

## Enhancement of Epoxy Composites with Benzoylated Fibres of *Demostachya Bipinnata* (Darbha): Impact on Mechanical Properties

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**Benzoylation treatment represents a promising strategy to enhance adhesion between plant fibres and polymer matrices. This study aims to improve the properties of epoxy composites using benzoylated fibres of *Demostachya bipinnata* (Darbha). The analysis focuses on the impact of chemical treatment on the physicochemical and mechanical characteristics of the fibres and resulting composites. After retting and alkaline pre-treatment of the fibres, benzoylation using 10 wt % concentrated benzyl chloride is applied to introduce benzene groups, thereby enhancing the density and chemical stability of the fibres. Results highlight increased density (1869 kg.m<sup>-3</sup>) and enriched crystalline cellulose composition in benzoylated fibres (BDE) compared to untreated fibres (UDE). FTIR analysis confirms significant structural modifications with the introduction of additional carbonyl and carboxyl groups, reinforcing essential interfacial bonds. Mechanical tests reveal 30% higher tensile and flexural strength for epoxy/BDE composites compared to epoxy/UDE composites, demonstrating the effectiveness of benzoylation in improving mechanical properties. Thermal analysis also shows improved thermal stability of BDE fibres, crucial for demanding industrial applications. In summary, this study demonstrates significant enhancement in performance of benzoylated Darbha fibres as composite reinforcements, opening new avenues for their use in sectors such as automotive and construction, where high mechanical strength is crucial.**

**Keywords:** *Demostachya bipinnata* fibre, Benzoylation, Plant fibres, Epoxy composites, Physicochemical properties

### 1 Introduction

The growing global interest in natural fibres is driven by their evident advantages over synthetic fibres, including superior mechanical performance, low density, high flexibility, competitive cost, recyclability, and biodegradability, making them particularly suitable for fibre-reinforced composites [1–6]. Natural fibres such as flax, hemp, jute, and henequen are commonly explored as viable alternatives to glass fibres, despite their often-deficient interfacial properties due to their hydrophilic nature. To address this limitation, various chemical, biological, and physical treatments such as silane treatment, alkaline treatment, acetylation, enzymatic treatment, maleic coupling, plasma, and corona treatment are applied to enhance fibre-matrix

adhesion [3,7–11].

Chemical modification plays a crucial role in optimising interfaces between fibres and polymeric matrices. These treatments reduce interfacial barriers and create a more reactive fibre surface. Chemical coupling agents are designed to react with the hydroxyl groups of fibres and functional groups of the matrix, thereby improving interlocking and enhancing mechanical properties of the composites. For instance, alkaline treatment (NaOH) increases fibre surface roughness and reaction sites, while silane treatment enhances tensile strength. Additionally, alkaline treatment followed by acetylation offers increased thermal stability, while benzyl chloride treatment reduces fibre hydrophilicity and enhances fibre-matrix adhesion and thermal stability [12].

Benzoylation, particularly promising, has shown significant results. For example, it has been successfully applied to *Acacia pennata* to improve its mechanical, thermal, and structural properties [13]. Similarly, benzoylation of sisal fibres increased their crystallinity and thermal stability [14,15], while sugar palm fibres benefit from improved interfacial shear strength (IFSS) due to this treatment [14]. These advantages position benzoylation as an effective method to enhance fibre-matrix adhesion, reduce hydrophilicity, and improve mechanical and thermal performances of composites [8,14,15]. Furthermore, various natural fibres from grasses such as Mendong grass, Snake grass, Kusha grass, Elephant grass, Napier grass, Broom grass, and Belulang grass are currently being studied for their potential in composites, as well as their physical and chemical characteristics [16]. Among these, *Demostachya bipinnata*, commonly known as Kusha or Darbha in India, holds recognised medicinal value for its anti-inflammatory, antimicrobial, and analgesic properties, frequently mentioned in literature [17–19].

