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ORIGINAL ARTICLE

Effect of eco-friendly chemical sodium bicarbonate treatment on the mechanical properties of flax fibres: Weibull statistics

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Abstract

In this work, a chemical treatment with different concentrations of $NaHCO_3$ (sodium bicarbonate) of 5%, 10% and 20% on the surface of the flax fibre for a period of 120 h at room temperature is achieved. The purpose of this study is to observe the effect of different treatment processes on flax fibres, which is to say on its mechanical properties such as strength and strain at fracture and Young's modulus. An important campaign of the test of more than 480 tests is carried out. Due to the variability of plant fibres, more than 120 samples were tested for each group at a gauge length (GL = 20 mm). The tensile mechanical property values of the flax fibres present a large dispersion of results; this is typical for natural fibres, hence the need for a statistical study. This dispersion has been studied and carried out by means and statistical tools such as the distribution of Weibull at two and three parameters by applying a prediction model to a confidence level at 95% CI and one-way ANOVA analysis of variance.

Keywords Natural fibres · Flax fibre · Statistical analyses · Mechanical testing · Weibull · ANOVA

1 Introduction

The use of natural fibres has many advantages because they are derived from a renewable and biodegradable resource. A major advantage is that they can be easily discarded at the end of their life cycle by cutting or by other methods that exist in the industry, which is not possible with synthetic fibres such as glass and carbon fibres. Indeed, these fibres offer particular advantages such as low cost, low density, lower pollutant emissions, acceptable special properties, renewable characteristics, improved energy recovery, non-abrasivity, availability, recyclability and a total biodegradability [1–7]. Consequently, these natural fibre advantages encourage the development of new applications in composites. The available technical studies suggest that these fibres have a real competitive advantage over synthetic fibres [1, 2]. Currently, lignocellulosic fibres are used as reinforcements in technical applications mainly in the automotive, packaging industry and in parts where high capacity is not required [1, 3, 4]. Today, the industrialists think about integrating bio-fibres into biocomposite parts in the aeronautical field [5]. The application of natural fibres is primarily motivated by a combination of environmental and economic concern [6].

Different types of plant fibres such as flax [8–12], jute [13, 14], sisal [15–17], *Phoenix dactylifera L.* [18], *Agave americana L.* [19], artichoke [20], hemp [21], *Native African Napier Grass* [22], *Furcraea foetida* [23], *Aegle marmelos* [24] and other lignocellulosic fibres are used as reinforcement for biocomposites.

Natural fibres such as flax fibres are derived from lignocellulosics and have shown their ability to be good candidates as a reinforcement for biocomposite materials [25, 26]. However, natural fibres have high moisture absorption. They are of hydrophilic nature, and therefore, they become weak at the surface between the fibre and the matrix. Due to the low fibre/matrix adhesion, the mechanical properties of biocomposites in natural fibre reinforcement are lower and therefore it becomes important to improve their adhesion by

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chemical methods, since the insertion of hydrophilic fibres into a hydrophobic matrix is not simple [27–30].

On the other hand, the structure of elemental flax fibre (*Linum usitatissimum*) is very complex and composed of cellulose, hemicellulose, pectin, lignin and other components [31, 32]. The flax fibre is indeed far from being considered as a single mono-filament fibre. The applications of green composites reinforced with flax fibres exist in the industry but are still limited because of their poor performance. In addition, the mechanical properties of these biocomposites depend principally on the fibre/matrix adhesion [33–36].

Additionally, like most natural fibres, the mechanical properties of vegetal fibres are also very variable, different parameters can influence their properties such as the variety of the fibre, its structure, the micro-febrile angle and its cellulose content [30–36]. From an environmental point of view, the main advantage of biofibres is its degradability. However, it also has disadvantages which are mainly its high moisture absorption, essentially due to cellulose, its sensitivity to acids, chemicals and bacteria, mainly due to the presence of hemicellulose, and finally its sensitivity to radiation ultraviolet due to lignin [37, 38].

The different treatments that can be applied are generally classified into two categories: chemical and physical. The former is the subject of investigation in this work. Among the chemical treatments available are sodium hydroxide (NaOH), acetylation (–CO-CH_3), the permanganate treatment ($MnO4^-$), silane (SiH_4) and pre-treatments with different coupling agents [39, 40]. Indeed, the role of these treatments makes it possible to clean the surface of the fibre, to modify its chemical composition, to lower the retention of moisture, to increase its roughness, to modify the mechanical properties and to improve its thermal stability. The chemical treatment of the fibre activates the hydroxyl group present in the cellulose, the major component of the fibres and the lignin where it can introduce the new parts which can actually engage with the polymer matrices [39, 40].

The chemical modifications that have been imposed on natural fibres by various researchers have resulted in an improvement in surface quality that adheres well to the polymer matrix [40]. The most commonly used fibre treatment is alkaline treatment, also known as mercerization. This is an ancient method widely used in the textile field [41]. It is also widely used with vegetal fibres when it is used to reinforce thermosets and thermoplastic matrix [42, 43]. The type of alkaline treatment and the percentage of concentration, the temperature, and the duration of the treatment influence the mechanical and physicochemical properties of the fibre [44].

In the study presented by Reddy et al. [45], the influence of the alkaline chemical treatment of *Borassus fruit fine* fibre for a concentration of sodium hydroxide of 5% for the duration of 1, 4, 8 and 12 h at a treatment temperature of 30 °C has been evaluated. The results obtained from the mechanical properties (tensile strength, strain at failure and Young's modulus) from this treatment respectively are 121 MPa, 58% and 35 GPa for 8 h of duration show the best behaviour for this plant fibre. Another similar work [46] presents the effect of different concentration of NaOH (0.5, 1, 1.5, 2.5 and 5 %) at a temperature of 100 °C for 1 h of time on date fibres (date palm tree). In this work [46], the average mechanical properties obtained are 840 MPa and 165 GPa respectively for the strength and Young's modulus, while Lu and Oza [47] for their part showed the chemical treatment effect such as silane and NaOH on the hemp fibre used as reinforcement in a high-density polyethylene matrix in order to show the change in the thermal properties of the hemp/HDPE composite. The hemp fibres have been immerged in a solution of NaOH at a concentration of 5% for a period of 16 h at a temperature of 50 °C. However, Venkateshwaran et al. [48] focused their work on the development of a Banana Fiber Reinforced Composite (BFRC) treated with an alkaline treatment (NaOH) at different concentrations (0.5, 1, 2, 5, 10, 15 and 20%). The analysis of their results showed that the composite produced by the treated fibres at low concentration (1% NaOH) gave much higher mechanical properties compared to other composites made from untreated fibres, or treated with the other concentrations.

According to the literature [12, 13, 20, 21, 49–53], lignocellulosic fibres are characterized by large dispersions of mechanical properties, hence the need to use statistical analysis methods such as two and three Weibull parameters. Virk et al. [13] conducted more than ten series of 100 specimens per GL of 100 to 300 mm, a total of 785 samples of jute fibres were tested under tensile static loading to study the influence of GL on the mechanical properties. The authors used various statistical analysis tools such as two Weibull parameters and log-normal probability density function of the values of the tensile strength and strain at failure [13].

Recently, Belaadi et al. [52] have determined the quasi-static tensile mechanical properties of more than 150 of natural sisal yarns, having a twist angle of 13° with a linear density of 232 ± 49 tex (g/1000 m). The authors used five different gauge lengths GL = 50, 100, 150, 200 and 300 mm to determine the influence of this parameter (GL) on the mechanical performance of the yarns. During the tests, the researchers noticed that the result obtained contain a large dispersion. To remedy this behaviour, the results were studied and analysed by probabilistic and statistical tools such as the Weibull distribution with two and three parameters using different probability and estimation index and also one-way analysis of variance (ANOVA).

The modification of vegetable fibres by sodium bicarbonate is an ecological technique (sodium bicarbonate, $NaHCO_3$, also known as baking soda). This ecological product is used in cooking, in the garden, in cosmetics, for medical purposes, to clean, without endangering public health or the environment (nature-friendly) [28]. There are few studies in the literature that use the treatment with sodium bicarbonate to improve the adherence of fibre/matrix [27–30]. In this context, the aim of this study was to investigate the mechanical behaviour in quasi-static tensile loading of over 480 untreated flax fibre samples and treated with $NaHCO_3$ samples at various concentrations with a period of emersion of 120 h. Then, the obtained results were analysed by applying probabilistic and statistical approaches such as the Weibull distribution in two and three parameters by the least square estimation method (*LS*), maximum likelihood (*ML*) and the analysis of variance one-way ANOVA with a prediction model to a confidence level of 95% CI using Minitab statistical software. Finally, to the best knowledge of the authors, this is the first time this approach is used for flax fibres.

2 Experimental procedure

The Flax is an annual herbaceous plant of temperate regions, belonging to the Linaceae family. The stem of this plant can reach 0.6 to 1.2 m in height and 1 to 3 mm in diameter. The flax fibres come from the plant stem. On a cross section of the flax stem, there are 20 to 40 bundles. Each bundle contains on average 300 to 400 of single fibres [54–56]. The flax fibres have an approximate diameter of 18 μm and a length of 0.8 to 1 m (Fig. 1). The flax fibres used in this work were provided by the BLIDA packaging and ropes factory in Algeria.

Natural flax fibres were immerged in $NaHCO_3$ solution (Sodium bicarbonate) with different concentrations (5%, 10% and 20%) for a total of 360 fibres treated for 120 h at room temperature. Then, the fibres are immerged in the distilled water for 2 to 5 min to remove any impurity, and finally, the fibres were dried in an oven at a temperature of 50 °C for a period of 6 h. Before conducting the quasi-static tensile tests, the diameters of the flax fibres are measured in ten places along the fibres. These measurements were taken using a ZEISS optical microscope equipped with a digitally controlled Moticam 2500 camera driven by a MoticImages Plus V2.0 image processing program. Ten diameter measurements are thus obtained for each fibre, and the diameter retained is the average of these ten values. The average diameter measured is 18.68 ± 3.43 μm. The section of the fibre is considered circular. This area is calculated from the average diameter of the fibre. The surface of the single untreated flax fibres was analysed using a JSM-6360 scanning electron microscope (SEM). To make the fibres conductive, the samples were sputter-coated with a thin layer of gold. The SEM images were obtained at an accelerating voltage of 10 kV, as shown in Fig. 1b–d.

