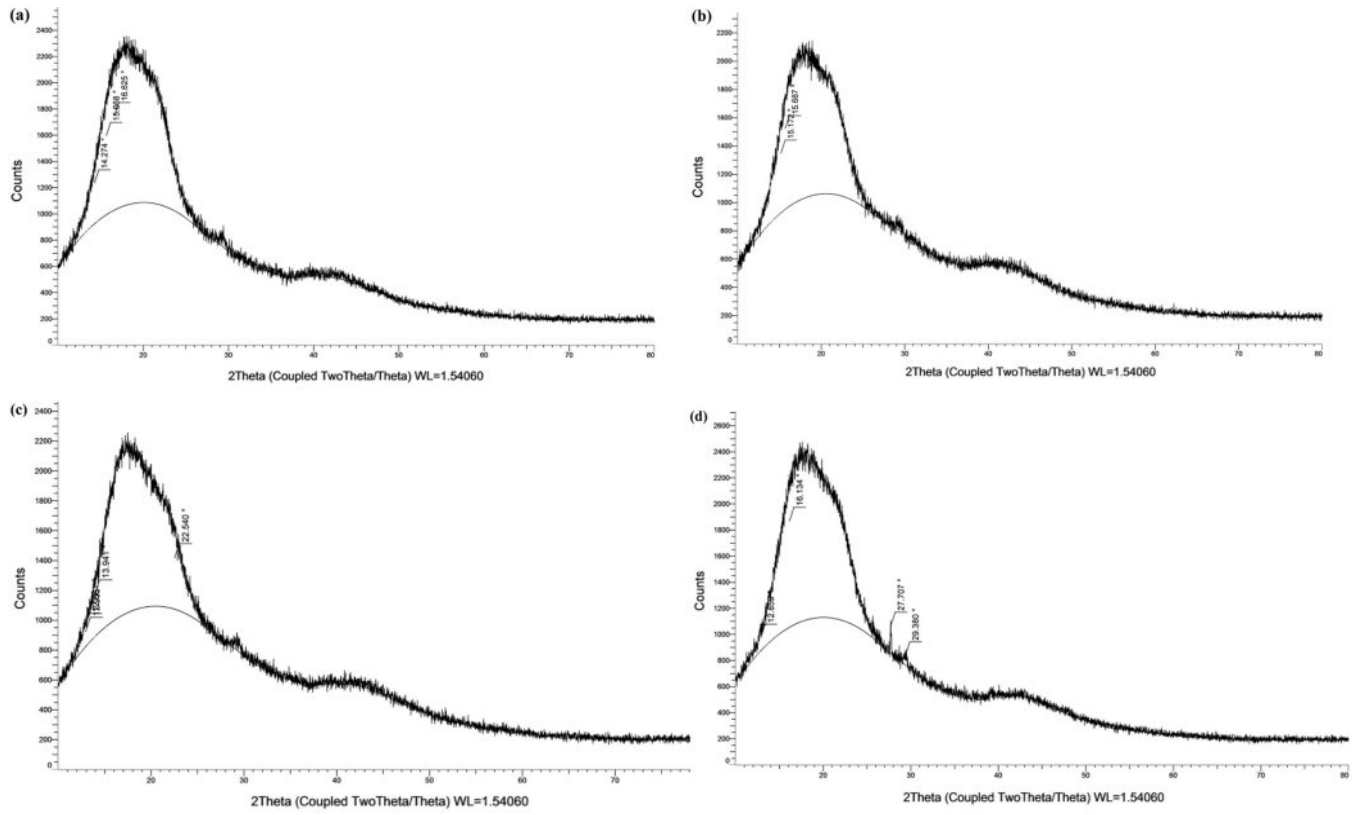

Figure 4. XRD results of fibre treatments CQF: (a) before; (b) after BF; (c) before; (d) after.

