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Effect of Fibre Loadings on the Tensile Properties of PALF/PHBV Composite: A Two- Parameter Weibull Analysis

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Abstract

Interest in biodegradable and sustainable is steadily rising which has made the investigation into natural fibre reinforced biopolymers. Among these, poly(3-hyroxybutyrate-co-3-hydroxyvalerate) (PHBV) stands out for its biodegradability and biocompatibility, but its inherent brittle and has few or no properties conducive to application to wider engineering fields. Additionally. The natural variability of fibres can lead to inconsistent mechanical performance, yet only a few studies have examined this variability from a statistical perspective. In this study, the influence of adding pineapple leaf fibres (PALF) at different loading (10-40 wt.%) was investigated on the mechanical behaviour of PHBV. The tensile strength was analysed using a linearized two-parameter Weibull method to determine the scale (η) and shape (β) parameters, providing insight into both the typical strength and the variability of the composite's performance. The results show that tensile strength increases with fibre loading, with highest of 46.54 MPa (±4.12) at 30 wt.% PALF, corresponding to a 73% rise in scale parameter compare to other loadings. A strong correlation was established between the experimental data and the Weibull model, with ($R^2 > 0.9$) and the differences are below 1 % and 7 % for neat PHBV and PALF/PHBV respectively. Scanning electron microscopy (SEM) analysis revealed signs of fibre rupture and fibre extraction, indicating that the composite's failure was a result of poor interfacial bonding between the matrix and fibre. The Weibull analysis able to provide more comprehensive evaluation of the composite mechanical reliability as it taken account the statistical distribution of all measured data rather than just the means value. The two-parameter Weibull model demonstrated that the composite containing 30 wt.% PALF attained the maximum tensile strength with satisfactory reliability. This finding signifies an ideal equilibrium between performance and variability, essential for the dependable design of sustainable PHBV-based composites.

***Keywords**: tensile strength; PALF; fibre loadings; Weibull distribution; PHBV polymer*

1. Introduction

Growing concerns on the impact of the consumer products on the environment are shifting interest to use more sustainable and environmentally friendly source. Materials such as natural fibre reinforced composites and degradable polymer offer potential solution because of their ability to decompose and their mechanical properties ALF, an agricultural by product presents significant potential as a degradable reinforcement characterized by outstanding mechanical properties and renewability. [1]. This is attributed to its high cellulose content and low-density attribute. When PALF used in degradable polymer matrix system, such as polylactic acid (PLA), poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV), poly (butylene succinate) (PBS), it is capable to enhance the mechanical performance of the resulting composite as along with retaining the degradability properties [2]. Hazwani et al. [3] indicated that incorporating PALF into the epoxy resin matrix significantly enhances the tensile properties of the composite. The performance of composites based on PALF can be further improved when combined with synthetic fibres like Kevlar [1] suggesting that PALF is excellent reinforcement option when both mechanical strength and suitability are essential.

PHBV is a thermoplastic polymer synthesized by microorganisms. It demonstrates superior biodegradability, but possesses only moderate mechanical strength. PHBV is widely used in packaging, agricultural and biomedical applications because of its renewable origin and environmental compatibility. However, one of the shortcomings of the PHBV is its inherent brittleness [4]. Therefore, in order to enhance its mechanical performance, PHBV can be bounded with more ductile polymers or combined with a natural fibre such as PALF, bast, kenaf, diss and other natural fibres exhibit a significant enhancement in mechanical properties [5-8]. Although there maybe challenges in combining a hydrophilic fibre with the PHBV polymer matrix, the overall improvement in mechanical performance of the resulting composite would depend on variables such as fibre contents, dispersion and interfacial bonding [9]. Poor interfacial bonding between natural fibre and PHBV polymer is due to their polarity mismatch. Natural fibre such as PALF are hydrophilic in nature, while PHBV is a more hydrophobic material. Therefore, it prevents good compatibility during fabrication, resulting in poor bonding at the matrix and fibre interface and may weaken the resulting composite produced [10]. At the same, PHBV is quite sensitive to thermal degradation provided only a small processing window and prevent thorough blending or melt mixing during fabrication. This will reduce the efficiency of the fibre-matrix interaction and consequently the mechanical performance [11]. Thus, usage of proper surface treatment is crucial in order to ensure better compatibility and stronger bonding between matrix and fibre [10]. To enhance material polarity and eliminate surface impurities, this study used an alkaline surface treatment. All PALF was treated with a 5 wt.% sodium hydroxide (NaOH) aqueous solution, specifically Prior to composite fabrication.