The uniaxial tensile tests on single flax fibres are difficult to implement because of the small dimensions of the fibre. However, the fineness of the flax fibres imposes us to the development of a particular protocol (Fig. 2). This action is motivated by the need to obtain a good behaviour of the load-displacement curves. First, the fibres are separated manually from the bundles, in order to obtain unit fibres, and then are glued on a paper frame 60 mm high and 20 mm wide, having, at its centre, a square hole of 10 mm × 20 mm. The length of the gauge used is therefore 20 mm. The frame is then placed in the mechanically clamping jaws of the testing machine. The mechanical properties (tensile strength, Young's modulus, and ultimate strain) of the flax fibres are determined according to the standard ASTM D 3822-07 [57] using a gauge length (*GL*) of 20 mm. Due to the variability of natural fibres, 120 samples are tested for each batch, totalling more than 480 tests performed. The tests were conducted on a Zwick/Roell universal tensile machine with a capacity of 2.5 kN. The tensile tests were carried out with a speed of 1 mm/min. Young's modulus was calculated in the elastic region at 0.1–0.8% of the deformation value by determining the slope of the tensile strength/strain curve. The mechanical properties namely Young's modulus, the tensile strength and strain at failure were calculated individually for each fibre tested. All tests were carried out at room temperature of 25 °C and relative humidity of approximately 42%.

The statistical analysis such as Weibull analysis (two and three-parameter), one-way ANOVA and Anderson–Darling test and boxplot (notched) were performed in Minitab and Matlab software.

3 Statistical analysis

The Weibull model [58], also called the weakest link theory, is a statistical and empirical model allowing to take into account the random aspect of the data. This approach has already been widely used for fragile materials where the distribution of defects plays a major role in the ruin of the material. The Weibull model is widely used to describe the breaking behaviour of natural or cellulosic fibres such as flax [49, 50], hemp [21], sisal [17], *Agave Americana* [19], palm [18] and jute [13]. However, some studies also used this formulation in the case of glass fibre bundles [59] as well as carbon fibres [60]. Also, for ductile polymer materials, like PET fibres, the choice to represent the distribution of the strength at failure by a model deserves to be justified, even if its use is found in the literature [61, 62]. In order to determine if the sample of the values of the tensile strength follows a Weibull law, a test of 'Kolmogorov-Smirnov' is done [63].

The Laplace–Gauss is the normal distribution and continuous probability law that depends on two factors μ and δ. The first parameter, mean, gives information about the centre of the distribution. The second parameter (δ) is the standard deviation (SD) which gives the information about its spread. The Gaussian (Normal) probability density function (PDF) is defined by Eq. (1) [64, 65]. The reduced centred normal variable is $y = (x - \mu)/\delta$, and for this variable ($\mu = 0$, $\delta = 1$), there is a density of the probability, which is given by Eq. (2). Moreover, the log-normal distribution (*LND*) is defined by ξ (scale parameter) and λ (location parameter), for $\lambda > 0$, and one obtains for the density function (Eq. (3)) [64, 65]. The $ln(x)$ indicates the arithmetic mean of natural logarithms of mechanical properties, and ξ is the standard deviation of the natural logarithms of mechanical properties.

$$f(x|\mu,\delta) = \frac{1}{\sqrt{2\pi}\sigma} e^{\left(-\frac{(x-\mu)}{2\delta}\right)^2} \tag{1}$$

$$\varphi(y) = \frac{1}{\sqrt{2\pi}} e^{-\frac{y^2}{2}} \tag{2}$$

$$f(x|\xi,\lambda) = \frac{1}{x\xi\sqrt{2\pi}} e^{-\frac{1}{2}\left(\frac{\ln(x)-\lambda}{\xi}\right)^2} \tag{3}$$

The probability density function of the three-parameter Weibull distribution, also known as the cumulative distribution function (CDF), is defined by Eq. (4) [58, 66, 67]. Weibull model assumes that fibre fracture is caused by the breakage of its weakest element and can effectively model the mechanical properties of fibres in many cases [12, 51, 66].

$$P(x|s_0,s,m) = 1 - \exp\left[-\left(\frac{x-s_0}{s}\right)^m\right], \quad x \geq s_0 \tag{4}$$

where $s_0 = 0$, Eq. (5) becomes:

$$P(x|s,m) = \frac{m}{s}\left(\frac{x}{s}\right)^{m-1} \exp\left[-\left(\frac{x}{s}\right)^m\right] \tag{5}$$

After simplification of Eq. (5), in this case, the two-parameter Weibull survival probability *P(x)*, assuming that the threshold is zero ($s_0 = 0$), can be described by Eq. (6) [13, 68–72]:

$$P(x|s,m) = 1 - \exp\left[-\left(\frac{x}{s}\right)^m\right] \tag{6}$$

The parameters m, s, and s_0 can be determined using the least squares estimate (LS). After rearrangement and rewriting the previous equation as [73] (Eq. (7)):

$$\ln\left[\ln\left(\frac{1}{1-P}\right)\right] = m \ln(x) - m \ln(s) \tag{7}$$

where x, s, s_0 and m are all positive real ($R > 0$), with s_0, being the threshold (a location parameter) that represents an average value of the parameter x (minimum life), and $s > 0$ represents the scale parameter (characteristic value) and m is the shape parameter or Weibull modulus.

Also, the parameter s_0 in our study represents the characteristic mechanical properties namely σ_0, ε_0 and E_0 which are the characteristic tensile strength and strain at failure and the Young modulus, respectively. The form $\ln\left[\ln\left(\frac{1}{1-P}\right)\right] = f(\ln(x))$ is a linear representation of the data, if the Weibull model is adapted. If we represent this formula, we obtain a line (linear model) of slope m. Then, the ordinate at the origin of this line makes it possible to deduce the parameter s. The difficulty of this method is to obtain an estimate of P (probability of survival). Thus, the value of $P_\mathrm{i}(x)$ of the rank sample i can be calculated using different estimators or the empirical index (probability index). The general formula of the estimator is as follows: $P_\mathrm{i} = \frac{i-\alpha}{n-\beta}$ where $\alpha = 0, 0.3, 0.375, 0.5$ and $\beta = 0, 0.25, 0.4, 1$. In more detail, four estimators are widely used in the literature [60, 74–80]. The first estimator $P_i = \frac{i}{n+1}$ was most used for sample populations greater than 20. However, more recent studies have shown that it skewed the results more than the estimator $P_\mathrm{i} = \frac{i-0.5}{n}$. This second estimator is the most appropriate for sample populations between 20 and 50 and is consequently the estimator that will be used here in our study. However, the three estimator $P_i = \frac{i-0.3}{n+0.4}$ is used in the case of populations of samples smaller than 6, while the estimator four $P_\mathrm{i} = \frac{i-0.375}{n+0.25}$ is used for sample populations less than 10.

Another method used to estimate parameters of the Weibull probability is the ML method of maximum likelihood which is defined by the following formula [81–83]:

$$\mathrm{Likelihood}(m,s|x_1,\ldots,x_2) = \prod_{i=1}^{n} P(x_i) = \prod_{i=1}^{n} \frac{m}{s}\left(\frac{x_i}{s}\right)^{m-1} \exp\left[-\left(\frac{x}{s}\right)^m\right] \tag{8}$$

The *ML* is an efficient estimation technique that has remarkable mathematical properties such as normality. This method consists of determining the parameters that maximize the likelihood of sample populations. That is to say, statistically, this estimation method is favoured for its power. Also, it allows simple construction of the confidence intervals which leads to the automatic determination of the uncertainties of Weibull parameters at a 95% CI.

Minitab software version 16 was used in this study. Minitab uses some commands and function that generate an adjusted statistic, while several options are available to estimate the P_i index and choose the method or statistical approach used (LS where ML).

4 Results and discussions

4.1 Quasi-static tensile behaviour of flax fibre

The mechanical behaviour of a cellulosic fibre is complex. The natural appearance of the material causes large dispersions of the measurements made and makes it difficult to choose values that can be used for sizing. Unlike synthetic reinforcing fibres such as glass fibres, the tensile behaviour of flax fibre is non-linear according to Baley et al. [55, 56].

