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RESEARCH ARTICLE

PINEAPPLE LEAF FIBRE/POLY (3-HYDROXYBUTYRATE-CO-3-HYDROXY VALERATE) DEGRADABLE COMPOSITE: MORPHOLOGY AND FAILURE PREDICTION OF MECHANICAL STRENGTH USING WEIBULL ANALYSIS

Zaleha Mustafa[1,*], Ain Sappa Amirah[1], Siti Hajar Sheikh Md Fadzullah[2], Siang Yee Chang[1], Nadlene Razali[2], Lin Feng Ng[3], Thanate Ratanawilai[4]

[1]*Fakulti Teknologi dan Kejuruteraan Industri dan Pembuatan, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia.*
[2]*Fakulti Teknologi dan Kejuruteraan Mekanikal, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia.*
[3]*Centre for Advanced Composite Materials (CACM), Faculty of Mechanical Engineering, Universiti Teknologi Malaysia, 81310, Johor Bahru, Malaysia.*
[4]*Department of Industrial and Manufacturing Engineering, Faculty of Engineering, Prince of Songkla University, Hat Yai, 90110 Songkhla, Thailand.*

Abstract. This study investigates the mechanical properties and failure behaviour of biodegradable composites made with pineapple leaf fibre (PALF) and poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) as the matrix. The study focuses on the effect of varying fibre loadings and their failure behaviour. The PALF was treated with a 5% NaOH solution, and the PALF/PHBV composites were prepared using solvent casting, followed by compression moulding. Flexural tests showed that increasing PALF content significantly improves both flexural strength and modulus, with the highest values observed at 40 wt.% PALF, reaching 108.36 (±8.53) MPa and 6.39 (± 0.71), respectively. Morphology analysis through scanning electron microscopy (SEM) revealed better stress transfer and fibre-matrix adhesion at higher loadings, although some fibre pull-out and debonding were observed. Statistical analysis using a two-parameter Weibull distribution demonstrates that higher fibre content enhances mechanical strength and has a lower Weibull modulus than neat PHBV. This indicates greater variability of the composite strength, possibly caused by uneven stress distribution and poor matrix/fibre interface bonding. The study concludes that optimising fibre content is crucial for maximising the mechanical performance of PALF/PHBV composites. These findings contribute to the advancement of sustainable materials, highlighting the potential of the materials in developing high-performance bio-composites where sustainability, strength, and biodegradability are important considerations, such as automotive components, construction materials, agriculture and others.

Keywords: PHBV, flexural analysis, pineapple leaf fibre, weibull distribution.

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***Corresponding author: zaleha@utem.edu.my**

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1. INTRODUCTION

The growing concerns effects of tremendous usage of non-degradable synthetic polymers on ecosystems and human health have increased the interest and focus on the development of more sustainable and biodegradable polymers. Bioplastics, often derived from renewable resources, provide a suitable alternative to traditional polymers in terms of mechanical properties but without environmental issues [1,2]. Poly (3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV), a polymer from the polyhydroxyalkanoate (PHA) family, is sourced from bacteria, making it a more sustainable option than petroleum-based polymers due to their eco-friendly properties and versatility in various applications. These biopolymers are both biodegradable and can be processed using a normal thermoplastic route. However, PHBV's mechanical properties and processability are limited due to its low thermal degradation temperature; thus, incorporating nucleating agents or blending with more thermally stable biopolymers can enhance its thermal resistance [3,4].

Natural fibre, such as pineapple leaf fibre (PALF), has excellent biodegradability, renewability, and low-weight properties. These fibres are an eco-friendly alternative to traditional synthetic fibres, making them ideal for composite materials. PALF, in particular, is a promising choice due to its high strength, low density, and abundance as a pineapple cultivation by-product [5-7]. Mansingh et al. (2022) [8] reported that PALF reinforcement improves the mechanical characteristics of PLA composites, with alkali-treated PALF at a 3 wt.% loading achieving optimal tensile and flexural strengths. Despite these benefits, there has been little research into the use of PALF in a degradable polymer matrix when compared to other commonly used lignocellulosic fibres, such as jute, sisal and others.