In the case of banana fibre, the XRD analysis before treatment reveals peaks at 12.35°, 13.95°, and 22.54°. These peaks represent the fibre's initial crystalline structure. After treatment, the XRD peaks shift to 12.859°, 16.134°, 27.707°, and 29.30°. The emergence of new peaks and the shift in existing ones indicate significant changes in the crystalline structure

of the banana fibre. These changes suggest that the treatment process has effectively modified the fibre's molecular structure, potentially enhancing its crystallinity and overall material properties.

Scanning Electron Microscopy (SEM): In this study, a SEM instrument equipped with high-resolution imaging capabilities was employed to delve into the fiber-matrix interface and fiber dispersion within the composite. The procedure involved meticulously preparing samples by coating them with a thin layer of gold, a common practice aimed at enhancing conductivity and minimizing charging effects during imaging. Micrographs were then captured at 4000X magnification to meticulously scrutinize the microstructural characteristics of the composite material. This multi-scale approach enabled the observation of fine details, such as the alignment of fibers, the distribution of the matrix material, and any potential defects or irregularities present within the composite structure. Scanning Electron Microscopy (SEM) analysis provides detailed visual evidence of the structural changes in *Cissus quadrangularis* and banana fibers before and after treatment with calcium hydroxide, as depicted in Figure 5. Before treatment, Figure 5(a) illustrates *Cissus quadrangularis* fibers with a rugged surface, irregularities, and bundled structures typical of their natural state. The SEM images reveal varying pore sizes and distributions, emphasizing the heterogeneous nature of these fibers. In contrast, Figure 5(c) shows banana fibers with a smoother surface and less pronounced pore distribution, reflecting their inherent structural uniformity.

Following treatment with calcium hydroxide, SEM analysis Figure 5(a) and 5(b) for *Cissus quadrangularis* and Figure 5(c) and 5(d) for banana fibers demonstrate significant modifications in surface morphology and structural integrity. Figure 5(a) and 5(c) indicate that the alkaline treatment has smoothed the surface of *Cissus quadrangularis* fibers, reducing irregularities and potentially enhancing fiber uniformity. Some fibers may exhibit signs of degradation or breakdown, visible in the SEM images, with changes in pore sizes and more defined cross-sectional profiles. Meanwhile, Figure 5(b) and 5(d) illustrate that the banana fibers maintain their structural integrity post-treatment, with minimal surface etching and subtle improvements in pore distribution.

Composite Fabrication. The composite fabrication process began with the preparation of fibers, where treatedfibers were cut into 6 mm lengths. Following this, the epoxy resin and hardener were mixed in a weight ratio of 10:1, adhering to the manufacturer's instructions. During the composite mixing stage, the fibers were evenly distributed within the epoxy resin mixture, by varying a fiber ratio of in sample weight percentage were shown in Table 2. The composite fabrication process began with the preparation of fibers, where treated fibers were cut into 6 mm lengths. Following this, the epoxy resin and hardener were mixed in a weight ratio of 10:1, adhering to the manufacturers instructions. During the composite mixing stage, the fibers were evenly distributed within the epoxy resin mixture, by varying a fiber ratio of in sample weight percentage were shown in Table 2. Subsequently, the mixture is mixed by mechanical stir-