Darbha fibres treated with peroxide and benzyl chloride have been specifically studied for their acoustic properties [20]. Our previous research has also demonstrated the benefits of benzoylation of *Lycium ferocissimum* in enhancing mechanical performance and hydrophilicity resistance of epoxy composites [21]. Additionally, integrating *Demostachya bipinnata*

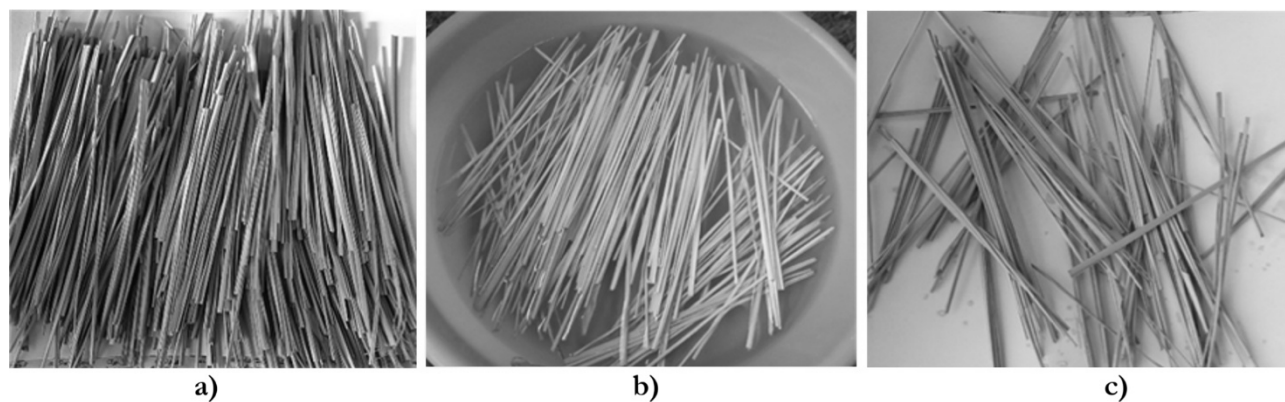
up to 5% in polymer composites has led to a significant increase in tensile strength while maintaining low compressive strength [22,23].

This study thus focuses on exploring the effects of benzoylation on *Demostachya bipinnata* fibres, aiming to deepen our understanding of the physicochemical interactions between modified fibres and polymeric matrices. It also aims to assess the potential enhancements in performance of the resulting composites from this innovative treatment.

## 2 Materials and methods

### 2.1 Extraction, Alkaline Pre-treatment, and Benzoylation Treatment of Fibres

*Demostachya bipinnata* plant, commonly known as Darbha fibre and belonging to the Poaceae family, is a perennial herb widely distributed in Asia. The stalks used in this study as raw materials were harvested from Chennai, Tamil Nadu, India, for this study. After harvesting, the stalks undergo manual retting following established standards to facilitate fibre extraction. Following this step, the inner fibres were gently separated from the outer sheath and thoroughly cleaned with distilled water. The prepared fibres were then dried in a hot air oven at 65°C for one day to ensure complete drying, obtaining the untreated Darbha fibres (Fig. 1a) required for subsequent processes.



**Fig. 1** a) Untreated DP stems; b) Stems bathed in 10 wt% benzyl chloride; c) Treated DP stems

The untreated Darbha fibres underwent an alkaline pre-treatment using a 5 wt% NaOH solution for approximately 30 minutes at room temperature ( $30 \pm 2$  °C), followed by thorough washing with distilled water to remove any NaOH residue. This alkaline treatment activates the fibre surfaces by introducing O-Na groups [23].

Subsequently, in the benzoylation process, the alkali-treated fibres were immersed in a 10 wt% benzyl chloride solution for 2 hours at room temperature ( $30 \pm 2$  °C) (Fig. 1b). This treatment aims to replace surface O-Na groups with O-C<sub>6</sub>H<sub>5</sub> groups, while depositing NaCl salt [9,24]. Any unreacted residues of salt and benzyl chloride were removed through multiple

careful rinses with distilled water and ethanol. Finally, the treated stems (Fig. 1c) were dried at 80°C in a ventilated oven for 12 hours to stabilise the obtained chemical structure. The untreated and treated stems were designated as UDE and BDE, respectively.

### 2.2 Preparation of Epoxy Composites Using Darbha stem

The examined composites were manufactured using a well-established open moulding technology. A square mould measuring 300 mm × 300 mm × 5 mm was prepared by first applying a thin layer of Manson polishing wax to facilitate demoulding, followed by a coating of PVA to prevent composite adhesion.

The adhesive was prepared by mixing epoxy resin with its hardener in a mass ratio of 10:1. Subsequently, Darbha fibres, pre-cut to a length of 10 mm and weighed to achieve a mass fraction of 25 wt%, were added to this adhesive and mixed for approximately 20 minutes to ensure homogeneous distribution. This mixture was then carefully poured into the prepared mould to ensure even distribution and minimize air bubble formation. After closing the mould, it was left at room temperature ( $30 \pm 2$  °C) undisturbed for 24 hours to allow the composite to fully cure. The manufactured benzoylated darbha fiber epoxy composite shown in (Fig. 2).