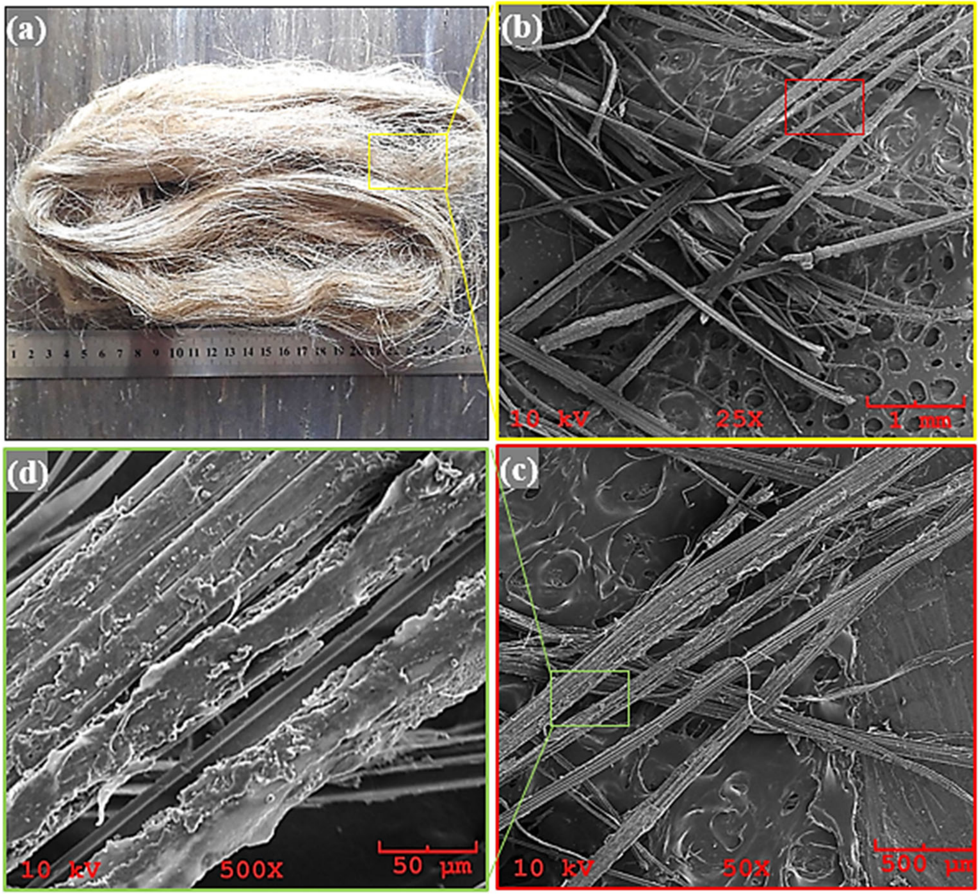


Fig. 1 (**a**) Untreated flax fibres used in this work. (**b**) SEM micrograph of untreated flax fibres. (**c**) SEM micrograph of zoomed details from (**b**). (**d**) SEM micrograph of zoomed details of a single flax fibre from (**c**) topography surface

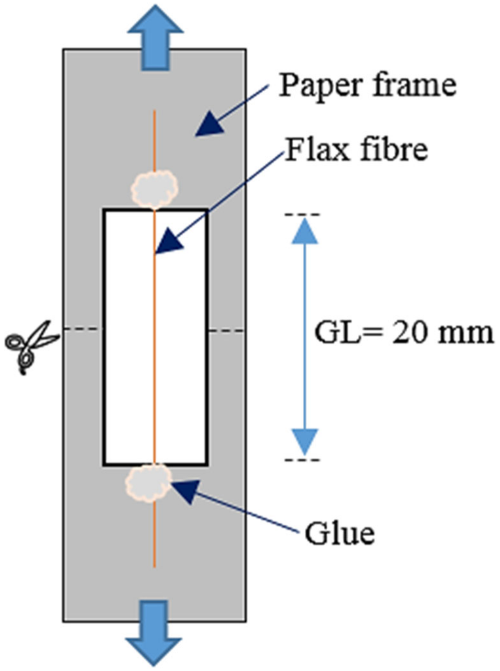
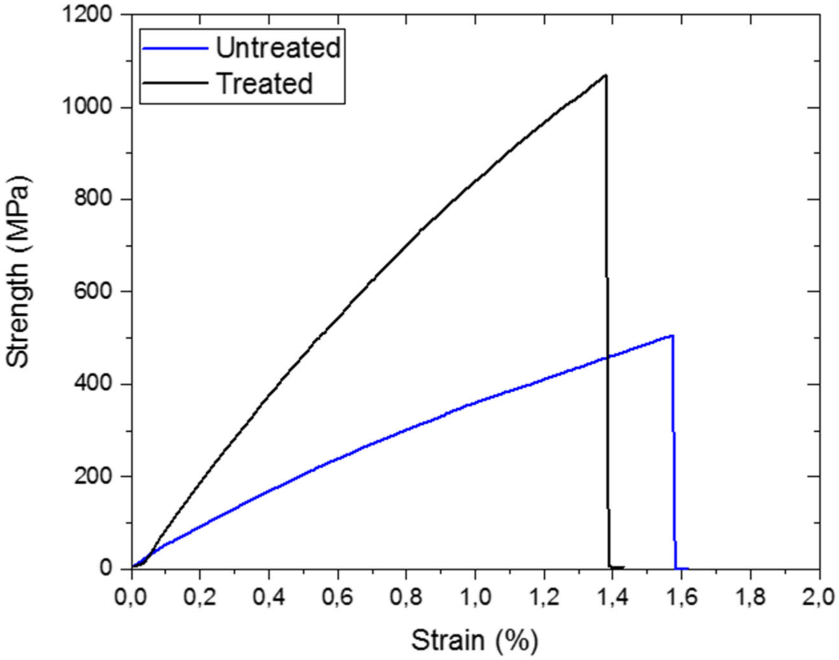


Fig. 2 Schematic arrangement of experimental tensile test of the single flax fibre

Figure 3 shows the typical stress/strain curve of an untreated flax fibre compared with that treated under tensile static loading of single fibre flax. However, it is accepted that cellulosic fibres have behaviour that breaks down into two phases during a tensile test. A first quasi-linear zone situated at the foot of the curve corresponds to the overall loading of the fibre and the free reorientation of the fibril in the tensile direction. Then, the second phase corresponds to a quasi-linear elastic behaviour until rupture followed by a load recovery accompanied by an increase of the linear modulus of elasticity.

Figures 4 and 5 show a scatter plot of the mechanical properties of 4 lots with a total of 480 tests of untreated flax fibre and treated with $NaHCO_3$ at different concentrations with an exponential prediction model at a confidence level of 95% (error 5%). The ratio between the modulus of elasticity (E) and the tensile strength (σ) (Fig. 4a–d), and also Young's modulus versus the strain at failure (ε) (Fig. 5a–d) is that when σ and ε increase, Young's modulus (E) decreases. The results also indicate a significant dispersion of properties. However, despite these dispersions, it is possible to define minimum properties above which the properties of most of the batches are located. The dispersion of the mechanical properties values between different batches could be due to characteristic parameters (fibre varieties, geographical area, and climatic

Fig. 3 Strength vr. strain curve of an untreated flax fibre compared with that treated with $NaHCO_3$



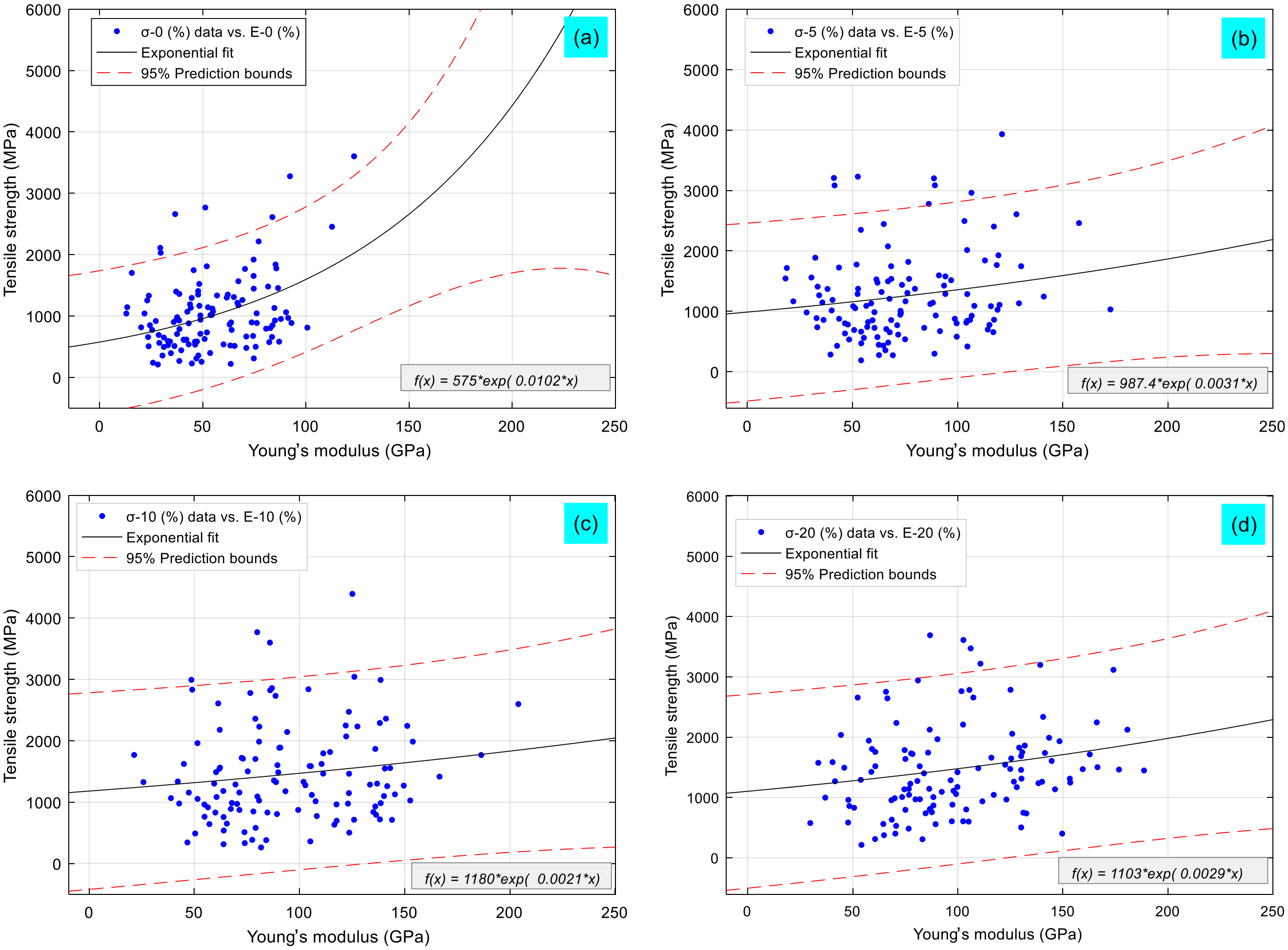


Fig. 4 Tensile strength as function of Young's modulus for all tests of flax fibres (**a**) untreated, (**b**) treated with 5% of $NaHCO_3$, (**c**) treated with 10% of $NaHCO_3$ and (**d**) treated with 20% of $NaHCO_3$