Composites materials made from natural fibre, such as PALF which have higher variability in their properties compare to synthetic materials due to their inherent characteristics. As a result, it is critical for the researchers and designer to properly understand and quantify this variability in order to ensure their reliability in practical applications. Statistical method, such as Weibull analysis, are commonly used in reliability engineering to analysis the failure probability and variability material properties. The two-parameter Weibull distribution can provide valuable information into the statistical distribution of material properties [12]. Therefore, this enables researchers to estimate the failure probability and consistency of the materials performance under varying conditions or stress loads [13-14]. Djeghader et al. [15] utilized the Weibull analysis to evaluate the reliability of jute/polyester composites. On the other hand, Aziz et al. [16] applied the same approach to evaluate the performance of UHMWPE composite laminates. However, the application of Weibull reliability analysis to PALF- based composite systems remains limited. Most of the studies highlighted the surface treatment effect, fibre loadings, or processing conditions on average mechanical properties with few addressing the statistical reliability and variability under loading utilizing probabilistic models like the Weibull distribution.

Thus, the objective of the present work is to evaluate the mechanical performance of the PALF/ PHBV composite by varying its fibre loading from 10 to 40 wt.% and to predict the influence of fibre loading on the Weibull distribution. The experimental tensile strength data were analysed using a two-parameter Weibull distribution to assess the reliability and predict the composite mechanical performance. The theoretical values were calculated by linear curve fitting of the Weibull model and the correlation between the estimated and experimental results was examined. The outcome of this study is expected to provide insight into the contribution of natural fibres in bio composite in general, aiding in the prediction of their variability and providing a better reliability model for their properties to facilitate the development of high performance and eco-friendly materials for engineering applications.

2. Materials and Methods

The matrix used in this study is a thermoplastic PHBV (Enmat Y100P), purchased from TianAn Biopolymer (China). It contains 3% molar content of 3-hydroxyvalerate (HV), a density of 1.25 g/cm^3, a melt flow index of 5.2 g/10 min and a melt flow window of 170-180 ºC. Pineapple leaf fibre (PALF) (Serat Alfibre, West Java, Indonesia), with a density of 1.52 g/cm^3 was used as a reinforcement. Sodium hydroxide and chloroform, each with a purity of 99.99% (laboratory grade), were purchased from Merck and Co. and used as solvents.

2.1 Composite production

The PALF was alkaline-treated with a 5% NaOH aqueous solution (w/v) before fabrication. After treatment, the fibres were cut into 6 mm using a precision paper cutter. To ensure consistency, random samples of the cut fibres were observed under an optical microscope The length variation was about ±0.5 mm. The PALF fibres were made into a thin film using a paper-making method and stored in a drying cabinet until needed

The PHBV pellets were dried in an oven at 60 ºC to eliminate a moisture. A 5 wt.% PHBV solution (w/v) was prepared via dissolving them in chloroform. PALF films were dipped into the PHBV solution for coating and then dried in a vacuum oven at 60°C overnight to ensure the solvent was completely removed. The pre-pregs PALF and plain PHBV films were stacked and compression moulded at 175°C, as shown in Figure 1. The composites were made with different PALF contents, namely 10,20,30, and 40 wt.%. Using pre-impregnated PALF film helped to ensure proper fibre alignment and consistent infiltration within the composite.