**Fig. 2** Surface appearance of composite made from ground benzoylated DP stems

### 2.3 Chemical, Physical, and Thermal Characterisation of Darbha Fibres

The chemical composition of Darbha fibres, whether treated (BDE) or untreated (UDE), is determined with a precision of  $0.001 \text{ g.cc}^{-1}$  using a pycnometer. Quantification of cellulose is performed according to Kushner and Hoffer methods [25], while lignin and hemicellulose are analysed using Klason [26] and NFT-120-008 methods [26], respectively. Additionally, wax content is evaluated using the Conrad method [27]. The functional groups of Darbha fibres are analysed by Fourier-transform infrared spectroscopy (FT-IR) using a Perkin Elmer spectrometer (Model: Spectrum 2), to understand the impact of benzoylation treatment. Measurements are conducted in the wavenumber range of  $4000$  to  $400 \text{ cm}^{-1}$  in transmittance mode, with a resolution of  $4 \text{ cm}^{-1}$ . Thermal stability evaluation of BDE and UDE samples is conducted by thermogravimetric analysis. Approximately  $6 \text{ mg}$  of sample is placed in an alumina crucible and heated from  $30$  °C to  $600$  °C in a furnace under a nitrogen atmosphere, at a heating rate of  $10$  °C/min and a nitrogen flow rate of  $20 \text{ ml.min}^{-1}$ .

### 2.4 Characterisation of Epoxy Composites Based on Darbha Fibres

The mechanical properties of the composites were evaluated through several ASTM standard tests. A tensile test was conducted on a universal mechanical testing machine at a crosshead speed of  $10 \text{ mm.min}^{-1}$ , using dumbbell-shaped samples cut from the composite plates, with a gauge length of  $30 \text{ mm}$ . Similarly, the flexural test was performed on the same machine using samples with dimensions of  $127 \text{ mm} \times 12.7 \text{ mm} \times 5 \text{ mm}$ , a span length of  $30 \text{ mm}$ , and at a speed of  $10 \text{ mm.min}^{-1}$ , following well-established practices in the specialized literature [28]. The Izod impact test, performed according to ASTM 256 [29], assessed the impact strength of unnotched specimens with dimensions of  $122 \text{ mm} \times 13 \text{ mm} \times 5 \text{ mm}$ . Finally, the Shore D hardness of Darbha fibres was measured using a Shore durometer to evaluate the penetration resistance of the composites, following similar procedures recently described in the literature. Each type of test was conducted on five samples of each type (BDE and UDE), ensuring a statistically significant representation of the mechanical properties of the studied materials.

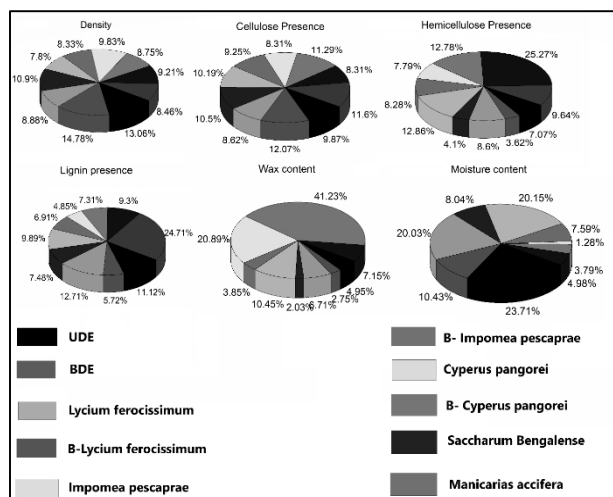
The surface morphology of fractured BDE and UDE samples is visualised using a Zeiss scanning electron microscope (SEM) (SIGMA model), Bruker. This analysis is crucial in this study to examine the microstructural characteristics of the composites after mechanical failure.