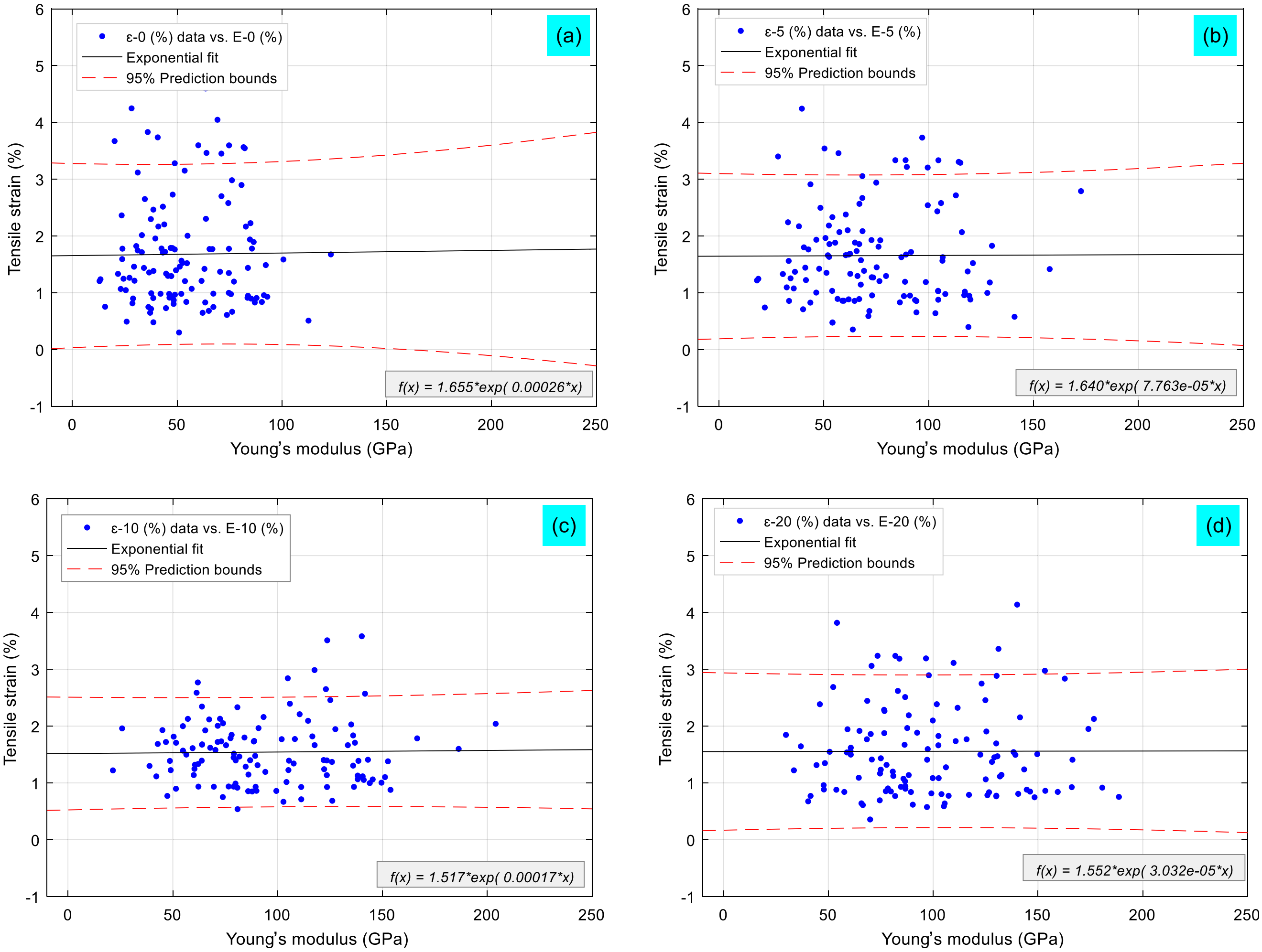


Fig. 5 Tensile strain as function of Young's modulus for all tests of flax fibres (**a**) untreated, (**b**) treated with 5% of $NaHCO_3$, (**c**) treated with 10% of $NaHCO_3$ and (**d**) treated with 20% of $NaHCO_3$

condition). Within the same lot, properties are also scattered. This behaviour is similar to that described by Virk et al. [13, 70] in the case of jute single fibres. While this dispersion can also be attributed to the difficulty of accessing the actual resistant section of the fibres, and the fact that the development of the fibres is spread over time in the course of growth.

Figure 6 shows the relationship between the bicarbonate concentration ($NaHCO_3$) and the mechanical properties of the fibres with bonds of prediction at 5% error (95%). According to Fig. 6a–c, it is noted that the tensile strength versus $NaHCO_3$ concentration follows a power-type trend, i.e. the mechanical characteristics also increase with increasing $NaHCO_3$ concentration for 5% and 10%. However, for 20% of $NaHCO_3$, a slight decrease and stabilisation of properties are recorded. Similarly, the Young's modulus increases gradually with the increase of $NaHCO_3$ (5%, 10% and 20%), whereas the strain at failure tends to decrease with a slight slope.

Figure 7 a–c shows the variation of the average mechanical properties presented in the form of boxes or Tukey diagram (Box plots) for tensile strength and strain at failure and Young's modulus of untreated flax fibres and treated at different concentrations of bicarbonate ($NaHCO_3$). This simple and original form of representation (Fig. 7) allows for illustration and summary of variable data in a simple and visual way such as extreme values: maximum, minimum and quartiles in the form of a rectangle ranging from the first quartile (Q1) to the third quartile (Q3) and cut a central value which is by the median. The ends of whiskers are extreme values. For example, the values of the tensile strength at 5% concentration of bicarbonate ($NaHCO_3$) are 1090, 756, 1540, 190 and 3930 MPa for the median, Q1, Q3, minimum and maximum, respectively. On the other hand, Young's modulus values are 63, 52, 100, 18 and 172 GPa for the same sample group respectively (5% for $NaHCO_3$).

Table 1 summarizes the results values of tensile tests (Young's modulus, tensile strength and strain at failure) on single fibres of untreated and treated with $NaHCO_3$. Also, an analysis using the coefficient of variation (*CoV%*) is performed. Indeed, by definition, *CoV* in percent (%) is defined

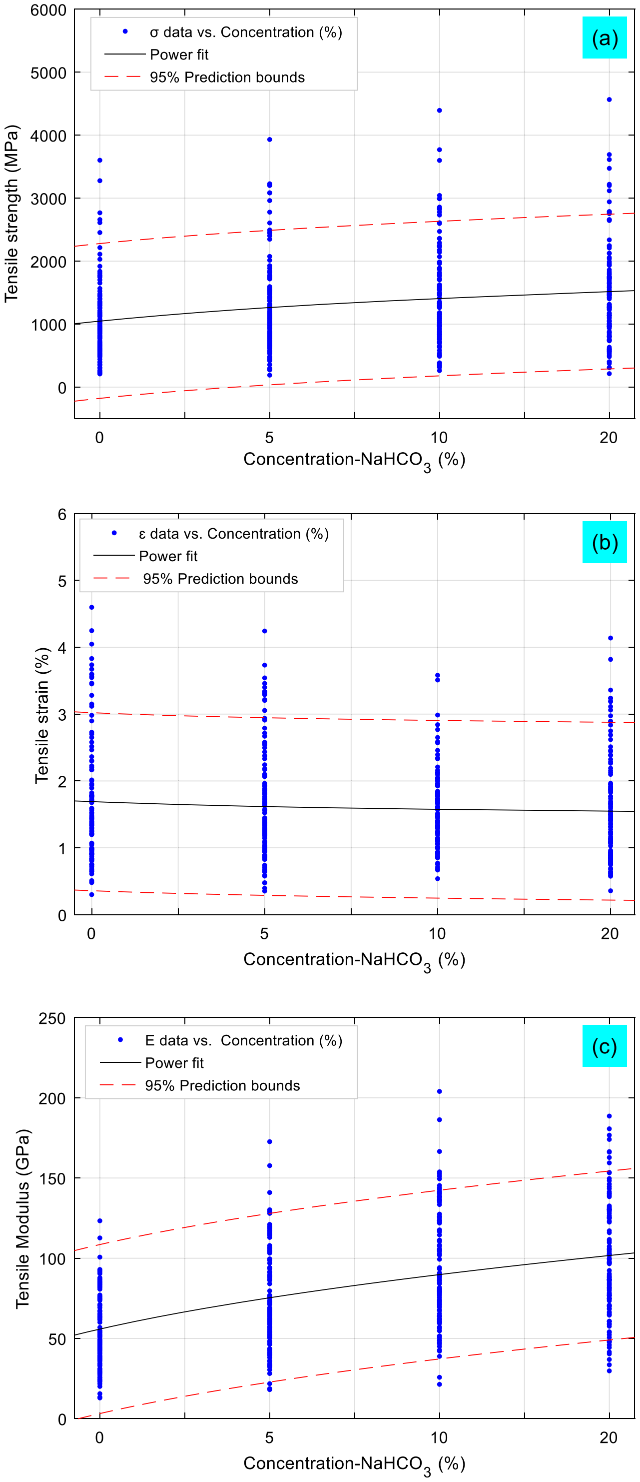


Fig. 6 Variation of tensile mechanical properties as a function of different concentrations of sodium bicarbonate (a) Tensile strength (MPa) vs. $NaHCO_3$ (%), (b) Tensile strain (%) vs. $NaHCO_3$ (%) and (c) Modulus (GPa) vs. $NaHCO_3$ (%)