Because of the narrow processing temperature range, high-temperature and high-shear methods, such as extrusion or injection moulding, can lead to thermal degradation. To avoid this, low temperature solution casting and hot-pressing method have been used in this study, to preserve the integrity of PHBV and improve fibre wetting. Details about the alkaline treatment, pre-pregging parameters, and fabrication process can be found in previous publications [5].

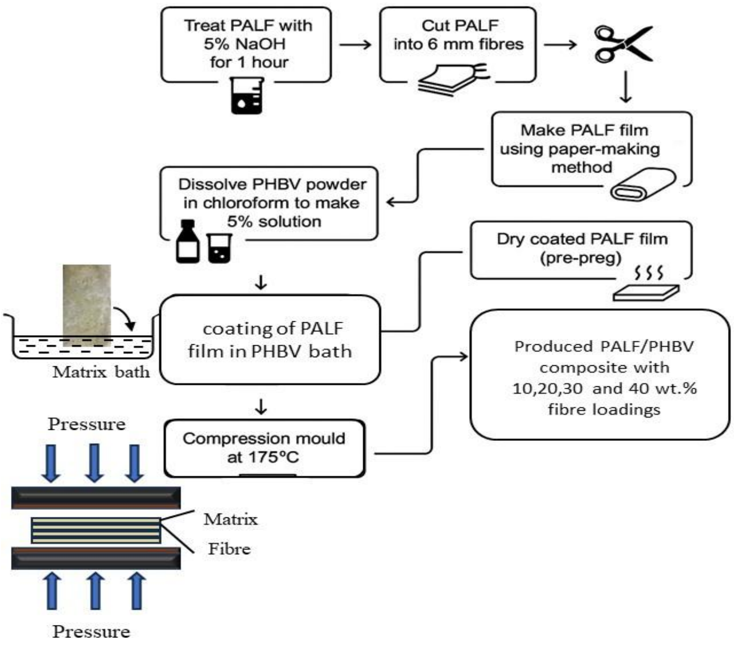

Fig. 1. Preparation and fabrication processes of PALF/PHBV composites

2.2 Tensile tests

The tensile properties of PALF/PHBV composites were measured using a 20 kN Universal Testing Machine (AGS-X Shimadzu, Japan) following the ASTM D3039 standard. The tests were conducted at a crosshead speed of 2 mm/min on samples with 20 ± 0.1 mm x 3 ± 0.1 mm dimensions with a gauge length of 80 mm. The samples were manually secured using serrated mechanical grips without using the end tabs. The applied force (N) and elongation (mm) were recorded. Five samples were tested for each fibre loading condition, and the reported average value. All samples were conditioned under controlled conditions of 23 ± 3ºC and 50 ±10% RH.

2.3 Weibull distribution

In this study, a two-parameter Weibull statistical analysis was applied to evaluate the variability in tensile strength of PALF/PHBV composites at different fibre loadings. The cumulative distribution function of the two-parameter Weibull distribution is expressed by Equation 1 [5, 14,16].

$$F = 1 - \mathrm{e}\left(-\frac{\sigma}{\eta}\right)^{\beta} \qquad \dots (1)$$

Where F is the cumulative density probability function (failure probability) at a tensile stress, σ, while η is the scale parameter (Weibull strength); and β is the shape parameter (Weibull modulus), which can be written in the form of a linearized equation (Equation 2) [5,14,16].