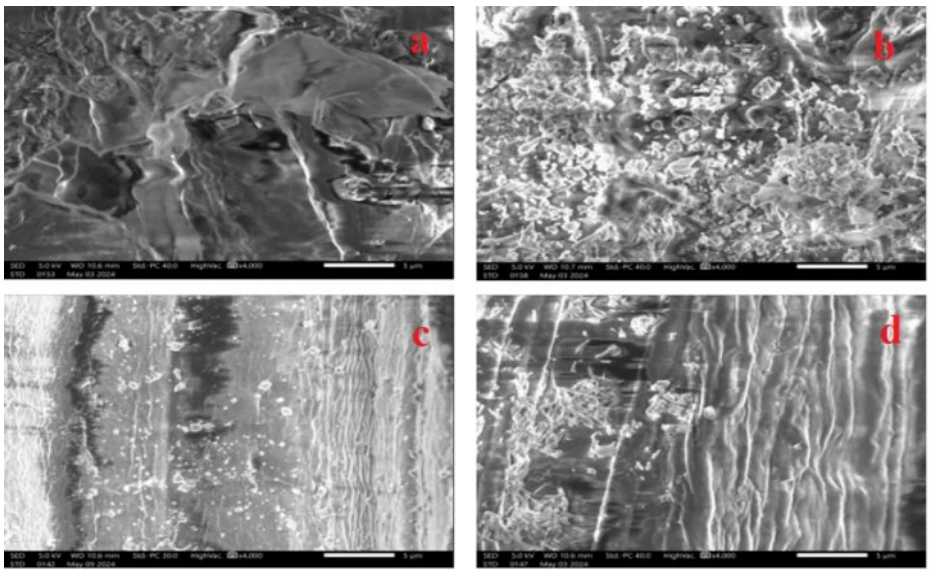

Figure 5. SEM images magnification 4000x fibre treatments CQF: (a) before; (b) after; BF: (c) before; (d) after.

Table 2. Preparation of CQF/SF Specimens

Specimens	wt% of fibers		Binder ratio (Epoxy: Hardener) in wt%	Resin%
	CQF	BF		
S1	0	100	10:1	52.38
S2	20	80	10:1	52.38
S3	40	60	10:1	50
S4	60	40	10:1	46.67
S5	80	20	10:1	41.67
S6	100	0	10:1	33.33

Figure 6. Manufacturing process of the composite specimen.

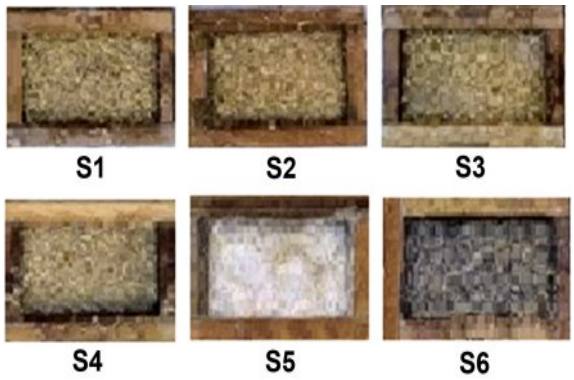

Figure 7. Prepared composite specimens.

rer and it was poured into a mould with dimensions of 80 × 25 × 10 mm using the hand lay-up method shown in Figure 6. Figure 7 Shows the prepared composite specimens (80 × 25 × 10 mm) of CQF and Banana chopped Fibers with different weight percentage as shown in Table 2 and the samples are allowed to cure at room temperature at 33 ℃ for 3 days. Six sample compositions were prepared with varying ratios of CQF (0, 20, 40, 60, 80, and 100% respectivey). Here the samples are named as S1, S2, S3, S4, S5, and S6.

Mechanical Testing. Hardness Test: Hardness testing is a critical process for assessing a material's resistance to deformation, especially permanent indentation. In this particular

Figure 8. Hardness measurement.

series of tests, a Shore D durometer was used, an instrument specifically designed to measure the hardness of harder plastics and rubbers. The testing adhered strictly to the guidelines outlined in ASTM D2240, a standard that details the precise methodology and conditions for determining the hardness of samples using a durometer, as shown in Figure 8. The procedure involved taking ten distinct measurements at various locations on each sample. This method ensures that any potential variability across the sample surface is accounted for, providing a more comprehensive understanding of the material's hardness. After obtaining these measurements, the average value was calculated and recorded as the final hardness value for each sample.

Wear Rate Analysis. Wear rate testing was conducted using a tribometer (pin-on-disk) testing machine, following ASTM G99 standards. In this procedure, a composite sample was mounted onto the machine, with a standard steel pin serving as the counter material. The test was performed under specific parameters: a load of 10 N, a sliding speed of 365 rpm,track radius of 0.04 m and a duration of 660 seconds. The goal was to evaluate the material's resistance to wear under simulated conditions, as illustrated in Figure 9. The wear rate and coefficient of friction were calculated using Eqs. (1) and (2).

$$\text{Wear rate } (k) = \frac{\Delta m}{FL} \tag{1}$$

Where,

Δm = mass loss in grams

F = normal load applied to the material in Newton

L = sliding distance in meters.

$$\text{Coefficient of friction } (\mu) = \frac{F}{F_f} \tag{2}$$

Figure 9. Tribometer.