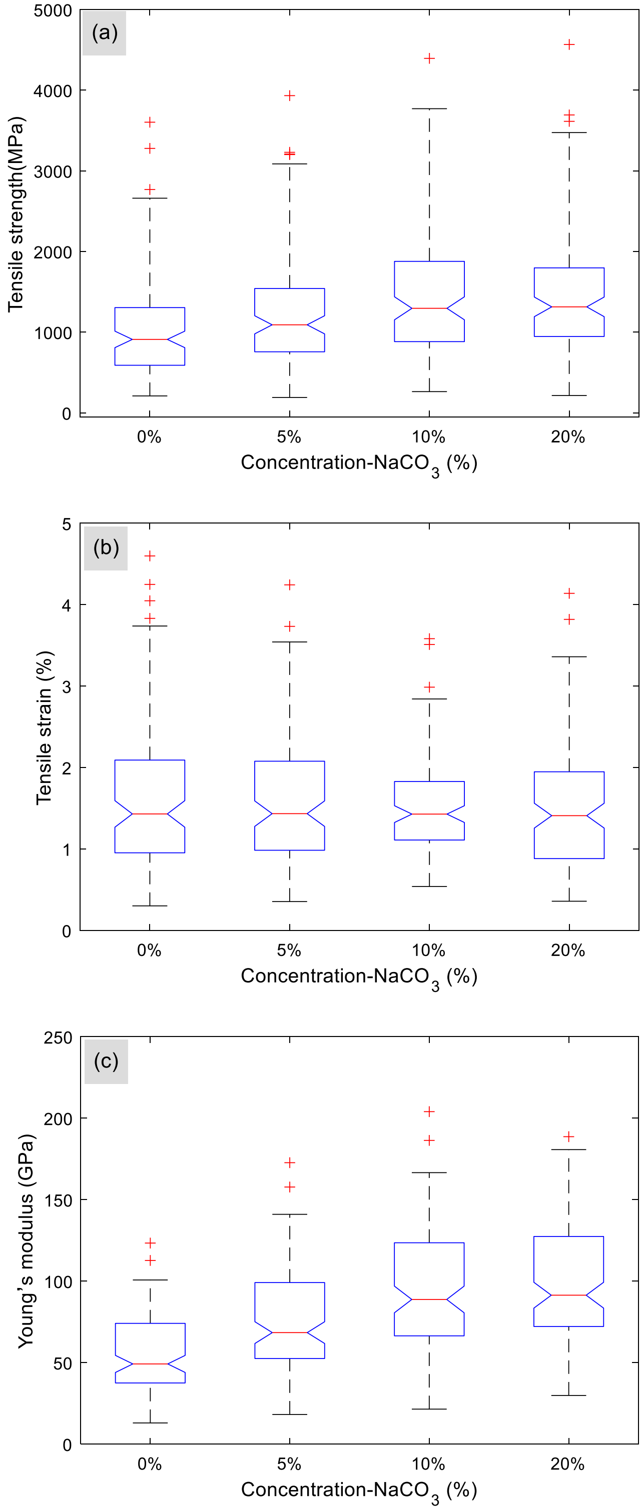


Fig. 7 (**a**) Average tensile strengths, (**b**) strain and (**c**) Young's modulus for untreated, 5, 10 and 20% *w/w* of sodium bicarbonate solution treated flax fibres

by a ratio between the standard deviation (δ) and the mean (μ) given by this formula: $CoV(\%) = [(\delta/\mu) \times 100]$ [80, 84].

At a low value of (CoV), there is a little variation in the results. According to the Table 1 and visualization of Figs. 4, 5, 6 and 7, a strong dispersion of the results of the mechanical properties is obtained during the quasi-static tensile tests of the four batches (0%, 5%, 10% and 20%). For example, CoV obtained for the values of the tensile strength of the four groups of

Table 1 Mechanical properties of treated and untreated flax fibres tested under tensile quasi-static load

Concentrations (%)	Strength (MPa)				Strain (%)				Modulus (GPa)			
	Mean	SD	CoV (%)	Inc (%)	Mean	SD	CoV (%)	Dec (%)	Mean	SD	CoV (%)	Inc (%)
0	1036.96	621.44	59.93	–	1.68	0.95	56.48	–	54.52	22.70	41.63	–
5	1259.56	737.09	58.52	21.46	1.65	0.85	51.40	− 1.78	74.82	29.91	39.98	37.23
10	1459.08	795.53	54.52	40.70	1.54	0.58	37.41	− 8.33	95.00	35.53	37.40	74.24
20	1478.62	807.69	54.62	42.59	1.56	0.80	51.63	− 7.14	98.28	41.08	41.80	80.26

Inc increase, *Dec* decrease

untreated flax fibre and those treated with $NaHCO_3$: 5%, 10% and 20% are equal to 59.93, 58.52, 54.52 and 54.62, respectively, while the Young's modulus is 41.63, 39.98, 37.40 and 41.80.

It can be seen from Table 1 that the effect of sodium bicarbonate treatment is clearly visible and the increase in mechanical properties strongly depends on the concentration of the chemical used ($NaHCO_3$). For example, the untreated fibre has an average tensile strength and Young's modulus of 1036 MPa and 54.52 GPa, respectively. On the other hand, increases in the strength of 21%, 40% and 42% are recorded respectively for treated fibres (Table 1) having the following concentrations 5%, 10% and 20%, while the increases in Young's modulus are 37%, 74% and 80%. However, we note a decrease in the strain at failure respectively − 1.78%, − 8.33% and − 7.14 (5%, 10% and 20% $NaHCO_3$). In terms of average values, the Young's modulus of fibres treated with

Table 2 Mechanical property results available from open literature of treated and untreated flax fibres

Material	Treatment	GL (mm)	Number of tests	Strength (MPa)	Strain (%)	Young's Modulus (GPa)	Refs
Flax	Untreated	10	70	789 ± 276	2.4 ± 1.1	45.2 ± 12.9	[85]
Flax	Thermal treatment at 140 °C	10	70	821 ± 326	2.0 ± 0.5	51.0 ± 16.3	[85]
Flax	Thermal treatment at 190 °C	10	70	754 ± 296	2.0 ± 0.6	49.9 ± 13.4	[85]
Flax	Thermal treatment at 250 °C	10	70	252 ± 178	0.9 ± 0.4	30.9 ± 17.1	[85]
Flax	Untreated	10	30	602.6 ± 198.4	–	–	[49]
Flax	Acetylated at 1 h	10	30	613.6 ± 143.2	–	–	[49]
Flax	Stearic acid treated with 12 h at 105 °C	10	30	591.2 ± 167.4	–	–	[49]
Flax	Untreated	5	30	906.4 ± 246.3	–	–	[49]
Flax	Acetylated at 1 h	5	30	840.6 ± 234.3	–	–	[49]
Flax	Stearic acid treated with 12 h at 105 °C	5	30	863.2 ± 223.7	–	–	[49]
Flax	Untreated	8	74	621 ± 295	1.3 ± 0.6	52 ± 18	[86]
Flax	Enzyme ratted	9	20	591 ± 250	1.4 ± 0.9	57 ± 22	[87]
Flax	Untreated	8	23	1100	–	–	[88]
Flax	Acrylic acid	5	8	1369	–	–	[88]
Flax	Vinyl trimethoxy silane	5	8	1000	–	–	[88]
Flax	Untreated	10	37	1454 ± 835	2.3 ± 0.6	68.2 ± 35.8	[89]
Flax	Untreated	10	–	1339 ± 486	3.27 ± 0.84	54.08 ± 15.12	[55]
Green flax	Untreated	75	19 to 90	305 ± 120	1.3 ± 0.4	31 ± 12	[90]
Green flax	(Treated with polygalacturonase)	75	19 to 90	325 ± 115	1.9 ± 0.9	22 ± 12	[90]
Green flax	Enzymatic treatment (pectate lyase)	75	19 to 90	470 ± 165	1.4 ± 0.5	37 ± 15	[90]
Green flax	Untreated	10	58	670 ± 315	3.5 ± 1.1	36 ± 15	[90]
Green flax	Enzymatic treatment (pectate lyase)	10	38	635 ± 245	3.4 ± 1.1	36 ± 10	[90]
Raw flax	Untreated	2	15 to 20	802	–	46.9	[91]
Raw flax	10% MA (maleic anhydride) for 25 h	2	15 to 20	724	–	34.6	[91]
Raw flax	20% NaOH treated at 1 h	2	15 to 20	572	–	13.5	[91]
Raw flax	Untreated	2	15 to 20	1054	–	33.2	[92]
Raw flax	10% MA (maleic anhydride) for 25 h	2	15 to 20	992	–	35.8	[92]

Table 3 Adjusted Anderson-Darling goodness-of-fit estimates for different distributions methods (2P Weibull, 3P Weibull, Normal and Log-normal distributions)

	Strength (MPa)				Strain (%)				Modulus (GPa)			
	Normal	Weibull	Log-normal	3P-Weibull	Normal	Weibull	Log-normal	3P-Weibull	Normal	Weibull	Log-normal	3P-Weibull
0%	3.433	1.798	0.427	0.623	4.682	4.474	0.611	2.103	1.246	1.109	0.931	0.597
5%	3.834	2.137	0.399	0.996	3.223	2.919	0.446	0.963	1.232	1.112	0.768	0.570
10%	2.324	1.210	0.401	0.406	1.464	2.182	0.410	0.552	1.317	1.381	0.940	0.939
20%	2.408	1.112	0.624	0.578	3.603	4.555	0.915	1.962	0.976	1.169	0.681	0.392

20% $NaHCO_3$ (98 ± 41 GPa) is higher than that without treatment (54 ± 22 GPa), as is the tensile strength of the treated fibres with 20% of $NaHCO_3$ (1478 ± 807 MPa) is higher than that of untreated flax fibre (1036 ± 621 MPa). However, the strain at failure of the 20% treated flax fibres (1.56 ± 0.80%) is slightly lower than the strain at failure of the untreated fibres (1.68 ± 0.95%).