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

The Weibull parameters specifically (shape, β, and scale, η) were determined using the linearized probability plotting method which applies least-squares regression of the transformed Weibull equation. Parameters' shape and scale were derived from the slope and intercept of the linear regression. The accuracy of fit was evaluated through the coefficient of determination (R^2). The cumulative probability density is expressed in terms of the median rank formula [5,14,16] as given by Equation 3:

$$\mathrm{P} = \frac{i\text{-}0.3}{n + 0.4} \qquad \dots (3)$$

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

3. Result and Discussion

3.1 Tensile properties of the composites

The neat PHBV and PALF/PHBV composites with different fibre loadings were tensile tested until fracture (Figure 2). All the samples fractured within the gauge length. The tensile property values are shown in Figure 3. The addition of PALF loadings from 10 to 40 wt.% improves the tensile performance of the PALF/PHBV composites. Statistical analysis conducted using a one-way ANOVA indicated that the incorporation of PALF into the PHBV matrix significantly ($p<0.05$) enhanced the tensile properties of the bio composite. The maximum tensile strength and modulus of elasticity were achieved with the addition of 30 wt.% PALF, yielding 46.54 (± 4.12) MPa and 10.36 (± 0.81) GPa, respectively.

The improvement in the tensile strength and modulus is due to effective stress transfer between the PALF and PHBV matrix [2, 9, 17]. Observation of the fractured surface of 30 wt.% PALF/PHBV composite (Figure 4) shows fibre fracture extending across the surface of cracks, commonly referred to a fibre bridging. This shows the composite can withstand stress prior to failure, improving its tensile strength and underscoring the effective role of the reinforcement in transferring load. These findings are consistent with earlier reports that highlight the mechanical benefits of PALF reinforcement due to its high cellulose content and effective stress transfer within the matrix [18].

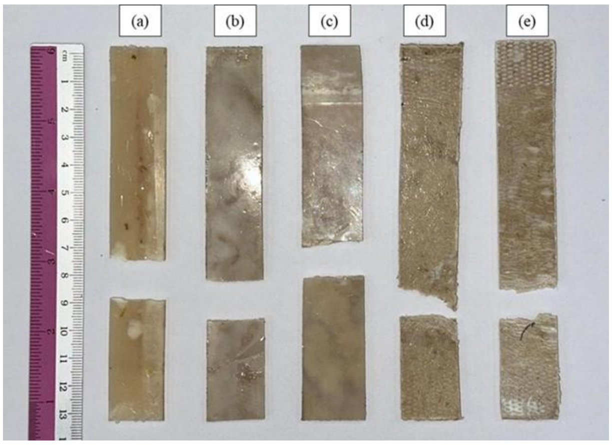

Fig. 2. Fractured specimens of PALF/PHBV composites with different fibre loadings (a) unfilled, (b) 10 wt.%, (c) 20 wt.%, (d) 30 wt.% and (e) 40 wt.%.

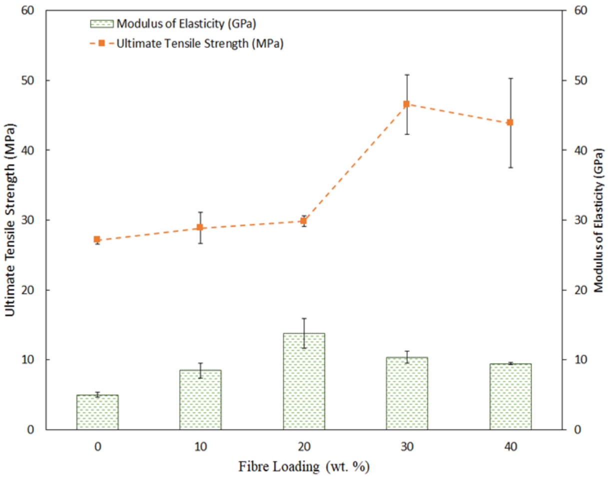

Fig. 3. Influence of fibre loadings on tensile properties of PALF/PHBV composites

