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The mechanical properties of natural fibre composite laminates: a statistical study

J.P. Torres[a,*], L.-J. Vandi[b], M. Veidt[a], M.T. Heitzmann[a]

[a]*School of Mechanical and Mining Engineering, The University of Queensland, Brisbane, QLD 4072, Australia*
[b]*School of Chemical Engineering, The University of Queensland, Brisbane, QLD 4072, Australia*

Abstract

The use of long natural fibres (LNF) as reinforcement in composite systems for structural applications has been steadily growing in the automotive and construction industries as these materials offer sustainability benefits combined with high specific strength and stiffness. However, the performance of natural fibres has been questioned by a high variability in their mechanical properties and design data for structural reliability analysis of LNF composites are not yet available. Here, we present a statistical study of the elastic modulus, strength and failure strain of a comprehensive set of LNF composite systems. We have found that the variability of LNF laminate properties is similar to that of carbon fibre laminates. We provide recommendations to apply the statistical parameters determined here to the design of natural fibre composite structures. Our findings provide a deeper understanding of LNF composites reliability and are important for the further acceptance of these materials by the industry.

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[*] Tel: +61 7 3365 3987
Email address: `juan.torres@uq.edu.au,juantorresmdp@gmail.com` (J.P. Torres)

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1. Introduction

Driven by the introduction of regulatory norms demanding more environmental-friendly products [1, 2], the use of natural fibres as reinforcements for composites materials has increased significantly in the last decade [3]. Natural fibres such as flax, jute, hemp and kenaf offer low carbon footprint and biodegradability advantages combined with a high specific strength and stiffness at a low-cost [4, 5, 6]. The use of short natural fibres for semi-structural or non-structural components has already been embraced in the automotive industry where lighter vehicles imply reductions in fuel consumption and carbon emissions [7]. On the other hand, structural components demand superior mechanical properties which can be achieved with the use of long natural fibres (LNF), now increasingly used for civil engineering applications [8]. However, design data for structural reliability analysis of LNF reinforced composites is rare and their performance has been questioned by a perceived high variability in their mechanical properties [9, 10]. These uncertainties have partially discouraged the commercial application of LNF composites. Therefore, developing a deeper understanding of the variability in the mechanical properties of these materials is necessary before they can be further accepted and adopted by industry. Of special interest is the generation of material databases that will allow the development of design guidelines and allowables based on the statistical behavior of LNF reinforced systems. These property databases can be further used to implement models and perform numerical simulations which would reduce the need for costly physical experimentation and prototype testing.

Current design practices for composite laminate structures [11] involve the determination of design allowables either by testing the laminate itself or by combining a ply-level material property database with composite lamination theory [12]. In this case, the ply-level properties are typically determined by testing unidirectional specimens in tensile, compression and shear modes. To a higher degree than other materials, the mechanical properties of composites are statistical in nature due to inherent uncertainties in design variables such as fibre and matrix mechanical properties, fibre and void volume ratios, ply misalignment and thickness, temperature and moisture [13, 14, 15]. This difficulty can be treated using probabilistic design methodologies which account for the scatter in these variables by integrating their statistical distributions in an overall statistical analysis framework. The use of this approach is relatively well established in the aerospace and civil engineering sector [16, 17, 18] and several software codes for reliability evaluation have been developed [19, 20, 21]. As opposed to the traditional deterministic design practices where material parameters are treated as known constants and large safety factors are employed, the probabilistic design methodologies allow to *quantify* the inherent risk of failure. However, a significant drawback of this approach is the large experimental data that is required to establish input distributions.

So far, probabilistic design in composites has been exclusively used for carbon fibre and glass fibre composites. Conversely, the use of natural fibres as reinforcement for structural applications is only beginning to emerge