Table 2 shows a comparison in the experimental data with the mechanical properties values of single flax fibres found in the literature [85–92]. Indeed, the comparison of the latter is

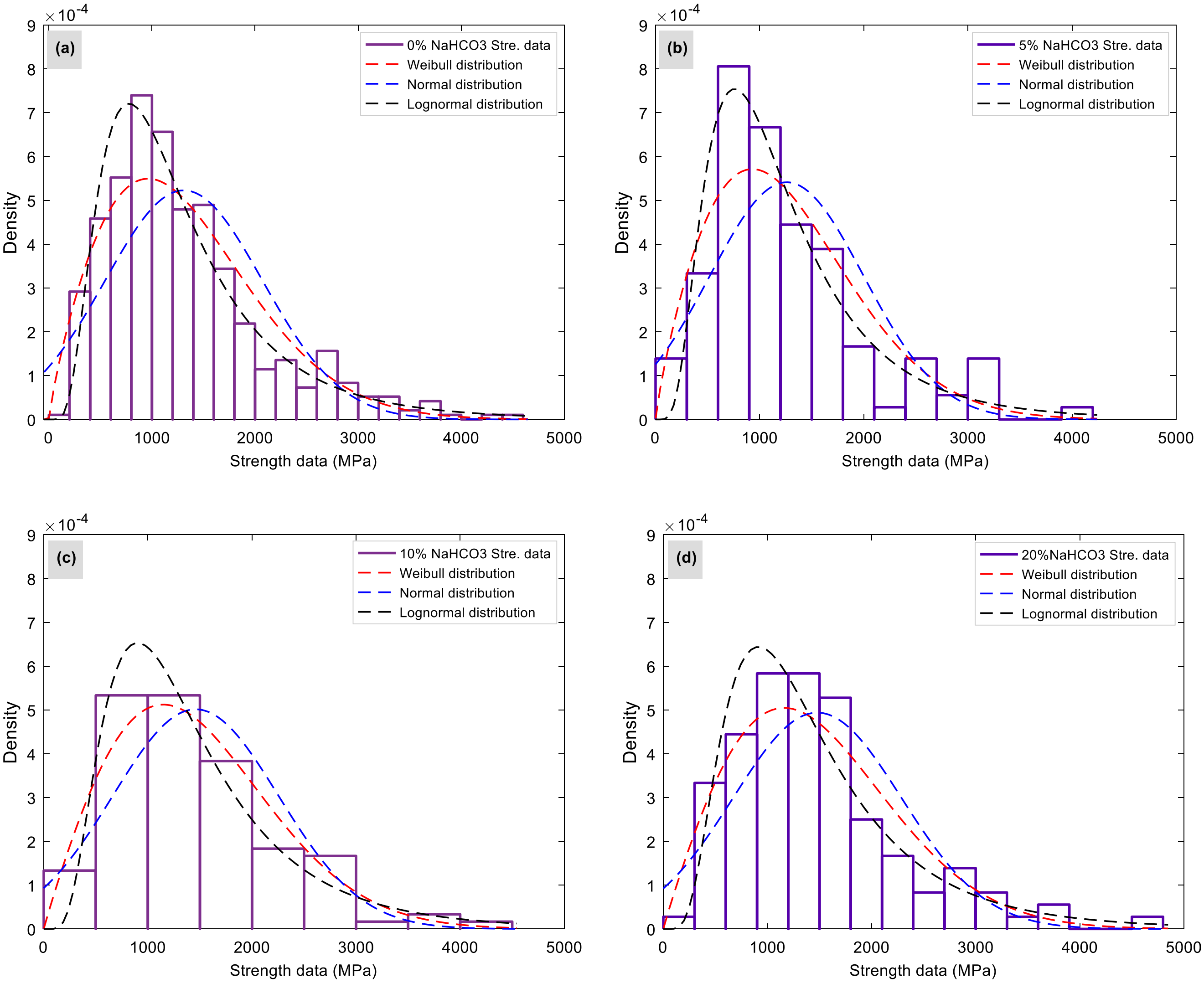


Fig. 8 The histograms of the tensile strength data of the flax fibres at different concentrations with the estimation of density functions Weibull, Normal and Lognormal for (a) 0% $NaHCO_3$, (b) 5% $NaHCO_3$, (c) 10% $NaHCO_3$ and (d) 20% $NaHCO_3$

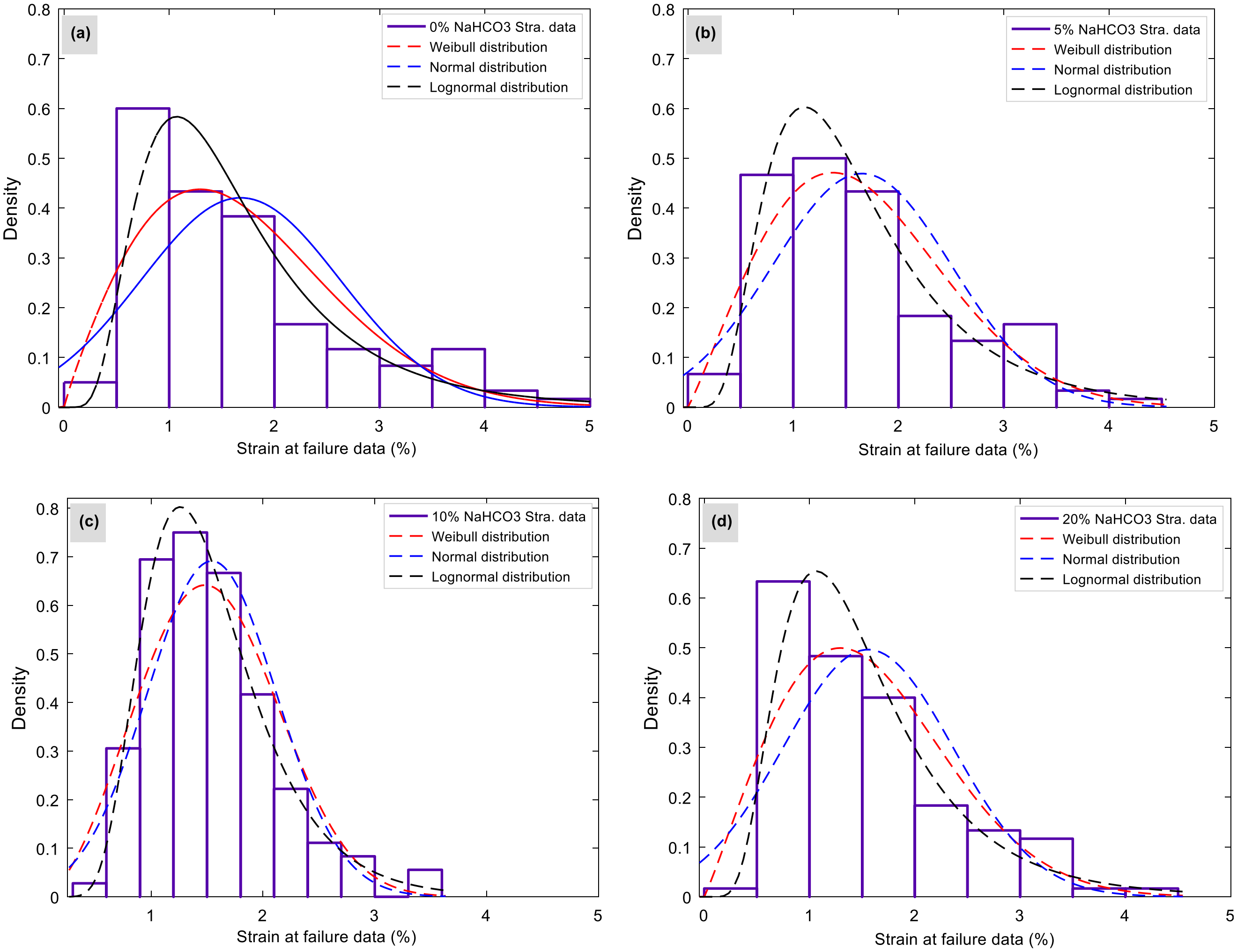


Fig. 9 The histograms of the strain at failure data of the flax fibres at different concentrations with the estimation of density functions Weibull, Normal and Lognormal for (a) 0% $NaHCO_3$, (b) 5% $NaHCO_3$, (c) 10% $NaHCO_3$ and (d) 20% $NaHCO_3$

difficult because of the nature and origin of fibre, their maturity, the climatic conditions of the development of these plants and also that these studies were carried out on different types of machines and test conditions including the solicitation speed [52, 93, 94]. The mechanical properties obtained in this study are in the same range [55, 88, 89, 92], and the dispersions of Young's modulus and tensile strength values are higher compared to literature data [48, 85, 86]. This is perhaps due to the number of tests carried out compared to our study which is 120 samples per batch. For comparison, the tensile strength obtained by our results for flax fibre treated with 5% $NaHCO_3$ is equal to 1259 ± 737 MPa with GL = 20 mm for 120 tests, and this value (1369 MPa) is almost equivalent to that found by Joffe et al. [88] for 8 samples of acrylic acid treated flax fibre with a gauge length of 5 mm, whereas the tensile modulus measured in our case for the same batch is equal to 74.82 ± 29.91 MPa which is significantly higher (2.03 times) than the reference [90] (37 ± 15 GPa) for the same fibre treated with enzymatic treatment (pectate lyase). However, the values of the strain at failure obtained in our study for the four lots (0%, 5%, 10% and 20% at $NaHCO_3$) are significantly lower compared to the results obtained by Alix et al. [90] for 38 samples of fibres treated with pectate lyase for GL = 10 mm in which a value of $\varepsilon = 3.40\%$ is reported [90]. The average Young's modulus for GL = 20 mm of the 120 specimens tested for treated fibres with 20% $NaHCO_3$ considered in this study (98.28 GPa) is clearly superior with a factor of 7.28 times more ($E = 13.5$ MPa) compared with the works of Arbelaiz et al. [91] for 15 to 20 specimens of flax fibre treated with 20% NaOH at a treatment time of 1 h (for GL = 20 mm).