Recent research has demonstrated that incorporating natural fibres into PHBV can improve its mechanical properties and sustainability. However, a major challenge in natural fibre-reinforced PHBV composites is their weak interfacial bonding, which can lead to fibre pull-out and reduce the strength of the composite [9]. Thus, it is critical to understand how fibre architecture influences mechanical properties and predict their behaviour under stress [10]. The fibre-matrix interface is a vital factor contributing to the mechanical performance of the fibre-reinforced polymer composite; therefore, the variability of the failure strength of the composite is often correlated with the fibre loading. A failure prediction model, such as Weibull Analysis (WA), is a statistical method for studying material life cycles by analysing failure trends and predicting failures from sample data. Its main strength lies in handling small sample sizes and quickly providing failure predictions. It also offers a visual representation of failure data. The Weibull distribution is commonly used for predicting failure times, as its probability curve can vary in shape by adjusting the two parameters: the shape and scale parameters [11].

This study aims to investigate the influence of the PALF loadings on the flexural properties and morphology when reinforced in a PHBV matrix to provide a better understanding of the optimal conditions for maximising the performance of these biodegradable composites. Furthermore, a Weibull failure prediction model was employed to understand the failure mechanisms and predict the reliability of the developed composites under different loading conditions. The findings from this study are expected to contribute to the advancement of sustainable material technology by providing a better understanding of the role of natural fibres in composite materials and guiding the development of high-performance, environmentally friendly alternatives to traditional non-degradable composites.

2. MATERIALS AND METHODS

Poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) was used as a matrix. It contains 3% of 3-(hydroxyvalerate) (HV) molar content under the trade name Enmat Y1000P (TianAn Biopolymer, Guangdong, China). It has a density of 1.25 g/cm3 with a melt flow index (MFI) of 5.2 g/10 min and is supplied in powder form. The pineapple leaf fibre (PALF) was used as reinforcement provided by Serat Alfibre, West Java, Indonesia. It has a density of 1.52 g/cm3. Sodium hydroxide (NaOH) and chloroform (CHCl3), lab grade with 99.9 % purity, were supplied by Merck and Co.

2.1 PALF Surface Modification – Alkali Treatment

The PALF were alkaline treated with a 5% NaOH aqueous solution (w/v) for an hour at room temperature and analysed with Fourier Transform Infrared Spectroscopy (FTIR) to identify the different functional groups present as described in detail by Ramli et al. (2017) [12].

2.2 Composite Production

All the PALF used in this study were alkaline and treated with 5% NaOH aqueous solution (w/v) for an hour at room temperature, as described previously [12]. The alkaline-treated PALF was cut into short fibres with a 6 mm critical length and processed into a thin film through a paper-making technique. One gram of PALF was soaked in distilled water for 30 minutes and then blended using a high-speed blender to enhance fibre fibrillation. The resulting mixture was poured into a sieve frame of 300 mm × 200 mm to form a thin layer of PALF film. The film was pressed in a 0.5 mm thick steel window frame to remove the access water, followed by being sun-dried for 3 hours and in a drying oven at 60 ℃ overnight to ensure complete water removal.

The PHBV powder was dissolved in chloroform to form 5 wt.% polymer concentration (w/v) using continuous stirring on a magnetic stirrer at 50 °C until the solution turned from white to yellowish-clear, indicating complete dissolution. The PHBV solution was poured into a glass container serving as a matrix bath. The PALF film was dipped in the matrix bath for approximately 10 seconds each to coat the surface with PHBV solution to form a thin pre-preg and dried overnight at room temperature. Afterwards, it was vacuum-dried at 60 °C for 24 hours to ensure all the solvent was removed. The weight of each PALF/PHBV pre-preg was measured before and after the coating process to determine the matrix content in each film produced. The dried pre-preg and neat PHBV film were stored in a desiccator until fabrication.

The PALF/ PHBV pre-preg and neat PHBV laminates were hand lay-up in a 200 mm × 100 mm × 3 mm closed mould in sandwich stacking order as shown in Figure 1. The composite was then compressed moulded at 175 °C using an optimised processing method as described in the previous study [13]. Each of the pre-preg was weighted and neat PHBV was added accordingly to produce composites with 10, 20, 30, and 40 wt.% PALF.