[22, 23]. The variability of plant fibre properties is influenced by variables such as chemical composition [24], plant species, location of fibre in the plant, environmental conditions in the cultivation area, and fibre processing methods [25]. In addition, the inherent irregularity of shape and cross-sectional area in natural fibres further contributes to their variability. These irregularities create experimental difficulties in the measurement of fibre endogenous parameters such as diameter or lumen size. This requires the use of techniques such as high resolution dimensional measurement [26], optical, laser or electron microscopy [27, 28], or X-ray computer tomography [10]. Several investigations have quantified the variability in stiffness, strength and failure strain of natural fibres using the coefficient of variation C_{V} (i.e. the ratio of standard deviation to mean value). Virk et al.[10] studied the fracture behavior of different sets of 50 single jute fibres and found maximum variabilities of 31% in stiffness, 46% in strength, and 35% in strain to failure. Baley tested flax fibres under uniaxial tensile loading and found a variability of 28% in stiffness, 36% in strength, and 26% in strain to failure [29]. Adusumali et.al.[30] also studied the tensile behaviour of flax fibres and found a scatter 48% in stiffness, 36% in strength, and 14% in strain to failure. Other publications regarding the variability of single fibres include the work of Eichhorn et.al. for hemp fibres [31], Xue et.al. for kenaf fibres [32], and Bodros et.al. for stinging nettle fibres [33]. However, the statistical aspects of the mechanical response resulting from assembling these fibre systems into a composite laminate has not been analysed in these investigations. Additional work by Virk et al.[34, 28] and Andersons et al.[35] have studied the strength and failure strain distributions of flax fibres using modified two-parameter Weibull

statistics. However, the statistical aspects of the mechanical response resulting from assembling natural fibre systems into a composite laminate has not been analysed in these previous investigations (it must be noted nevertheless, that a previous study by Bader et.al. on the strength of carbon fibres [36] had found a higher weibull modulus (i.e. lower variability) of carbon fibre composite compared to single carbon fibres).

A previous work by the authors has studied the statistical aspects of single flax fibres and unidirectional flax reinforced composites to conclude that when long natural fibres are used as reinforcements in composite laminates, the variability of the resulting composite is significantly lower than that of the constituent fibres. The present work work follows up to assess the statistical behavior of a wider range of LNF reinforced composite laminate systems. Further understanding of these systems can help in enhancing design and reliability assessment practices in natural fibre composites to extend their use in commercial applications.

2. Experimental

2.1. Composite laminate tests

An extensive experimental program was carried out to determine the statistical distributions of mechanical properties. Several material and geometrical configurations were studied. Reinforcement fibres include unidirectional flax fabrics FLAXPLY provided by Lineo [37], and jute and flax woven fabrics provided by Biotex [38]. The resin system used in all laminates is a SR-8100 Epoxy resin produced by Sicomin [39], except for Flax-VE-0° which uses an EPOVIA®OPTIMUM KRF2000SE vinil-ester matrix provided by Polyint

Composites Australia [40]. Fibre areal weight and fabric configuration are shown in Table 1.

Composite panels were manufactured by vacuum assisted resin transfer moulding (VARTM) on a rectangular tool plate. Before infusion, the fibres were dried in an oven at 60°for 6 hours. After infusion panels were left for 24 hours at ambient temperature and post-cured at 60°for 8 hours under a 80 kPa vacuum. Rectangular specimens of 250 mm x 25 mm dimensions were cut from the panels using waterjet cutting. Fibre weight before infusion and panel weight after manufacturing were measured to calculate composite fibre volume fraction according to $V_{\mathrm{f}} = w_{\mathrm{f}}\rho_{\mathrm{c}}/\rho_{\mathrm{f}}$, where V_{f} is the fibre volume fraction, w_{f} is the fibre weight fraction, ρ_{c} is the composite density and ρ_{f} is the fibre density. An important simplification is made in this calculation by assuming the composite has no voids. However, as we will show later, the conclusions reached in this paper are still valid for different volume fraction values. Further details including number of specimens, individual specimen geometry and testing results are detailed in the complementary Data in Brief article [41]. As is the typical case in conventional VARTM processes, fibre volume fraction and sample thickness were only partially controlled through the vacuum pressure level and the number of fibre plies [42].

Uniaxial tensile tests were carried out in an electromechanical INSTRON 5584 frame using a 30 kN load cell following the procedures depicted in [43]. Tensile specimens were supported with hydraulic grips with a gripping force that prevented both specimen sliding and premature failure at the grips. Engineering strain was measured using an optical extensometer. Elastic modulus was determined from the initial slope of the experimental stress-strain curves in the strain range 0.001 - 0.003 mm/mm. Strength and strain at failure were measured at the point corresponding to the maximum axial load in the stress-strain curves. Raw data files for all test results can be found in Ref [41].