## 3 Results and Discussion

### 3.1 Chemical and Physical Properties of Fibres

The density of untreated Darbha fibre (UDE) is  $1652 \text{ kg.m}^{-3}$ , a value close to that of pineapple fibre ( $1526 \text{ kg.m}^{-3}$ ) [30], but higher than that of lignocellulosic fibres like *Acalypha indica* L ( $1356 \text{ kg.m}^{-3}$ ) [16] and *Rhecktophyllum camerunense* ( $757 \text{ kg.m}^{-3}$ ) [2]. This relatively high density suggests a dense internal structure, potentially advantageous for certain composite applications where higher density is desirable to improve rigidity and strength. In contrast, benzoylated Darbha fibre (BDE) has a density of  $1869 \text{ kg.m}^{-3}$ . This substantial increase in density compared to untreated fibre (UDE) can be attributed to the introduction of benzoyl groups that occupy void spaces and increase fibre compactness. This high density also exceeds that of other benzoylated natural fibres such as *Manicaria saccifera* [31], *Lycium ferrocisum* [21], *Ipomoea pes-caprae* [15], *Saccharum bengalense* [32], *Ficus benghalensis* [33] and banana [34]. This increase indicates that benzoylation reinforces the fibre by reducing porosity and increasing internal cohesion, which could enhance mechanical performance, particularly in terms of tensile and flexural strength [14].

Chemical analysis revealed that untreated Darbha fibre (UDE) contains 9.87% cellulose, 7.07% hemicellulose, 11.12% lignin, and a moisture content of 23.7%. The mass percentage of cellulose, comparable to that of untreated fibres from *Ipomoea pes-caprae*, *Cyperus pangorei*, and *Manicaria saccifera*, suggests a typical cellulose composition in natural fibres, which can impart moderate rigidity and strength [15,35]. However, for treated fibre (BDE), the cellulose content increases to 12.07%, while hemicellulose and lignin contents decrease to 3.62% and 5.72%, respectively.

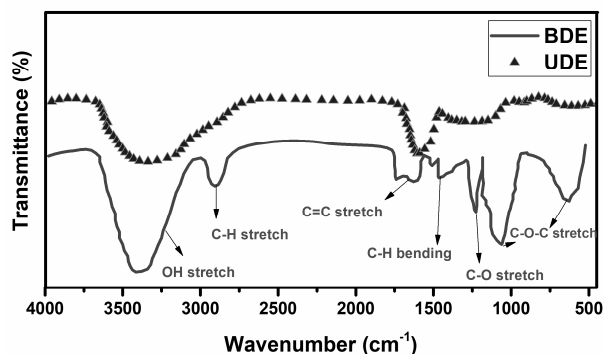


**Fig. 3** Pie Chart of the Physicochemical Properties of UDE and BDE Compared to Other Benzoylation Studies

These changes indicate that benzoylation treatment is effective in removing amorphous and low molecular weight constituents like hemicellulose and lignin, which can reduce fibre strength and dimensional stability [13,15]. This chemical modification enhances the proportion of crystalline cellulose in the fibre [13], which is crucial as higher crystalline cellulose content is generally correlated with better rigidity and tensile strength [16,30,36]. Furthermore, reducing the wax content to as low as 0.5% by mass in treated fibre promotes better adhesion between the fibre and polymer matrix, thereby improving composite cohesion and overall strength. These chemical and physical modifications potentially increase the fibre's ability to withstand high mechanical loads, making benzoylated Darbha fibre composites (BDE) particularly suitable for applications requiring superior mechanical properties. The pie chart in Figure 3 compares the physicochemical properties of benzoylated and untreated *Demostachya bipinnata* fibres with those of other benzoylated natural fibres used as reinforcements. It clearly demonstrates that BDE offers substantial advantages. BDE is an ideal candidate for polymer composite manufacturing, offering significant improvements in density and mechanical strength, which is promising for various industrial applications.

### 3.1.1 Functional Group Modifications

A comprehensive study of the surface chemistry of Darbha fibres, whether benzoylated or untreated, was conducted using Fourier-transform infrared spectroscopy (FTIR). The FTIR spectrum, shown in Figure 4, distinctly reveals various characteristic peaks of functional groups commonly found in lignocellulosic materials, including O-H, C=C, C-O-C, C-H, and C-O.



**Fig. 4** FTIR Spectrum of Untreated and Benzoylated Darbha Fibres

Firstly, the band between 500 and 1050  $\text{cm}^{-1}$  corresponds to C-O-C epoxy stretching. Additionally, peaks observed at 3400  $\text{cm}^{-1}$  and 1645  $\text{cm}^{-1}$  are characteristic of hydroxyl groups. Moreover, peaks at 2800  $\text{cm}^{-1}$  and 1520  $\text{cm}^{-1}$  indicate C-H stretching and bending, respectively, while the peak at 1745  $\text{cm}^{-1}$  signifies the presence of hemicellulose. Furthermore, the peak at 1230  $\text{cm}^{-1}$  is attributed to C-O stretching.