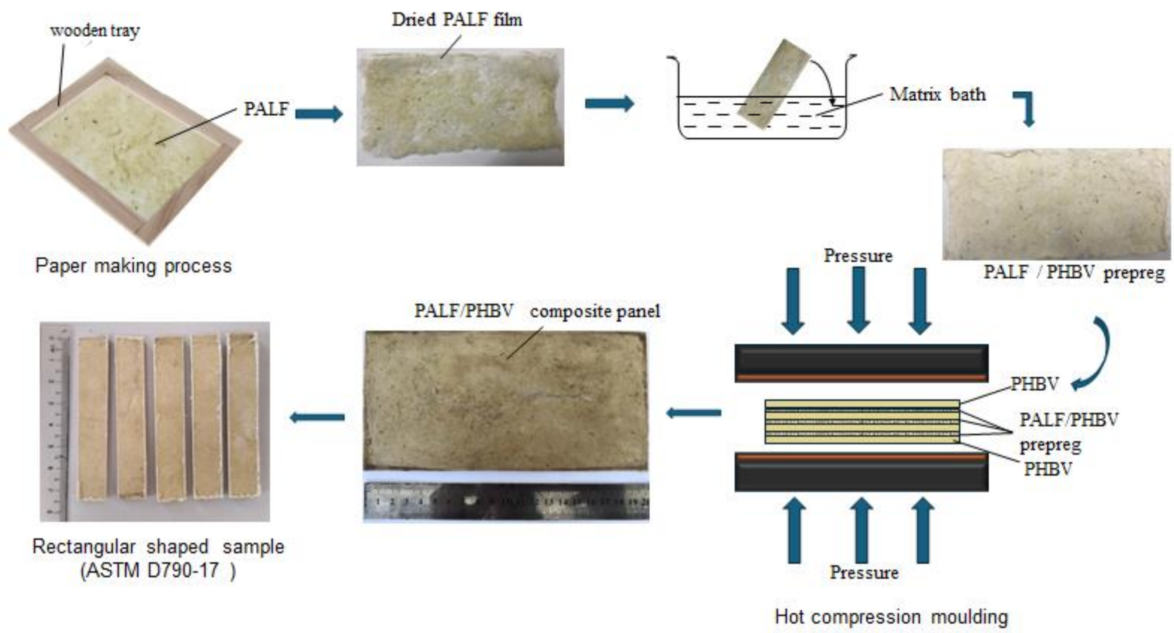

Figure 1: Preparation and fabrication process of PALF/PHBV composites

2.2 Flexural Tests

Three-point static flexural tests were conducted according to ASTM D790-17 standard. Specimens were cut into 72 mm in length, 12.7 mm in width and 3 mm in depth. The load was applied at the midpoint between supports, with a span of 54 mm. The crosshead speed was set to 1.28 mm/min. The tested specimens were performed using an AGS-X Shimadzu Universal Testing Machine equipped with a 20 kN cell load. For each condition, five specimens were tested, and their average and standard deviations were calculated. Each specimen was loaded until it reached 5% of the strain value.

2.2 Statistical Analysis – Two-parameter Weibull Statistical Analysis

In this study, a two-parameter Weibull statistical analysis was used to evaluate the variability in the flexural strength of PALF/PHBV composites at various fibre loadings. The two-parameter Weibull distribution expression for cumulative probability density is shown in Equation 1 [14,15].

$$P = 1 - \exp\left(-\frac{\sigma}{\eta}\right)^{\beta} \tag{1}$$

Where P denotes the probability of failure at flexural stress, σ, while η is the scale parameter (Weibull strength), and β is the shape parameter (Weibull modulus). This can be written in a linear relationship as in Equation 2:

$$\ln\left[\ln\frac{1}{1-P}\right] = \beta\ \ln(\sigma) - \beta\ln(\eta) \tag{2}$$

The cumulative probability density is expressed using the median rank formula [15] as given by Equation 3:

$$\mathrm{P} = \frac{i\text{-}0.3}{n + 0.4} \tag{3}$$

Here, i and n represent the current test number and the total number of tests in each set, respectively.