2.2. Determination of the statistical distribution parameters

Statistical distributions were fitted to the sample data using an optimization procedure based on the Nelder-Mead method which is readily available in the open source Python library scipy.optimize [44]. This operation iterates different estimations of the parameters in a chosen probability density function (PDF). For each parameter estimation, the corresponding PDF is compared to the experimental data through an objective function ϕ. This function ϕ is the least-squares difference between the experimental and distribution values. This procedure is repeated until a minimum in ϕ is found. Once an iteration sequence has converged to its minimum, the PDF parameters are stored along with their corresponding ϕ value. The best fit PDF is the one that returned the minimum ϕ value. In this investigation, we fitted the experimental results using uniform, normal, lognormal and Weibull PDFs which can be handled using the open source python library scipy.stats [45]. For this work, we have used the two-parameter Weibull distribution given by [46]:

$$f(x) = \frac{\beta}{\eta}\left(\frac{x}{\eta}\right)^{(\beta-1)} e^{-\left(\frac{x}{\eta}\right)^{\beta}} \tag{1}$$

where $f(x)$ is the probability density function, x is the distribution variable (e.g. strength or failure strain), β is the shape parameter, also known as Weibull slope or modulus, and η is the scale parameter. Note that β is dimensionless while η has the units of the distribution variable. Additional details regarding the calculation of the Weibull distribution parameters can be found in the accompanying Data in Brief article [41].

3. Results and discussion

3.1. Statistical nature of LNF composites properties

To study the main aspects of the stiffness and failure response of long natural fibre composites we carried out uniaxial tensile tests on rectangular specimens. For each material system (see Table 1) we have determined the elastic modulus, strength and failure strain together with a statistical analysis of their arithmetic mean μ, standard deviation σ_{sd}, and coefficient of variation $C_V = \sigma_{\mathrm{sd}}/\mu$. The general results are assembled in Fig.1 which shows whiskers plots for all 3 properties. The mean value is denoted with a vertical tick and a horizontal black whisker represents the width of one standard deviation above and below the mean value. In addition, the figure features colored dots that represent each of the individual values. Elastic modulus and strength of the carbon fibre specimens are shown in a separate chart since their absolute values are approximately one order of magnitude higher than those of natural fibres.

As expected, modulus and strength of the Flax-90° and Flax-Short systems are lower than those of the other systems where there is a greater contribution of the reinforcement fibres in the loading direction. Accordingly, the 0° unidirectional laminates show the highest stiffness and strength values. For those systems featuring long fibres aligned in the 0° and 90° directions (crossply, plain and twill weaves), the elastic modulus is fairly similar. On the other hand, the strength of Flax-CP is higher than that of Flax-Satin, which is higher than the twill and plain weave fabrics. We consider that this tendency is given by the fabric crimp (i.e. fibre ondulation), whereby an increase in crimp amplitude decreases the effective alignment of the fibre in the loading direction and thus, the global composite strength [47, 48]. This is consistent with the fact that each ply in the Flax-CP laminates is non-woven (i.e. has minimum crimp) while plain and 2x2 twill weaves have the highest crimp. On the other hand, the failure strain behavior among the different material systems does not show a clear correlation to the fibre architecture.

To compare the variability in elastic modulus, strength and failure strain, we have examined the coefficient of variation C_V corresponding to the experimental distributions. The C_V value is an appropriate comparative measure between the different materials and properties as it is a dimensionless ratio, normalized by the property mean value, whose absolute magnitude can vary greatly. The results are shown in Fig.2 and Table 2. Lower C_V values imply a smaller expected scattering in the corresponding variable. The short-fibre mat system (Flax-Short) shows the highest variability for all 3 properties. This observation can be attributed to the inhomogeneity of the mat reinforcement geometry, i.e., the uneven distribution of the local *mass/unit area* values which gives place to a non-homogeneous fibre volume fraction over the test specimen volume.