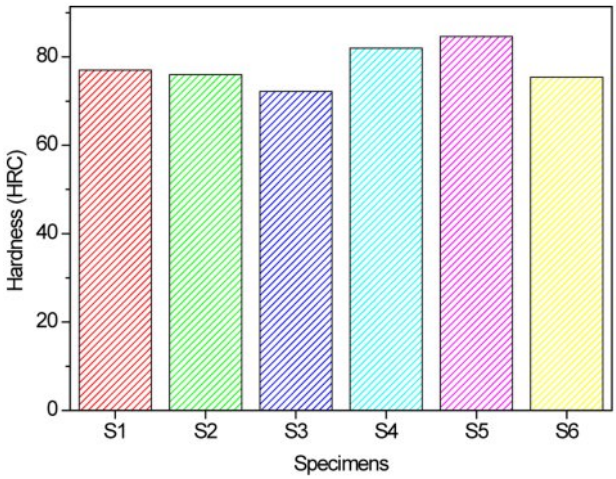

Figure 10. Hardness value of prepared specimens.

Where,

F = normal load in Newton

F_f = Frictional force in Newton

Results and Discussion

Hardness. The hardness of the samples varies, with S5 having the highest value at 84.6, indicating it is the most rigid and least flexible, followed by S4 at 82. S1 and S2 exhibit similar moderate hardness values at 77 and 76, respectively, suggesting balanced rigidity and flexibility. S6, with a hardness of 75.4, is slightly less rigid than S1 and S2. S3 has the lowest hardness value at 72.2, making it the softest and most flexible sample.

In contrast, sample S3, with a hardness of 75.4, is the softest, possibly due to lower cross-linking density, different filler content, or variations in processing conditions. Samples S1, S2, S4, and S6 fall between these extremes, with S4 (82) and S1 (77) closer to the higher end, and S2 (76) and S1 (77) nearer to the lower end. The results of specimen hardness are shown in Table 3 and Figure 10.

Table 3. Results of CQF/BF Specimens

Specimens	Hardness (Shore D)	Wear rate (k) (Kg/Nm)	Co-efficient of Friction (μ)
S1	77	3.96602E-09	0.47
S2	76	9.91505E-10	0.70
S3	72.2	1.98301E-09	0.54
S4	82	1.98301E-09	0.68
S5	84.6	1.98301E-09	0.65
S6	75.4	2.97451E-09	0.57

The relatively small range of hardness values suggests that the samples are likely produced from a similar base material with slight variations in formulation or processing. Consistent material properties are crucial for ensuring predictable performance in applications. Samples with higher hardness, such as S4 and S5, might be preferred for applications requiring higher resistance to deformation and wear, while samples with lower hardness, like S6, could be advantageous in applications where some degree of flexibility or impact resistance is desired.

Wear Rate Analysis. The wear rates among the samples show that S2 has the lowest wear rate at 9.91505×10^{-10} Kg/Nm, indicating the highest resistance to wear. S3, S4, and S5 all share a wear rate of 1.98301×10^{-9} Kg/Nm, demonstrating good wear resistance but not as high as S2, S6, with a wear rate of 2.97451×10^{-9} Kg/Nm, has lower wear resistance com-

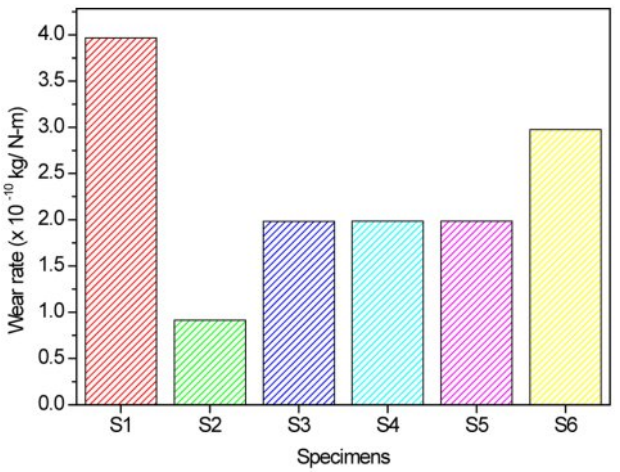

Figure 11. Wear rate of prepared specimens.