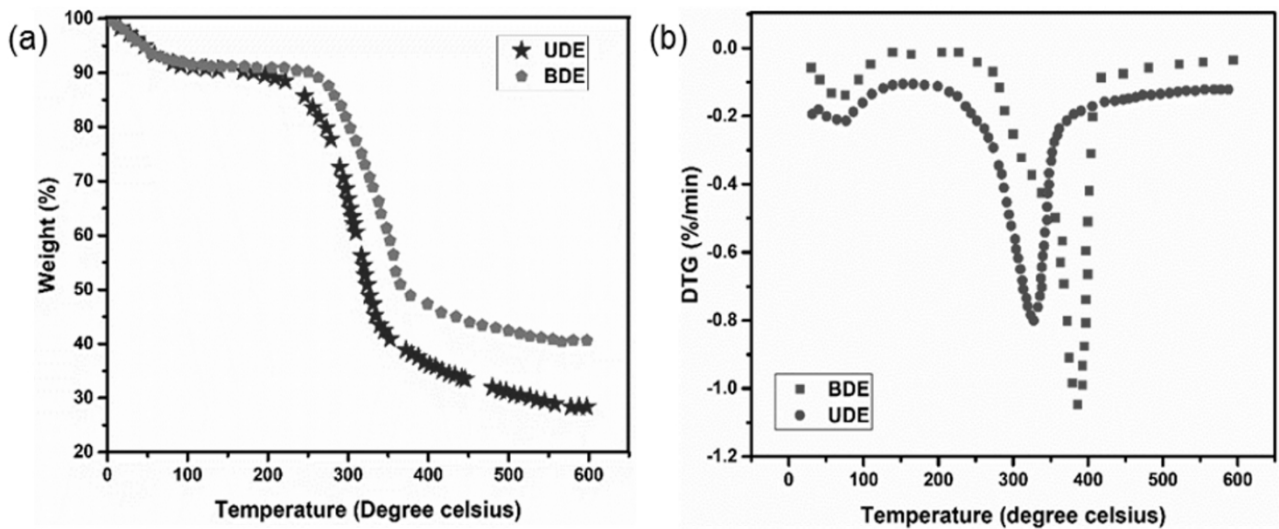
Benzoylation plays a crucial role in promoting the formation of additional carbonyl and carboxyl groups through the introduction of benzene rings on the fibre surface. This chemical modification generally leads to a significant improvement in fibre surface wettability and roughness, as supported by previous research [18]. A detailed comparison between treated and untreated fibres highlights significant differences in surface chemical composition and mechanical properties. Benzoylated fibres may exhibit enhanced interaction with polymer matrices and composite durability by reducing susceptibility to moisture and thermal degradation [13], which is crucial for the development of high-performance composites.

### 3.1.2 Thermal Degradation Properties of Fibres

Thermogravimetric analysis (TGA) assists in characterizing the thermal decomposition of Darbha fibres, whether treated or untreated, as illustrated in Figure 5a. Initially, a slight weight loss due to moisture release is observed between 40°C and 149°C, amounting to 10.4% for UDE and 9% for BDE. The second decomposition phase, between 150°C and 280°C, primarily involves the degradation of hemicellulose and cellulose, with weight losses of 11.3% for UDE and 3.1% for BDE. Subsequently, the third stage

(280°C-400°C) marks the complete degradation of hemicellulose and cellulose, with respective weight losses of 42.1% for UDE and 40.6% for BDE. The final stages of degradation, between 400°C and 650°C,

mainly involve the loss of pectin, wax, and other impurities, resulting in weight losses of 6% and 1.8% for UDE, and 5% and 1.7% for BDE.



**Fig. 5** Thermogravimetric (TGA) (a) and Derivative Thermogravimetric (DTG) (b) Analysis of Untreated and Benzoylated Darbha Fibers

The results from derivative thermogravimetric analysis (DTG), as presented in Figure 5b, confirm that the maximum degradation temperatures are 328°C for UDE and 388°C for BDE. These observations are consistent with the TGA data, demonstrating that BDE exhibits better thermal stability than UDE. This conclusion reinforces the effectiveness of benzoylation treatment of Darbha fibers, significantly reducing their thermal degradation and making them more resistant, even at high temperatures.