With the exception of the Flax-Short system, the variability in elastic modulus for all materials is between 5% and 10%, which is considerably lower than the reported values for single flax fibres in the range of 28% to 48% [29, 30]. The same considerations apply for the scatter in strength and strain to failure. These observations are of great importance for the understanding of LNF reinforced composites reliability as the variability of their mechanical properties is significantly lower to what was expected from the previous studies of single natural fibres. that For the woven fabric composites, strength systematically exhibits a lower variability ($\approx 5\%$) than strain to failure. This suggests that strength may be a more reliable predictor of failure in these material systems. The unidirectional 90° flax laminate presented strength and strain to failure C_V values slightly above 10%. Since the observed failure mode for this system was matrix cracking, we speculate that this greater variability can be attributed to a higher sensitivity of matrix failure initiation and sudden propagation to surface defects and porosity.

To compare the effect of matrix material in the composite variability we analyzed the results for epoxy (Flax-0°) and vinyl ester (Flax-VE-0°) matrix composites. A comparison of their C_V values indicates that the vinyl ester resin introduces a moderately higher scatter in the composite properties. Since manufacturing variables such as tool and bagging quality, vacuum level in bag, operator skills and layup tools can be considered constant throughout our study, the lower C_V of Flax-0° compared to Flax-VE-0° suggests that the fibre/resin wettability properties [49] can have a minor effect on the composite part variability.

To assess the effect of different reinforcement fibres in the composite variability, we compared the C_V results for flax (Flax-0°) and carbon fibre (Carbon-0°) reinforced unidirectional laminates. Results show that the variability in the flax reinforced composite is indeed lower than that of the carbon fibre laminate. Here again, since manufacturing variables have been kept constant, we suggest the variability may arise due to differences in reinforcement permeability, resin/fibre wettability and resin content [13]. Most important is the observation that variability in natural fibre composite laminates is indeed in the same order of magnitude to that of carbon fibre composites. This argument was proposed in a previous investigation by the authors, however we have now tested its validity for two material systems under the same manufacturing and testing conditions.

3.2. Implications for structural design of LNF composites

To characterize the statistical distributions of elastic modulus, strength and failure strain, we have fitted the experimental values to uniform, normal, lognormal and Weibull probability distribution functions (PDFs) and determined the best fit (see section 2.2). For all tested materials, a normal distribution best-fitted the elastic modulus and the Weibull distribution presented the best fit for both strength and failure strain, which is consistent with what is typically observed in other composite systems [15]. The results are summarized in Table 3. Fig.3 illustrates the typical aspect of the stress-strain curves (in this case for the Jute-Plain samples) together the corresponding shape and PDF fit for the strength and stiffness distributions.

We have mentioned in the introduction of this paper that the generation of design allowables for composites structures can be treated using ply-level material properties and that a more reliable design can be carried out by incorporating the statistical description of these properties in a probabilistic design framework to quantify the structural risk of failure. Typically, the required statistical descriptors are the PDFs that best-fit the experimental distributions of the mechanical properties and their associated parameters.

The estimation of these statistical parameters requires testing of large number of specimens which can be costly and time consuming. To overcome this obstacle, we propose that, when designing for a LNF composite in the same family of those analyzed here, the distribution shapes and variabilities determined in tables 2 and 3 can be combined with analysis of small data sets to provide an approximation of the statistical description of the material properties. The small additional data sets would be required to adjust the material mean value μ_{m} since they are dependent on manufacturing variables and especially the fibre volume fraction.

Following this approach, when modelling the elastic modulus with a normal distribution, the C_V values presented in table 2 can be readily used to estimate the standard deviation of the sample using:

$$\sigma_{\mathrm{sd}} = C_V \cdot \mu_{\mathrm{m}} \tag{2}$$

On the other hand, to model strength and failure strain distributions, the Weibull shape parameter β can be approximated from the results provided in table 3 and the scale parameter η can be adjusted from the data sets using the equation for the mean of the Weibull distribution [46]:

$$\mu = \eta \cdot \Gamma\left(\frac{1}{\beta} + 1\right) \tag{3}$$

where Γ is the gamma function given by $\Gamma(n) = \int_0^\infty e^{-x} x^{n-1} dx$ [50]. Therefore:

$$\eta = \frac{\mu_{\mathrm{m}}}{\Gamma\left(\frac{1}{\beta} + 1\right)} \tag{4}$$

Similarly, the same approach can be carried out if the mean value for the material is obtained through micromechanical analysis using the constituent material properties [12]. For instance, by using rule of mixture based models to determine elastic modulus and strength from fibre and matrix properties, and using the determined values as the μ_{m} values in the analysis described above (Eqs. 2 and 4).