3. RESULTS AND DISCUSSION

3.1 Chemical Characterisation of the PALF

Figure 2 shows the FTIR spectra of both untreated and alkaline-treated PALF. Untreated PALF is naturally hydrophilic and contains pectin, hemicellulose, lignin, and waxes, which can hinder good bonding with hydrophobic PHBV. In the untreated PALF, vibration peaks at 1229 cm^{-1} and 1436 cm^{-1} indicate the presence of lignin and hemicellulose, respectively [12]. The peak at 1724 cm^{-1} corresponds to the C=O stretching of carbonyl groups in hemicellulose, pectin, or wax [16]. After alkaline treatment, it was observed that the peaks at 1229 cm^{-1} and 1650 cm^{-1} had disappeared, possibly due to the effective removal of the lignin and reduction of hemicellulose by the NaOH. At the same time, it was observed that a decrease in peak intensity at 1436 cm^{-1} in alkaline-treated PALF also indicates the reduction of hemicellulose [16]. The vibration peak of carboxylic acids in ester groups at 1724 cm^{-1} was also absent. It can be concluded that the alkaline treatment used in this study has effectively reduced the hemicellulose, pectin, or wax content in the PALF. This finding is similar to those reported by Ismail et al. (2021) [17].

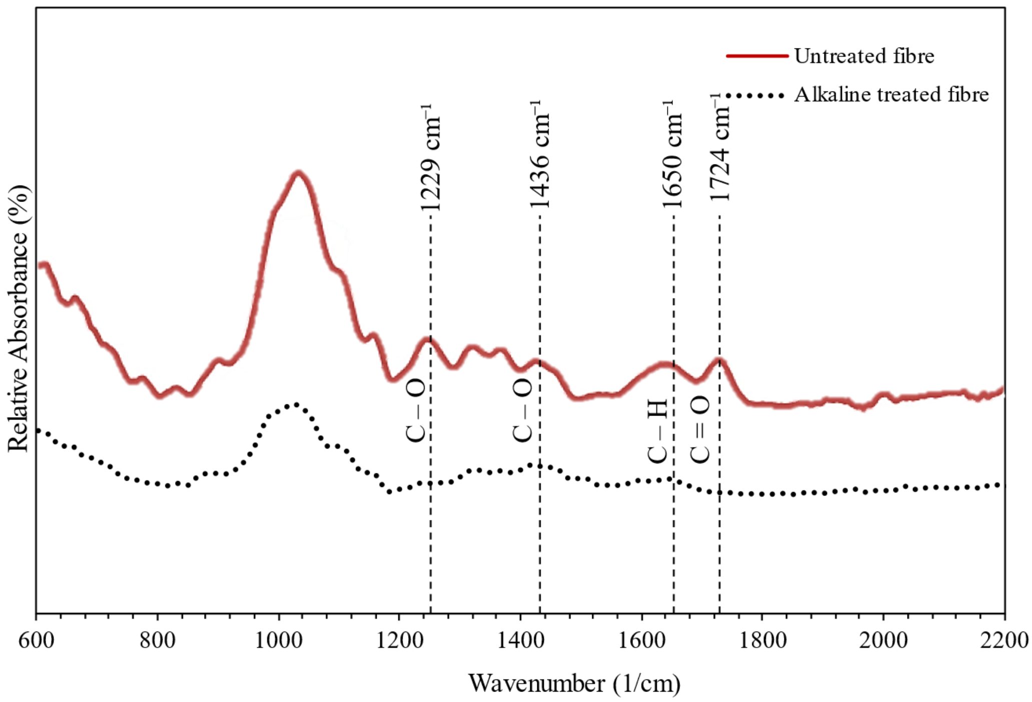

Figure 2: ATR-FTIR spectra of unmodified PALF and PALF with alkaline treatment

3.2 Mechanical Properties of the Composites

Figure 3 shows the effect of the PALF loadings on the ultimate flexural strength and flexural modulus of the PHBV-based composite. The initial flexural strength and modulus of neat PHBV are 92.13 (± 4.70) MPa and 6.83 (± 0.81) GPa, respectively. The introduction of 10 wt.% PALF reduces these values to 59.18 (± 7.62) MPa. The load-sharing capacity in the composite often relies on the interfacial bonding between the fibre and matrix [18]. Thus, this reduction may result from poor interface bonding, as indicated by morphology analysis in Section 3.3. Simultaneously, the presence of voids in the composite can create areas of stress concentration that lead to crack formation and weaken the composite [19]. With 20 wt.% PALF loading, the ultimate flexural strength improves to 82.59 (± 8.32) MPa, suggesting that the fibres begin to positively contribute to the composite's strength, likely due to better stress transfer between the fibres and the matrix. Although the strength of the composite shows slight improvement compared to the composite with 10 wt.% PALF loading, the fibre loading is still insufficient to create a robust structure and effectively distribute the applied force, thus limiting the enhancement in flexural strength compared to neat PHBV. This observation is often seen in composites with polymer matrices, where the reinforcement alone is not sufficient to overcome the inherent brittleness of PHBV [20]. With increases in PALF loadings to 30 wt.% and 40 wt.%, the ultimate flexural strength of the composite further rises to 86.04 (± 15.90) and 108.36 (± 8.53) MPa, respectively, indicating a more effective load-bearing role of the fibres as the fibre-matrix interaction is enhanced. This finding suggests that the fibre content at these levels optimises the reinforcement effect, likely due to improved stress distribution and better fibre-matrix adhesion [21].

The flexural modulus of PHBV/PALF composites also shows a similar trend. The neat PHBV exhibits a flexural modulus of 6.83 (± 0.84) GPa. At 10 wt.% fibre loading, the flexural modulus decreases to 3.63 (± 0.62) GPa, indicating reduced stiffness, possibly due to the presence of the voids that weaken the composite. As the fibre content increases to 30 wt.% and 40 wt.%, the modulus improves and reaches around 4.84 (± 0.23) GPa and 6.39 (± 0.71) GPa, respectively. An increase in the modulus indicates enhanced stiffness of the developed bio-composites. This greater stiffness can be attributed to a better interaction between the fibres and matrix within the bio-composites, facilitating more efficient stress transfer [18].

Thus, the flexural strength analysis reveals that the mechanical properties of PALF-reinforced composites are highly dependent on interface bonding and fibre contents. While low fibre content may not significantly enhance or could even reduce the composite's strength due to the poor interface bonding and the presence of pores that cause early failure, the PALF/PHBV composite at higher fibre loading, particularly at 40 wt.%, has shown an improvement in both the ultimate flexural strength and modulus.