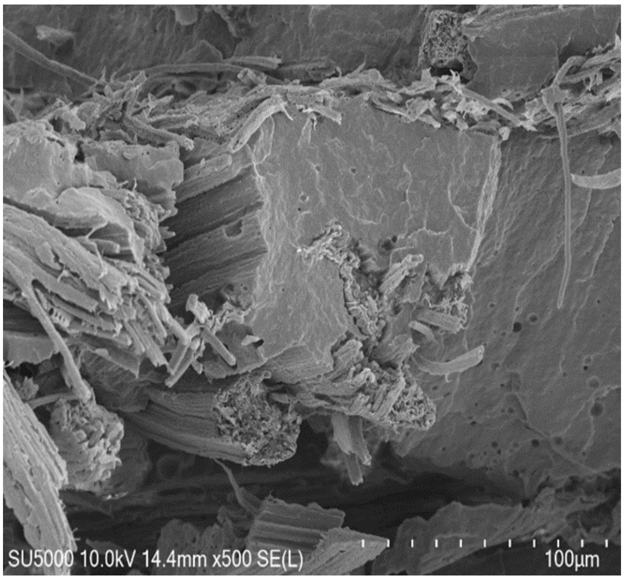

Fig. 4. Tensile fracture morphology of the PALF/PHBV composite containing 30 wt.% fibre loading

3.1 Weibull distribution

In this study, a two-parameter Weibull distribution was used to analyse the effect of the fibre loading on the PHBV and PALF/PHBV composite performance. The scale (η) and shape parameter (β) for the PHBV and its composite were determined using Equation 2, in addition to the corresponding strength values through a linear curve fitting. The Weibull plot parameters for the PALF/PHBV composite in terms of tensile strength with varying fibre loadings are shown in Figure 5. For each fibre loading, a total of 5 samples were subjected to testing. The correlation coefficient (R^2) values were calculated for each of the fibre loadings, all of which exceeded 90%. These high correlation values indicate a strong agreement between the experimental data and the fitted curve, as the previous study presented similar findings [16]. Table 1 shows the range of the Weibull modulus values for all fibre loadings.

The Weibull modulus (β) or shape parameter is indication of the degree of data variability. A higher value of shape parameter means the results are more uniform, while lower value indicates a wider variation in strength [5,14]. In this study, neat PHBV showed a relatively high modulus (β = 29.28), which point out to more consistent mechanical behaviour. On the other hand, the PALF/PHBV composites recorded lower β values, with the 40 wt.% PALF/PHBV sample produced the lowest (β = 6.14). This wider scatter probably due weak fibre- matrix bonding and uneven stress transfer within the composite [19, 20]. As fibre loading increased, the PALF layers become thicker, and this may hinder proper wettability during fabrication. The PHBV matrix might then fail to penetrate fully between the fibres, leaving voids or weak regions that reduce stress transfer efficiency. From the SEM images (Figure 6), fibre pull-out and debonding can be clearly seen, with little matrix residue on the fibre surface, which support this observation. Some of the variation could also be related to the inherent variability of the PALF itself.

A trade-off seems to be between improving tensile strength and maintaining reliability in the composites. The 30 wt.% PALF sample has showed the highest tensile strength, likely due to the effective transferring between fibre and the matrix. On the other hand, its lower Weibull modulus (β) indicates that the failure was less consistent variability might arise from uneven wetting or local stress concentrations when fibre loading becomes excessive. Although data on β values for PHBV based composite is quite limited, studies on other natural fibre systems have supported this finding. For example, Wand and Shao [21] reported β value ranging from 2 and 6 for bamboo fibre composites. As a result, this considerable variability has often been seen in natural fibre-based materials. Having compared the 30 wt.% PALF/PHBV samples, this study showed a β value exceeding 10 indicated much higher reliability. This is likely to reflect the positively effect of fibre treatment and fabrication method approach in improving both consistency and overall mechanical performance

In general, nature fibres have higher variability in mechanical properties comparing with synthetic fibres, due to inherent flaws and irregularities. Due to inherent flaws and irregularities of the high variability of nature fibres. The composite performance can be strongly impacted, reducing reliability and making failure behaviour are more difficult to predict. Adding more fibre can raise the risk of uneven distribution and inconsistent matrix–fibre bonding This variability is responsible for the wider scatter in tensile strength observed in the PALF/PHBV composites.