4. Conclusions

We have analyzed the statistical nature of the mechanical properties of several natural fibre reinforced composites. The variability of elastic modulus, strength and failure strain using the coefficient of variation C_V was assessed. Our results show that the variability of long natural fibre composites is in the same order of magnitude to that of carbon fibre composites manufactured with a comparable process. In addition, our results indicate that short fibre composites have a higher scatter than long fibre composites. Most importantly, we observe that the variability of long natural fibre composites is significantly lower to that observed in previous studies of single natural fibres.

We suggest that the statistical parameters of the fitted probability distribution functions can be used to model the scatter in similar material systems with the aim of determining design allowables and perform probabilistic design analyses. These results can assist in the development of design guidelines and reliability assessment practices for natural fibre composites and extend their use in commercial applications.

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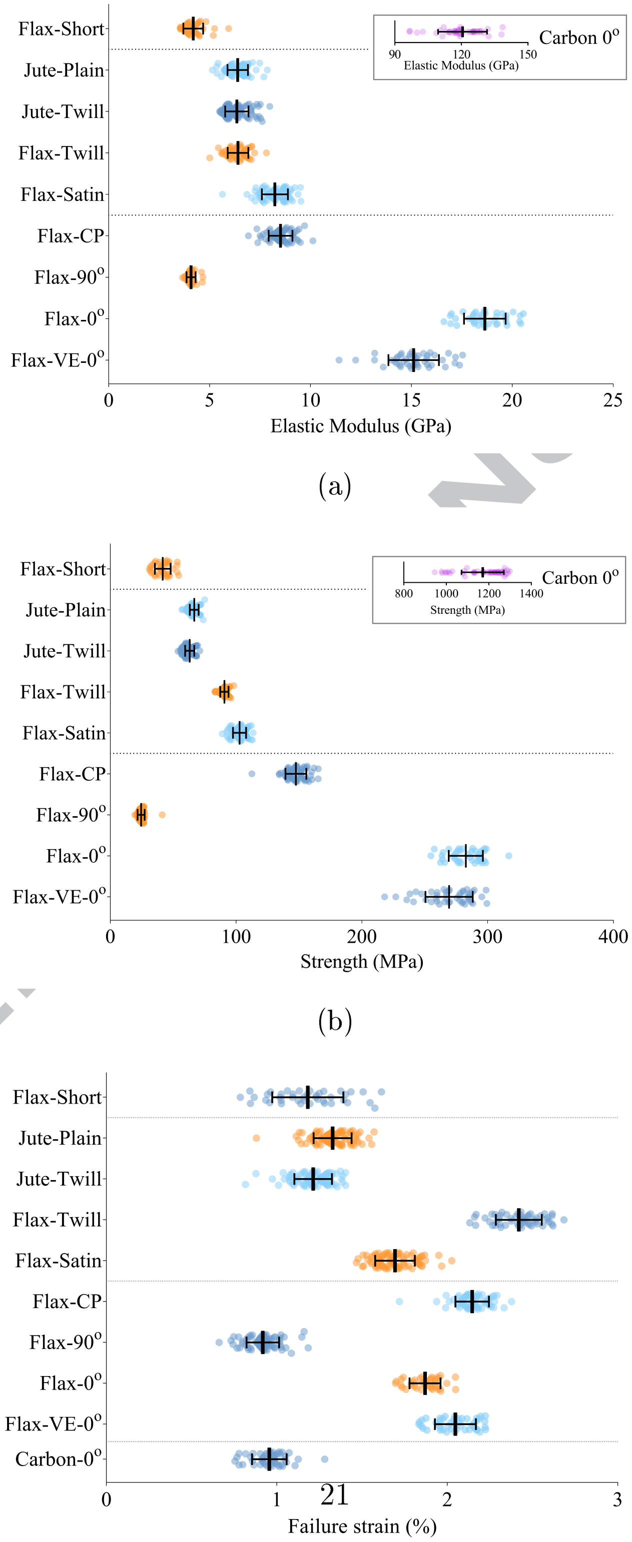

(a)

(b)

(c)

Figure 1: Elastic modulus (a), strength (b) and failure strain (c) distributions of the complete testing program. The bold vertical lines show the mean value; the ends of the whiskers show the width of one standard deviation; and the dots represent the individual experimental values.