### 3.2 Properties of epoxy-based composites reinforced with Darbha Fibers

#### 3.2.1 Tensile Properties

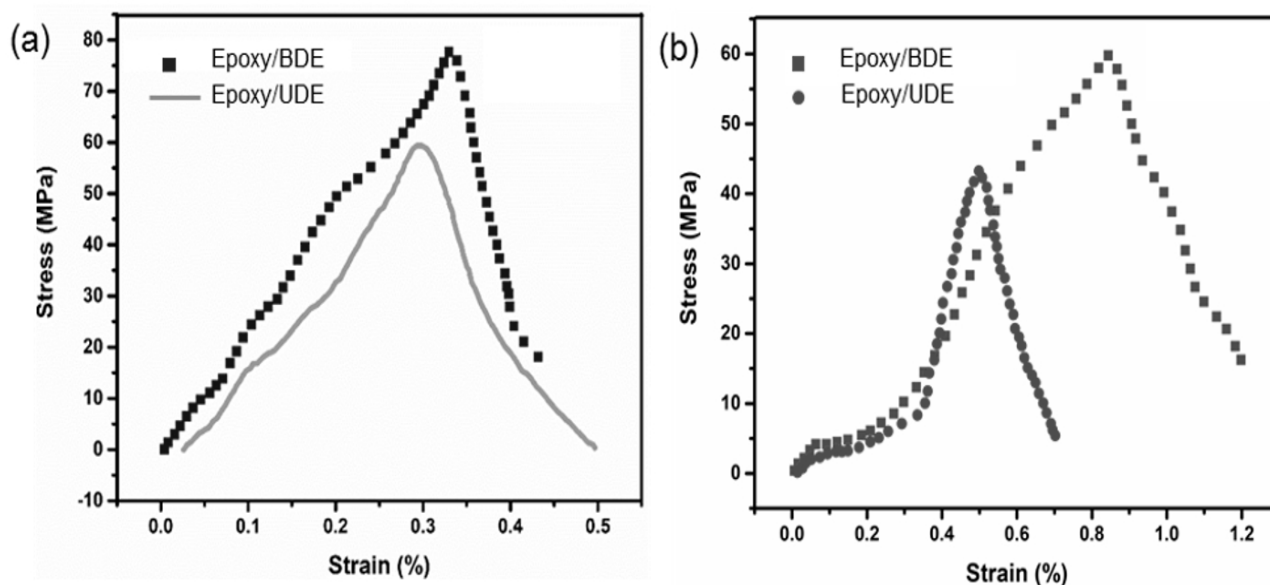
Figure 6a illustrates the stress-strain behavior for epoxy composites reinforced with untreated and benzoylated Darbha fibers. It is notable that the average stress-strain curve for composites containing benzoylated fibers (BDE) is above that of composites with untreated fibers (UDE). This corroborates previous findings suggesting that benzoylation treatment enhances fiber strength. The average tensile strength, determined at the peak of each curve, reveals that epoxy/BDE composites exhibit a tensile strength that is 30% higher compared to epoxy/UDE composites, which is around 59 MPa. These results are consistent with the findings of Vijay et al. [15], who observed similar improvements in epoxy composites reinforced with benzoylated fibers of *Impomea pes-caprae*. This significant improvement can be attributed to several factors. Firstly, fibrillation of fibers and removal of lignin and hemicellulose reduce fiber thickness and diameter, thereby enhancing mechanical performance

[7,21,37]. Secondly, benzoylation increases specific surface area and aspect ratio, creating a rough surface with numerous crevices. These characteristics promote better mechanical interlocking and improve interfacial adhesion between fibers and the matrix [8,13,38,39]. These observations underscore the importance of chemical treatments, such as benzoylation, in optimizing the mechanical properties of natural fiber-based composites. In conclusion, benzoylated Darbha fibers enable composites with significantly improved tensile strength, validating the effectiveness of this treatment for reinforcing composite materials.