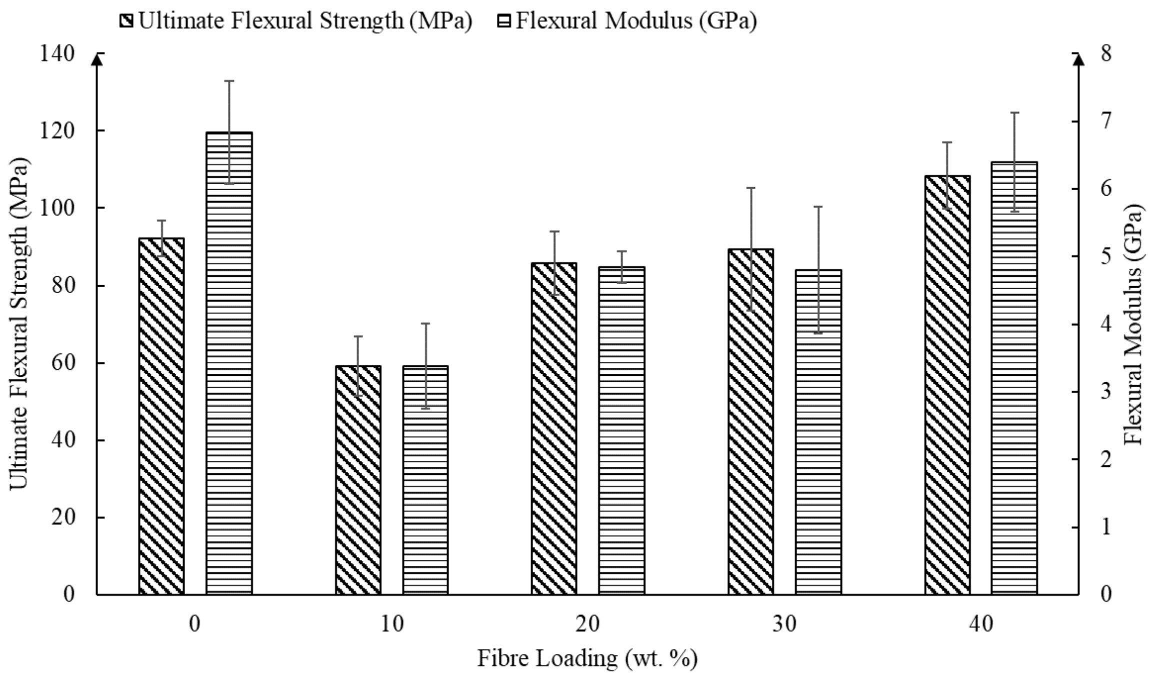

Figure 3: Flexural strength properties of PALF-reinforced PHBV composite

3.3 Composite Fracture Morphology

The fracture morphology due to the flexural stress of PHBV/PALF is represented in Figure 4, indicating that the composite fractured due to poor interfacial bonding, fibre breakage, matrix breaking, and fibre pull-out. Figure 4(a) shows the presence of gaps between the fibre and matrix and holes in the 10 wt.% PALF/PHBV composite. This observation suggests that some fibres align parallel to the applied stress direction. However, due to weak bonding with the PHBV matrix, the PALF detached, thus leaving a hole [22]. Pores could also be indicated as trapped air formed during the fabrication processing stage [23]. It was also observed that a significant portion of matrix breakage, suggesting the matrix-dominated fracture morphology, which reflects the lower flexural strength compared to the neat PHBV.

With the increase of fibre loadings, gaps between the matrix and broken fibre can still be found. However, the presence of the embedded fibre and full coverage in the matrix is more pronounced, indicating that the adhesion of the PALF and PHBV is improved (Figure 4(b)). It was also observed the presence of fibre clustering with splintered appearance and separated filaments in the 30 wt.% PALF composites (Figure 4(c)), might contribute to only small changes in their strength and modulus. At 40 wt.% PALF/PHBV, the fractured surface shows the present defibrillated fibres (Figure 4(d)), indicating that a slower crack propagation condition occurs during the load application, as more energy was used to draw the fibres out and may contribute to their better flexural properties [24].