4.1.1 Anderson–Darling goodness of fit for tensile flax fibres data

The four probabilities estimates (function) studied in this section are as follows (Table 3): Normal, 2P-Weibull, Lognormal and 3P-Weibull. The objective is to determine the best fit (goodness of fits) between the four probability distributions proposed. Indeed, using the Minitab software, an A-D test is executed for each estimation to evaluate the critical values of the latter that tends to be the lowest value (Anderson-Darling goodness-of-fit). The A-D test is actually a modification of the K-S test (Kolmogorov-Smirnov). The estimated values of the Anderson-Darling goodness-of-fit are presented in Table 3. This last shows that the data group follows and fits uniformly to the Log-normal distribution for tensile strength and strain at failure data. But, the Young's modulus values follow the 3-Weibull law and this is valid for all the experimental results. Figures 8, 9 and 10 of the Anderson-Darling goodness-of-fit contained in Table 3 confirm this statement. According to the literature [95, 96], Weibull distribution or Log-normal distribution is the best candidate for mechanical property data of natural fibres. As an example, Belaadi et al. [52] confirm that the best adjusted A-D-fit (Anderson-Darling goodness-of-fit) for mechanical properties of sisal yarn is obtained by the 3P-Weibull probability law for tensile strength and strain at failure. In contrast, the Young's modulus follows the adjustment of the distribution of 2P-Weibull.

4.1.2 Weibull distribution

One of the main interests of the two- and three-parameter Weibull model in this study is the prediction of the mechanical properties of untreated and treated flax fibres with sodium bicarbonate at various concentrations that have shown great dispersion. The other interest is that the plant fibres in general and in particular the flax fibres contain intrinsic defects developed during its growth but also defects induced by the various mechanical treatments for its extraction. A statistical approach according to Weibull formalism (Eq. (7)) was carried out on the values of tensile strength and strain at failure and Young's modulus for each of the four varieties studied (0%, 5%, 10% and 20%). We used the statistical approach of Weibull [58] to describe the brittle behaviour of flax fibres. This method of study provides an indicator of the reliability of a material representative of the dispersion of properties for a brittle fracture. This indicator is the Weibull module (*m*).

The statistical distribution curves *LS* (least squares estimates) and Weibull *ML* (maximum likelihood) at two and

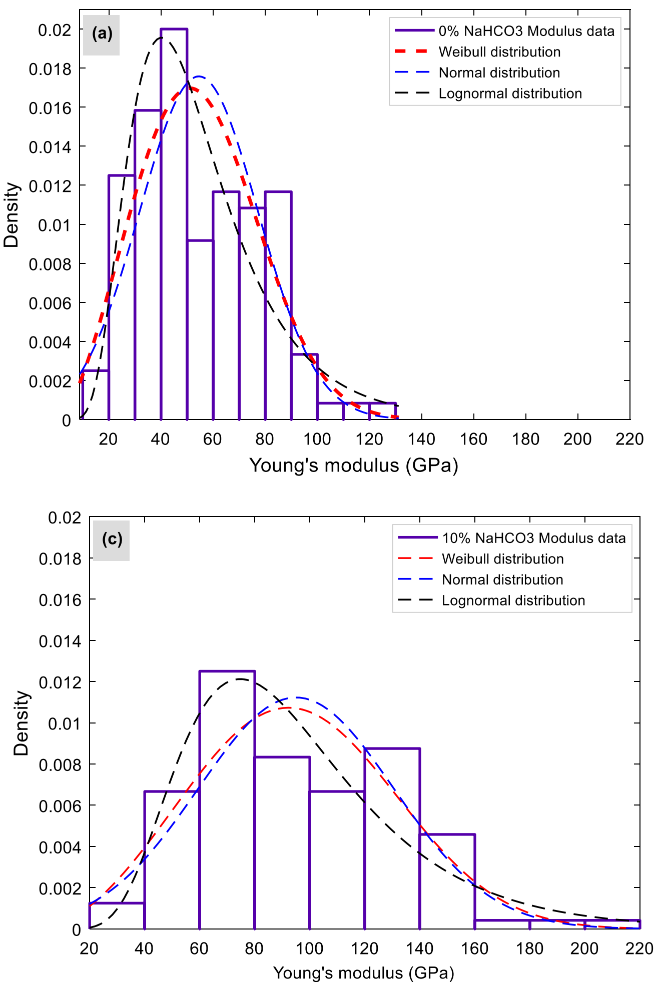


Fig. 10 The histograms of Young's modulus data of the flax fibres at different concentrations with the estimation of density functions Weibull, Normal and Lognormal for (a) 0% $NaHCO_3$, (b) 5% $NaHCO_3$, (c) 10% $NaHCO_3$ and (d) 20% $NaHCO_3$

three parameters obtained for the tensile strength and strain at failure and Young's modulus are presented in Figs. 11 and 12, respectively. The corresponding parameters are grouped in Table 4 using Minitab. However, we perceive that the Weibull straight lines at two and three parameters for the two estimation methods LS and ML (Figs. 11 and 12) of flax fibre treated at different concentrations of $NaHCO_3$ seem to follow a reasonable straight line fit. The adjustments of the experimental values present a quasi-linearity and quasi-superposition with a slight shift of each other. Similar behaviour is observed by other authors such as Belaadi et al. for sisal fibres [52], in the case of sisal single fibres [17] and also in the works of Bezazi et al. [19] in the case of *agave Americana* fibre. Table 4 also shows that the analysis of 2P-Weibull (for LS) gives a correlation factor R^2 which varies between 0.981 and 0.990, 0.958 and 0.974, and 0.984 and 0.991 for the tensile strength and strain at failure and Young's modulus, respectively. According to many authors [12, 13, 49, 50], the values of R^2 are indicators of the alignment, the adjustment and the variation of the Weibull module.

The Weibull parameters are indicators of the microstructure of a material and more particularly of the distribution of

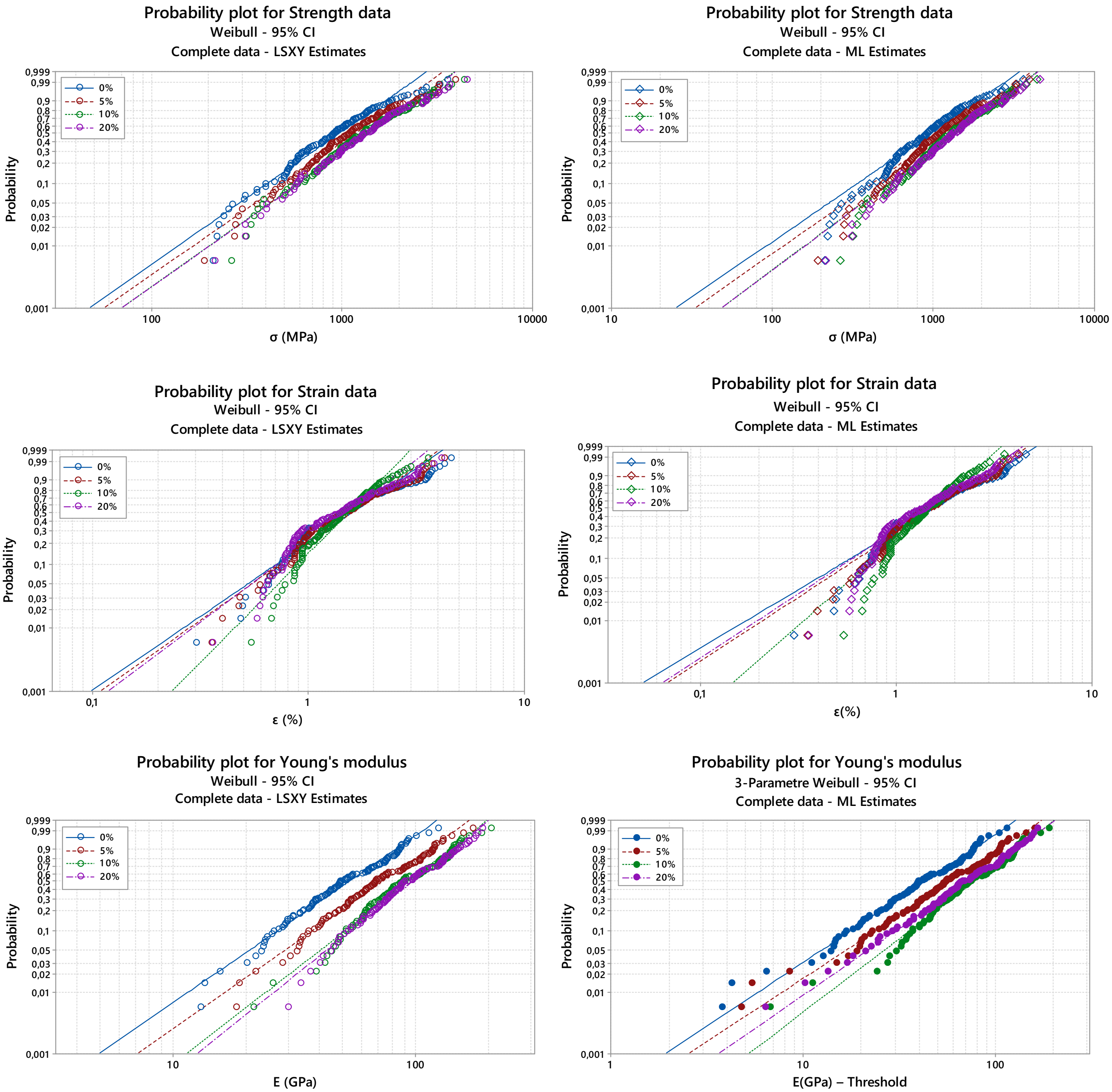


Fig. 11 Tow-parameter Weibull distribution for mechanical properties of the untreated and treated with different concentrations of $NaHCO_3$ of the flax fibres for *LS* and *ML* methods