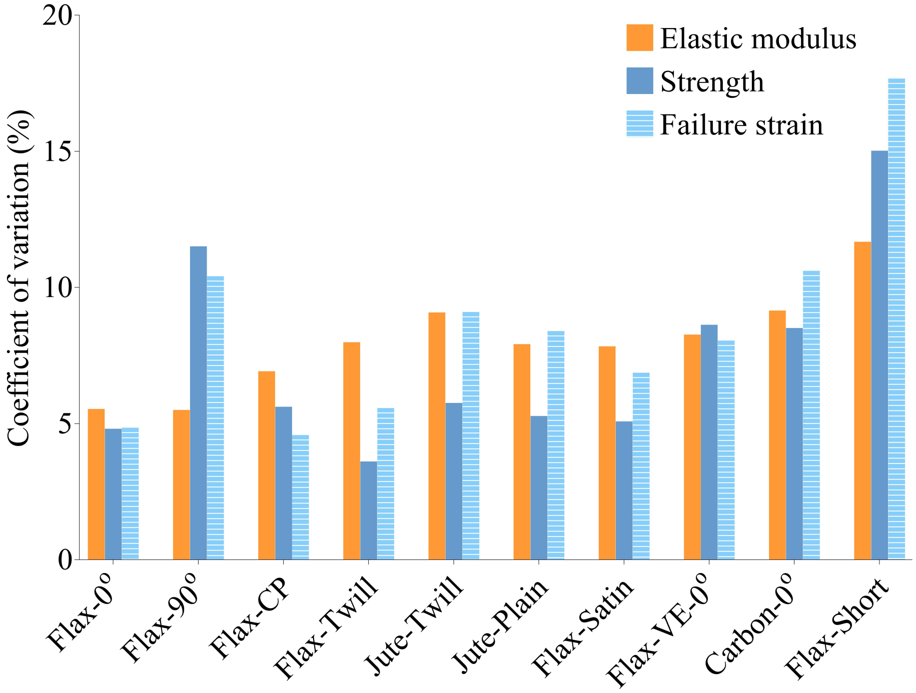

Figure 2: Elastic modulus, strength and failure strain variability, as measured by the coefficient of variation, for all the tested materials.