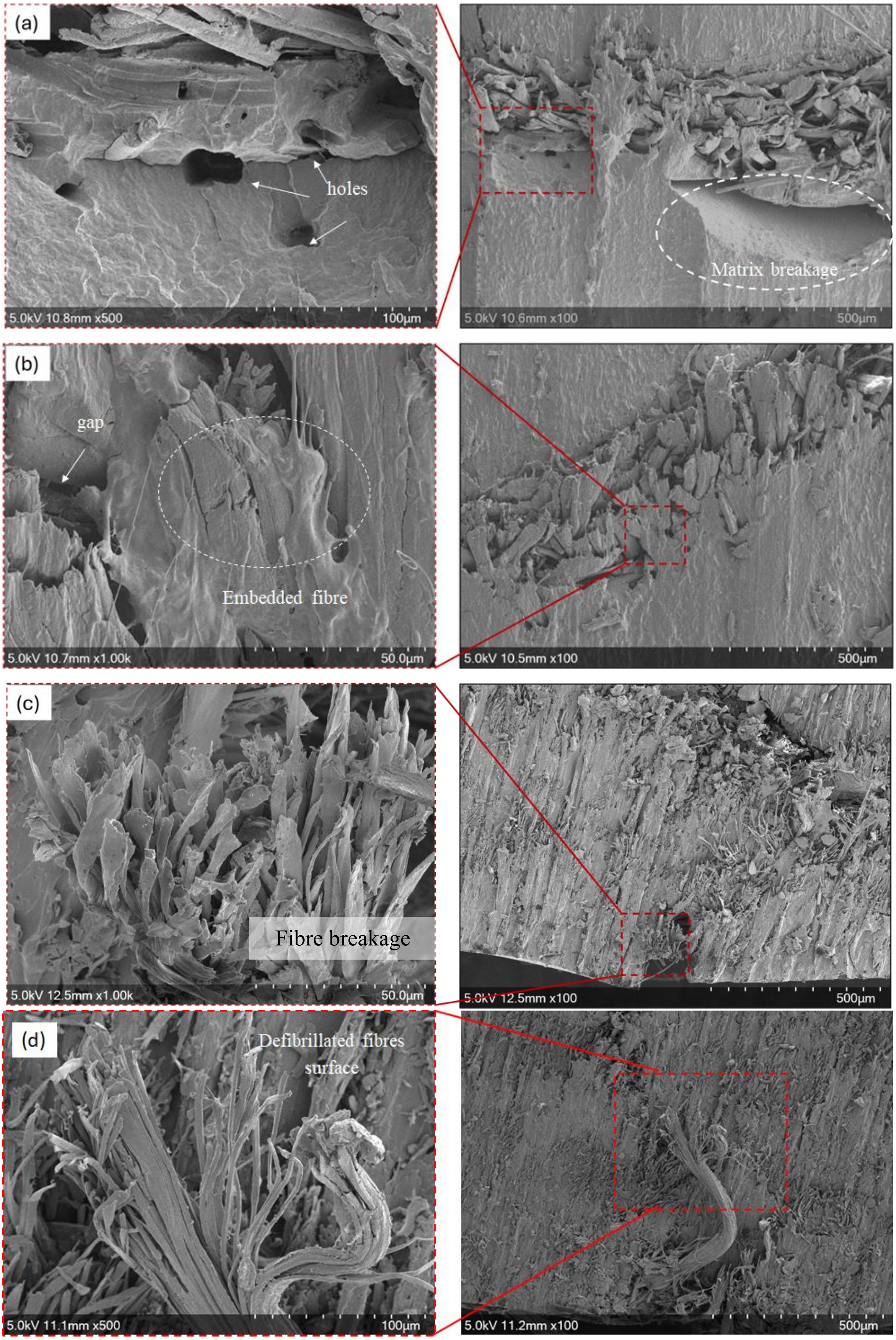

Figure 4: Flexural fracture morphology of PHBV/PALF composites at (a) 10 wt.%, (b) 20 wt.%, (c) 30 wt.% and (d) 40 wt.% fibre loading

3.4 Weibull Statistical Analysis

The Weibull plot parameters for the PALF/PHBV composite in terms of flexural strength with varying fibre loadings are shown in Figure 5. The results reveal that the composite generally displays a bimodal strength distribution, consisting of a combination of higher strength with greater