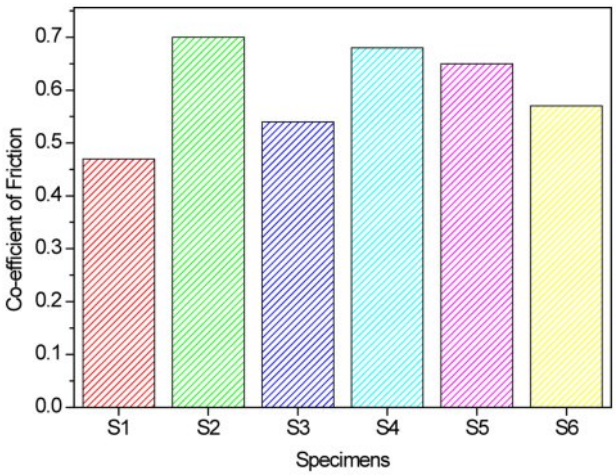

Figure 12. Coefficient of friction of prepared specimens.

pared to S3, S4, and S5. S1 exhibits the highest wear rate at 3.96602×10^{-9} Kg/Nm, indicating the least resistance to wear. The results of specimen hardness are shown in Table 3 and Figure 11.

Coefficient of Friction Analysis. The coefficients of friction for the samples indicate varying levels of resistance to sliding. S2 has the highest coefficient of friction at 0.70, indicating the most resistance to sliding, followed closely by S4 at 0.68 and S5 at 0.65. S6 shows moderate frictional properties with a coefficient of friction of 0.57, while S3 also has a moderate value of 0.54. S1 has the lowest coefficient of friction at 0.47, indicating the least resistance to sliding. .The results are shown in Table 3 and Figure 12.

Conclusions

In this study ciscuss quadrangularis and banana fibers are extacted mechanically and it is chopped.The chopped fibers are subject to alkali treatment. The FTIR, XRD, and SEM analysis are carried out before and after alkali treatment. After alkali treatment, the chopped fibers (reinforcement) are mixed mechanically with epoxy resin (matrix) and finally six composite specimens are S1,S2, S3, S4, S5 and S6 are prepared with the ratio of CSQ:BF (0:100, 20:80, 40:60, 60:40, 80:20, 100:0). For the prepared specimen the different tests such as mechanical analysis for hardness, tribological analysis for wear rate and coefficient of friction is carried out. Based on the comprehensive analysis of fiber treatments and mechanical properties, it is shows that the treatments significantly alter the structural and crystalline characteristics of both *Cissus quadrangularis* and banana fibers. The FTIR and XRD analyses confirm notable shifts in functional groups and crystallinity, respectively, indicating effective modification. SEM observations highlight varied effects of calcium hydroxide treatment on fiber surfaces, with *Cissus quadrangularis* showing smoother surfaces but some degradation compared to banana fibers. For hardness test the specimen S5 exhibits higher hardness. Samples S2 demonstrate superior wear resistance and S2 specimen has high coefficient of friction. Overall, these findings underscore the efficacy of the treatments in enhancing fiber properties, with implications for diverse industrial applications.

Highlights. After alkaline treatment of fibers

- **FTIR:** Possible formation of new compounds such as calcium carbonate
- **XRD:** Formation of new crystalline phases such as calcium silicate hydrates or calcium carbonate. Possible phase transformations due to treatment.
- **SEM:** Physical changes and deposition of treatment products.
- Specimen S5 shows maximum hardness.
- Specimens S2 shows the maximum wear rate.
- Specimens S2 show the maximum coefficient of friction.

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Conflict of Interests: The authors declare that they have no competing interests.

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