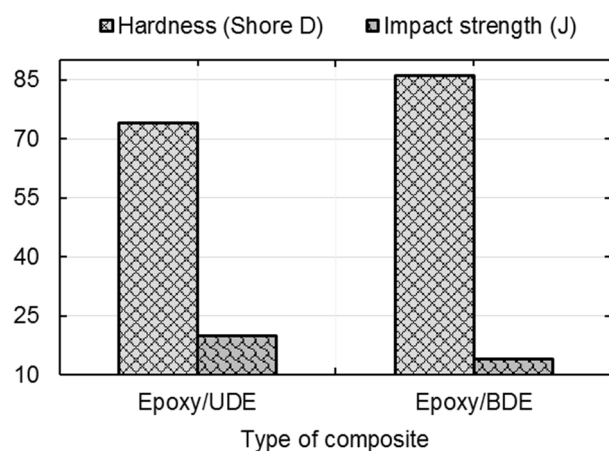
#### 3.2.2 Flexural Properties

The flexural strength of a fiber is determined by its ability to withstand bending forces applied perpendicular to its longitudinal axis [40]. The results of the bending tests in this study are illustrated in Figure 6b in terms of average nominal stress-strain curves. They reveal that benzoylation treatment of Darbha fibers significantly enhances the mechanical properties of epoxy composites. The flexural strength of composites reinforced with treated fibers is 59.7 MPa, compared to 43.3 MPa for those reinforced with untreated fibers, representing an increase of 37.8%. This improvement is attributed to fiber fibrillation, removal of lignin and hemicellulose, and introduction of benzene groups [8,12,40], increasing the specific surface area and roughness [13] of fibers for better interfacial adhesion with the epoxy matrix. These findings, consistent with the literature [13,15,40], demonstrate that treatments with benzyl chloride can make natural

fibers more competitive against synthetic fibers, while maintaining environmental advantages. This opens possibilities for the use of Darbha fibers in applications requiring high mechanical performance, such as in automotive industries and construction.



**Fig. 6** Stress-strain curves in tensile (a) and three-point bending (b) of epoxy composites reinforced with untreated (UDE) and benzoylated Darbha fibers (BDE)



**Fig. 7** Shore D surface hardness (a) and impact resistance (b) of epoxy/ UDE and epoxy/ BDE composites

The Shore D hardness of composites reinforced with treated fibers reaches 86, compared to 74 for composites reinforced with raw fibers. This approximately 16% increase in hardness indicates that benzoilation treatment enhances the rigidity and surface penetration resistance of the composite. Structural modifications such as fiber fibrillation and removal of lignin and hemicellulose increase cross-link density within the epoxy matrix [12,41,42], thereby reinforcing the composite hardness.

In contrast, the impact resistance of treated composites decreases significantly, from 20 J for raw composites to 14 J for treated ones. This 30% reduction

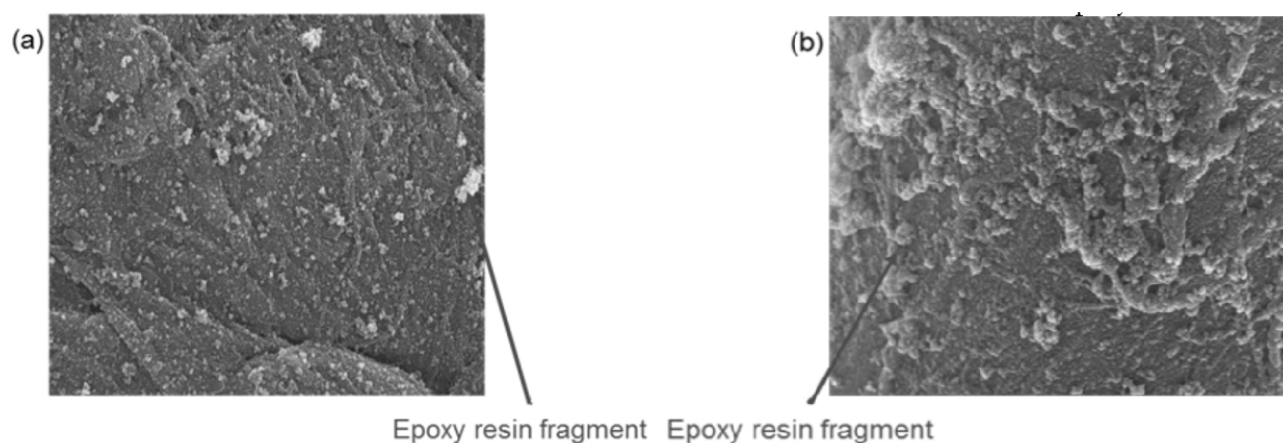
### 3.2.3 Impact and Surface Hardness Properties

Figure 7 shows a significant difference in impact resistance and surface hardness between epoxy composites reinforced with raw Darbha fibers and those treated with benzyl chloride.

may be attributed to the increased stiffness and compactness of treated fibers, which limits their ability to dissipate energy during impacts. A stiffer matrix can embrittle the fibers, thereby reducing the overall toughness of the composite [29,43]. These findings are consistent with literature where highly cross-linked matrices are often associated with increased brittleness.