In this study, the observed variation in tensile strength is determined as a result of the natural differences in PALF fibres, such as their diameter and overall structure. Given that the PALF film were produced using a paper making technique. The fibre may not be effectively distributed, which can introduce potential weak points. Consequently, Careful control over fibres processing and uniform dispersion is necessary to produce stronger and more consistent composites.

Table 1 shows that the theoretical mean tensile strength values agree closely with experimental measurements. For the neat PHBV, the difference between theoretical and experimental values was less than 1%, while the PALF/PHBV composites was about 7%.

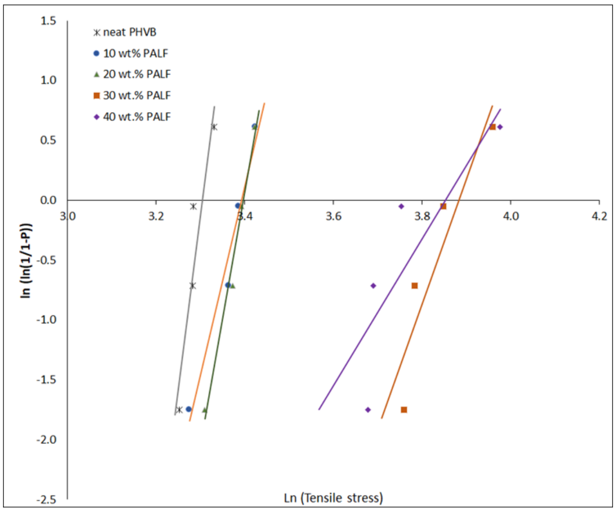

Fig. 5. The Weibull lines for PALF/PHBV composites at different fibres loadings

Table 1,
The Weibull parameters of the PALF/PHBV composites at different fibre loadings.

PALF loading (wt.%)	Weibull Strength, η (MPa)	Weibull Modulus, β	Experimental Strength, σ (MPa) (± S.D)	Percentage of error (%)
Neat PHBV	27.22	29.28	27.30 (±0.58)	0.31
10	29.74	15.66	28.29 (±2.20)	2.85
20	29.86	21.06	29.82 (±0.75)	0.11
30	48.57	10.43	46.54 (±4.12)	4.18
40	47.11	6.14	43.89 (± 6.41)	6.83

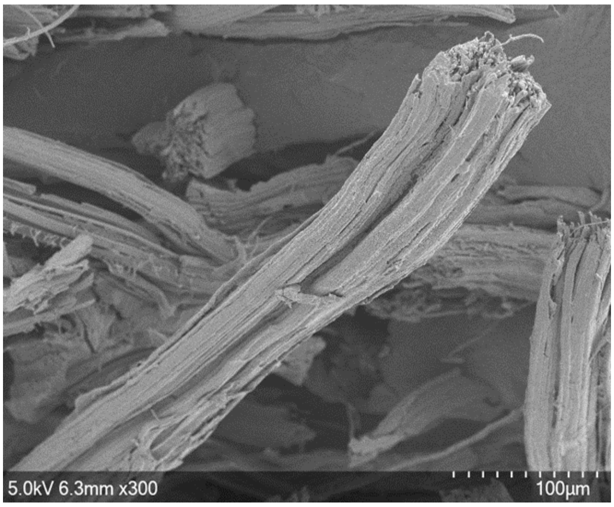

Fig. 6. Tensile fracture of the 40 wt.% PALF/PHBV composite

4 Conclusions

This study highlights the clear impact of adding PALF to the PHBV matrix on the composites' tensile strength and modulus. As the fibre content increased, both properties improved, peaking at 30 wt.% PALF with tensile strength and modulus reaching 46.54 MPa and 10.36 GPa, respectively. Interestingly, the Weibull analysis showed that these gains came with greater variability. In general, the PALF/PHBV composites had lower Weibull moduli, with the 40 wt.% PALF sample showing the lowest at β = 6.14. This increased variation is likely linked to weaker fibre/matrix bonding and the inherent irregularity of the PALF. At the same time, the scale parameter increased by 73% for the 30 wt.% composite compared with other loadings. SEM examination supports these observations, revealing both fibre pull-out ad breakage which suggest that the matrix/fibre interface was not fully bonded. In summary, while adding PALF may enhance tensile performance, achieving more uniform bonding and consistent reliability remains a key challenge for this system.