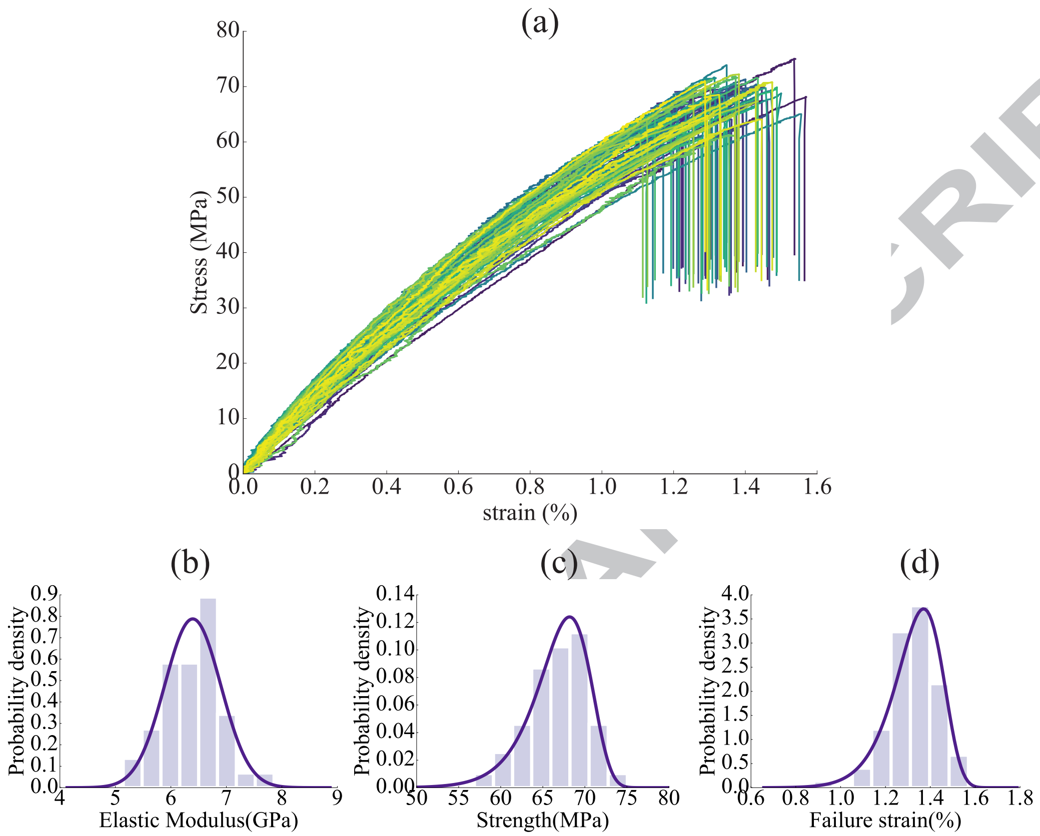

Figure 3: (a) Stress-strain curves for 86 samples of the Jute-Plain system. (b), (c) and (d): statistical distributions together with their probability distribution function fit: (b) Elastic modulus (Normal), (c) Strength (Weibull), (d) Failure strain (Weibull).

Table 1: Testing program for tensile tests

Material	Configuration	number of plies	V_{f}	Label
Lineo Flax 180g/m^2	0°	3	0.31	Flax-0°
	90°	4	0.31	Flax-90°
	$[0/90]_2\,s$	4	0.31	Flax-CP
Biotex Flax 400g/m^2	7x1 Satin Weave	4	0.35	Flax-Satin
Biotex Flax 200g/m^2	2x2 Twill Weave	3	0.35	Flax-Twill
Biotex Jute 550g/m^2	2x2 Twill Weave	4	0.36	Jute-Twill
Biotex Jute 500g/m^2	Plain Weave	4	0.40	Jute-Plain
Lineo Flax 180g/m^2 (vinyl-ester matrix)	0°	3	0.32	Flax-VE-0°
Carbon Fibre 300g/m^2	0°	6	0.48	Carbon-0°
Short-fibre Flax Mat	Random	1	0.25	Flax-Short

Table 2: Mean, standard deviation σ_{sd} and coefficient of variation C_V values for elastic moduli, strength and fracture strain

	Modulus (GPa)			Strength (MPa)			Fracture Strain (%)		
Material	Mean	σ_{sd}	C_V	Mean	σ_{sd}	C_V	Mean	σ_{sd}	C_V
Flax-0°	18.6	1.0	5.54	283.4	13.0	4.6	1.87	0.09	4.9
Flax-90°	4.1	0.2	5.50	26.1	0.8	2.9	0.89	0.09	9.7
Flax-CP	8.5	0.6	6.92	147.6	8.3	5.6	2.15	0.10	4.6
Flax-Twill	6.4	0.5	8.0	90.7	3.3	3.6	2.42	0.13	5.6
Jute-Twill	6.4	0.6	9.1	63.1	3.6	5.8	1.21	0.11	9.1
Jute-Plain	6.4	0.5	7.9	66.7	3.5	5.3	1.33	0.11	8.4
Jute-Satin	8.2	0.6	7.8	102.8	3.2	5.1	1.70	0.11	6.9
Flax-VE-0°	15.1	1.3	8.4	269.4	18.8	7.0	2.05	0.12	5.9
Carbon-0°	120.5	11.0	9.1	1171.2	100.4	8.6	0.95	0.10	10.5
Flax-Short	4.8	0.5	11.7	41.7	6.3	15.0	1.18	0.21	17.7

Table 3: Weibull distribution parameters for strength and fracture strain.

	Strength		Fracture Strain	
Material	Shape β	Scale η (MPa)	Shape β	Scale η (%)
Flax-0°	24.45	288.9	24.25	1.91
Flax-90°	41.10	26.4	11.93	0.93
Flax-CP	20.35	151.5	25.16	2.19
Flax-Twill	33.11	92.23	21.44	2.48
Jute-Twill	20.94	64.79	12.77	1.26
Jute-Plain	23.00	68.3	13.86	1.38
Jute-Satin	23.84	105.1	17.41	1.75
Flax-VE-0°	16.83	277.8	19.89	2.10
Carbon-0°	13.46	1216.7	11.11	1.00
Flax-Short	7.77	44.3	6.64	1.27