### 3.2.4 Fiber topography of tensile fracture surfaces

Figure 8 illustrates the surface of untreated Darbha fibers (UDE) and benzoylated fibers (BDE) after tensile testing, taken from the fracture surfaces. Scanning Electron Microscopy (SEM) analysis reveals that the surface of UDE fibers shows fewer fragments of polymerized epoxy resin compared to BDE fibers. This observation indicates moderate interfacial adhesion between UDE fibers and the epoxy matrix, whereas the BDE fiber-matrix interface demonstrates significantly stronger adhesion. This difference in interfacial adhesion is crucial in explaining the significant increase in mechanical properties observed in this study. The strong interfacial adhesion between BDE fibers and the epoxy matrix contributes to better stress distribution, thereby improving flexural strength, compression resistance, and penetration resistance. Indeed, BDE fibers treated with benzyl chloride exhibit a rougher surface and modified surface chemistry that promote enhanced interaction with the polymer matrix.



**Fig. 8** SEM images of fracture surfaces of untreated Darbha fibers (a) and treated fibers (b) after tensile testing.

Therefore, it is crucial to establish the relationship between surface hardness, impact resistance, flexural strength, and tensile strength. Improved surface hardness is often accompanied by better flexural strength, as observed with treated Darbha fibers showing higher flexural resistance. However, this increased stiffness can reduce impact resistance, as evidenced by the decrease in impact strength. Similarly, enhanced interfacial adhesion and a rougher surface promote higher tensile strength, thereby improving overall composite performance. These complex interactions underscore the importance of optimizing surface treatments to balance hardness, flexibility, and mechanical strength of composites.

#### 4 Conclusion

This study has demonstrated the effectiveness of benzoylation treatment on Darbha fibers (*Demostachya bipinnata*) to enhance their mechanical and physical properties, as well as the performance of epoxy composites reinforced with these fibers. Compared to untreated fibers, treated fibers show a significant increase in density, from  $1652 \text{ kg}\cdot\text{m}^{-3}$  to  $1869 \text{ kg}\cdot\text{m}^{-3}$ , and a change in chemical composition, with higher cellulose content and reduced lignin and hemicellulose content. These chemical and physical modifications induced by benzoylation treatment enhance the fibers' ability to withstand high mechanical loads, making benzoylated Darbha fiber composites particularly suitable for applications requiring superior mechanical properties. FTIR analysis revealed significant changes in surface functional groups of treated fibers, with additional carbonyl and carboxyl groups introduced, improving wettability and surface roughness of the fibers. These changes promote better adhesion with polymer matrices, crucial for composite durability and performance. Additionally, thermogravimetric analysis showed that treated fibers have improved thermal stability, with a maximum degradation temperature of  $388^\circ\text{C}$  compared to  $328^\circ\text{C}$  for untreated fibers. This enhanced thermal stability is essential for

high-temperature applications. Mechanical tests of epoxy composites reinforced with Darbha fibers yielded promising results. Tensile strength of composites containing treated fibers was 30% higher than those with untreated fibers, reaching 59 MPa. Similarly, flexural strength of composites reinforced with treated fibers increased by 37.8%, reaching 59.7 MPa. However, impact resistance of treated composites decreased by 30%, likely due to increased stiffness and compactness of treated fibers, limiting their energy dissipation capability during impacts. Surface hardness of treated composites also increased by 16%, indicating improved penetration resistance. SEM analysis of fractured surfaces after tensile tests revealed stronger interfacial adhesion between treated fibers and the epoxy matrix. This enhanced adhesion is attributed to the rougher surface and chemical modification of treated fibers, promoting more uniform stress distribution. Therefore, surface treatments such as benzoylation are crucial for optimizing the mechanical properties of natural fiber composites. However, further exploration of other chemical treatments for Darbha fibers and evaluation of their performance in different polymer matrices for specific applications, particularly in automotive and construction industries, are warranted.

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