Therefore, upcoming studies could focus on enhancing the PALF/PHBV composite through exploring strategies, such as fibre surface modification, usage of compatibilizer or coupling agents and better control of processing conditions. These approaches should also help minimize variability and improve the reliability of the PALF/PHBV composites for engineering purposes.

Acknowledgements

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تأثير احمال الالياف على مقاومة الشد للمواد المركبة المقواة بألياف PALF/PHBV: تحليل ويبل ثنائي المتغيرات

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المستخلص

يتزايد الاهتمام بالمواد القابلة للتحلل الحيوي والمستدامة بشكل مطرد، مما دفع إلى البحث في البوليمرات الحيوية المقواة بالألياف الطبيعية. من بين هذه البوليمرات، يتميز بولي (٣-هيدروكسي بيوتيرات-كو-٣-هيدروكسي فاليرات (PHBV) بقابليته للتحلل الحيوي وتوافقه الحيوي، إلا أنه يتميز بالهشاشة وخصائص محدودة أو معدومة تُمكّن من تطبيقه في مجالات هندسية أوسع. بالإضافة إلى ذلك، يمكن أن يؤدي التباين الطبيعي للألياف إلى أداء ميكانيكي غير متسق، ومع ذلك، فقد تناولت دراسات قليلة فقط هذا التباين من منظور إحصائي. في هذه الدراسة، تم التحقيق في تأثير إضافة ألياف أوراق الأناناس (PALF) عند أحمال مختلفة (١٠-٤٠٪wt) على السلوك الميكانيكي لـ PHBV . تم تحليل مقاومة الشد باستخدام طريقة Weibull ثنائية المتغيرات الخطية لتحديد متغيرات المقياس (η) والشكل(β) ، مما يوفر نظرة ثاقبة على كل من مقاومة الشد النموذجية وتباين أداء المادة المركبة. تُظهر النتائج أن مقاومة الشد تزداد مع تحميل الألياف، حيث بلغت أعلى قيمة ٤٦,٥٤ ميجا باسكال (±٤,١٢) عند ٣٠٪ wt من اليافPALF ، وهو ما يتوافق مع ارتفاع بنسبة ٧٣٪ في متغير المقياس (η) مقارنة بالأحمال الأخرى. تم إنشاء ارتباط قوي بين النتائج العملية ونموذجWeibull ، مع ($R^2 > 0.9$) والاختلافات أقل من ١٪ و ٧٪ لألياف PHBVالنقية وألياف PALF / PHBV على التوالي. كشف تحليل المجهر الإلكتروني الماسح (SEM) عن علامات تمزق الألياف وخروج الألياف من المصفوفة، مما يشير إلى أن فشل المادة المركبة كان نتيجة لضعف الترابط السطحي بين المصفوفة والألياف. تمكن تحليل Weibull من توفير تقييم أكثر شمولاً لموثوقية السلوك الميكانيكي للمادة المركبة لأنه أخذ في الاعتبار التوزيع الإحصائي لجميع النتائج المقاسة بدلاً من مجرد قيمة المتوسط. أظهر نموذج Weibullثنائي المتغيرات أن المادة المركبة التي تحتوي على ٣٠٪ wt من الياف PALF قد حقق أقصى مقاومة شد مع موثوقية مرضية. تشير هذه النتيجة إلى توازن مثالي بين الأداء والتباين، وهو أمر ضروري للتصميم الموثوق به للمواد المركبة المستدامة المقواة بالياف PHBV.