variability and lower strength with less variability. The coefficient of determination (R^2) values exceed 92%, indicating the reliability of the parameter estimates. The bimodal distribution likely reflects the complex and multifaceted origins of fracture initiation and propagation leading to breakage in the composites, as reported by Suzuki [25].

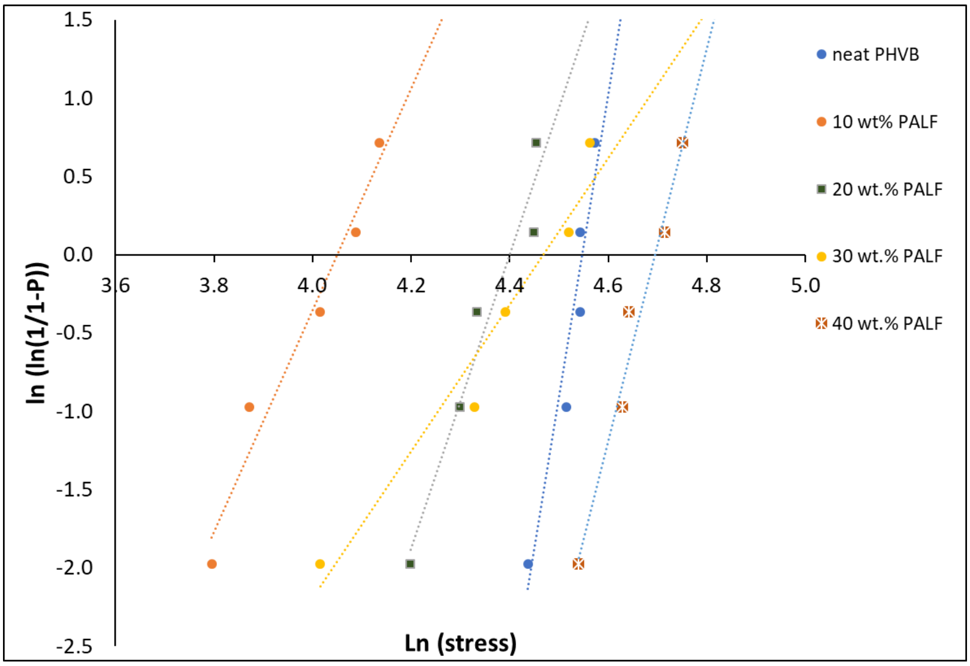

Figure 5: Two-parameter Weibull plot for flexural strength of PALF/PHBV composite at different fibre loadings

Table 1 presents the range of Weibull modulus values for all fibre loadings. A higher Weibull modulus indicates that the materials have less variability in strength, as given by the neat PHBV (β =19.3). In general, the PALF/PHBV composite has a lower Weibull modulus as compared to the neat PHBV. The relatively lower Weibull modulus values in the PALF/PHBV composite suggest variability in composite strength, likely due to the non-homogeneity of the fibres and poor interface bonding between the fibre and matrix [26]. The lowest value of Weibull modulus was observed in 30 wt.% PALF loading (β = 4.68), indicating a significant increase in the variability of the composite flexural strength. This is supported by the SEM analysis on the composite fractured surface (Figure 4(c)) shows the presence of fibre clustering, where bundles of fibres have collectively detached from the matrix. The pull-out fibres exhibit clean surfaces with minimal matrix adhesion as well as a splintered appearance and separated filaments, suggesting inadequate matrix wetting during composite fabrication. These characteristics point out that the composite failed due to poor fibre/matrix interface bonding, which might contribute to its low Weibull modulus value.

Natural fibres are inherently more variable than synthetic fibres as the defects or inconsistencies in the fibres have a more significant impact on the composite's overall performance, leading to lower reliability and more variable failure behaviour. With the increase of the fibre content, the possibility of uneven fibre distribution and variation of the matrix-fibre interaction may lead to more scattering values of the flexural strength of the PALF/PHBV composite. In this study, the short-fibre PALF film is prepared using a paper-making method. Thus, the possibility of non-uniform distribution on the surface of the PALF film during the fabrication process may occur, which leads to weak spots in the composite, making it more susceptible to failure under varying strength. To improve the Weibull modulus, controlling the processing conditions and better distribution of the fibre are needed to produce consistent material properties, leading to a more predictable failure behaviour. At the same time, it was observed in this study that the theoretical mean of the flexural strength of the PALF/PHBV composites closely matched the experimental values. The difference between the theoretical and mean experimental values is less than 5% for all fibre loadings tested, confirming that a two-parameter Weibull distribution can effectively predict the flexural strength of the composite.

Table 1: Flexural strength, shape and scale parameters for PALF/PHBV composite at different fibre loadings

PALF loading (wt.%)	Weibull Strength, η (MPa)	Weibull Modulus, β	Experimental Strength, σ (MPa)	Percentage of error (%)
Neat PHBV	94.22	19.3	92.13	2.22
10	57.62	7.07	59.18	2.71
20	81.38	9.37	82.59	1.48
30	87.12	4.68	86.04	1.24
40	109.27	12.51	108.40	0.84

4. CONCLUSIONS

The study demonstrates that incorporating pineapple leaf fibre (PALF) into the PHBV matrix significantly impacts the flexural strength and modulus of the resulting composites. Increasing the fibre content generally improves flexural strength, with the highest strength observed at 40 wt.% fibre loading. This suggests that higher PALF fibre content effectively improves the overall mechanical strength of the composites. Weibull analysis reveals that the strength increase is accompanied by greater variability in the mechanical properties. The low Weibull modulus in the PALF/PHBV composites indicates considerable scatter in the flexural strength data. While higher fibre loading enhances strength, it may also lead to inconsistencies due to factors such as fibre agglomeration and uneven stress distribution. This variability is particularly evident in composites with 30 wt.% fibre content, where the lower Weibull modulus values highlight substantial strength scatter, likely caused by poor fibre-matrix bonding or the presence of micro-voids. In conclusion, the optimal performance of PALF/PHBV composites is achieved at higher fibre loadings, where significant strength improvements are noted. However, it is crucial to address the variability in mechanical properties, particularly at both lower and higher fibre contents. Future research should focus on optimising fibre treatment and dispersion techniques to minimise variability and enhance the uniformity of the composites' mechanical properties, ensuring their effective use in a broad range of applications.

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Author Contributions

All authors contributed toward data analysis, editing the manuscript and agreed to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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