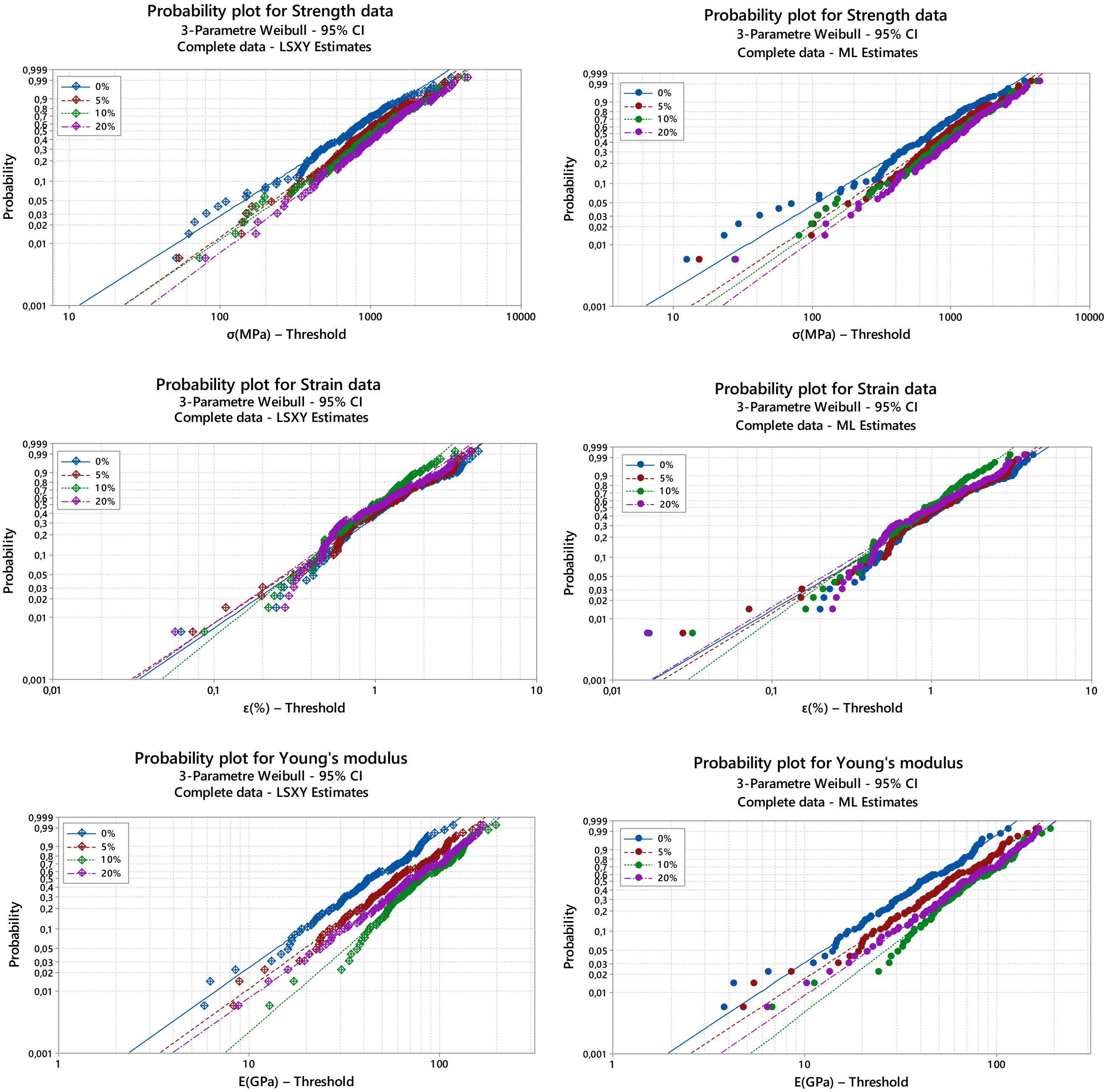


Fig. 12 Three-parameter Weibull distribution for mechanical properties of the untreated and treated with different concentrations of $NaHCO_3$ of the flax fibres for *LS* and *ML* methods

defects and local toughness. Also, the parameters of the Weibull model are characteristic values of the distribution for each property. The Weibull modulus (m) is more particularly related to the rupture of the largest defects. The factor m for 2P-Weibull by the *LS* method of the tensile strength and strain at failure m_σ, m_ε and Young's modulus m_E for different $NaHCO_3$ concentrations of the flax fibres are $m_\sigma = 2.23, 2.14, 2.19, 2.28$; $m_\varepsilon = 2.35, 2.44, 2.47, 2.58$; and $m_E = 2.76, 2.81, 3.11, 3.20$, respectively. According to Silva et al. [53], the Weibull model can also be used to give an approximation of the distribution of defects. A model of microstructure makes it possible to describe the population of defects which are at the origin of the quasi-static rupture. The authors also claim that the Weibull modulus for a single fibre of sisal tested under tensile static loading is significantly affected by the gauge length (GL). It should be noted that Weibull modulus in this study (m_σ = 2.28 for $NaHCO_3$ = 20%) has low value compared to single carbon fibres (m_σ = 6.7), while quasi-similar results of m_σ were found by Belaadi et al. [17] and Amroun et al. [97] in the case of sisal and date palm fibres, respectively. It is important to note that there is a slight dispersion between the LS and ML estimation methods of Weibull 2-parameter and 3-parameter (Table 4). By way of example, the maximum values of the Weibull modulus for a given bicarbonate concentration ($NaHCO_3$ = 20%) are respectively $m_\sigma = 2.19$, $m_\varepsilon = 3.47$ and $m_E = 3.20$, obtained with the *LS* method, while the minimum values are 1.95, 2.09 and 2.90 obtained by the ML method. Table 4 also shows, for comparative purposes, if we take the 2P-Weibull modulus (for ML method) of the tensile strength (m_σ) and the characteristic strength of Weibull (σ_0) of untreated flax fibre which is equal to 2.23 and 11,437 MPa, respectively, whereas, for 3P-Weibull analysis, we find lower values than Weibull 2-parameter ($m_\sigma = 1.39$ and $\sigma_0 = 920.25$ MPa). It can be seen that there is a slight dispersion between the two methods used (LS and ML). Moreover, the two-parameter Weibull analysis combined with the least square (LS) method is the most appropriate for the estimation of the values of the mechanical properties, in particular for the untreated and treated flax fibres with different concentrations sodium bicarbonate ($NaHCO_3$). For comparison, the statistical values of mechanical properties obtained by the distribution of 2P-Weibull (LS) are 1393 MPa, 1.83% and 83.54 GPa (Table 4) compared with those obtained experimentally for the flax fibres treated with 5% of $NaHCO_3$ which are of the order of 1259 MPa, 1.65% and 74.82 GPa (Table 1).

The graphical representations of the evolution of the survival probability of the various estimation methods (LS and ML) as a function of the mechanical properties namely the tensile strength and strain at failure and Young's modulus are presented in Figs. 13 and 14. We note on these figures the good adequacy of the experimental results. A total of 480 values for the different test campaign (0%, 5%, 10 and 20% of $NaHCO_3$) for each property (σ, ε and E) are used to determine Weibull parameters. The parameters obtained from the global distribution of Weibull will be used for plotting of the survival probability. The estimation error is almost nil thanks to a large number of available values (120 tests). The Weibull parameters are then determined for the four groups (0%, 5%, 10% and 20%) and validated by the *p* value of the test (CI = 95%). For example, the representation of the survival probability of the tensile strength (Fig. 13a) was plotted with the least square method estimate and the index $P_i = i/n + 1$ for four pairs of Weibull parameters for 0%, 5%, 10% and 20% which are as follows: $m_\sigma = 2.23$ and $\sigma_0 = 1143.57$, $m_\sigma =$

Table 4 Two and three-parameter Weibull statistics values for treated and untreated flax fibres

	Strength (MPa)				Strain (%)				Modulus (GPa)			
	m_σ	σ_0	σ_u	R^2	m_ε	ε_0	ε_u	R^2	m_E	E_0	E_u	R^2
Using least square (LS)												
Two-parameter Weibull												
0%	2.23	1143.57		0.982	2.35	1.84		0.960	2.76	60.92		0.990
5%	2.14	1393.19		0.981	2.44	1.83		0.971	2.81	83.54		0.991
10%	2.19	1624.2		0.984	3.47	1.69		0.974	3.11	105.75		0.984
20%	2.28	1648.51		0.990	2.58	1.71		0.958	3.20	109.21		0.991
Three-parameter Weibull												
0%	1.57	970.81	158.22	0.993	1.81	1.58	0.24	0.982	2.21	53.38	7.21	0.995
5%	1.73	1247.15	136.33	0.993	1.77	1.53	0.28	0.992	2.26	73.17	9.93	0.996
10%	1.68	1420.73	189.19	0.996	2.13	1.22	0.45	0.993	2.70	96.68	8.79	0.991
20%	1.83	1505.89	134.47	0.997	1.84	1.38	0.30	0.976	2.24	87.24	21.09	0.998
	Strength (MPa)				Strain (%)				Modulus (GPa)			
	m_σ	σ_0	σ_u	AD	m_ε	ε_0	ε_u	AD	m_E	E_0	E_u	AD
Using maximum likelihood method (ML)												
Two-parameter Weibull												
0%	2.08	1172.68		1.303	1.91	1.90		2.149	2.60	61.50		0.752
5%	1.73	1425.37		1.435	1.90	1.87		1.479	2.61	84.38		0.724
10%	1.95	1652.13		0.741	2.09	1.73		1.300	2.90	106.68		0.965
20%	2.15	1673.70		0.841	1.87	1.76		1.910	2.97	110.29		0.693
Three-parameter Weibull												
0%	1.39	920.25	196.66	0.689	1.55	1.55	0.28	1.215	2.11	51.18	9.18	0.586
5%	1.53	1207.49	174.52	0.815	1.61	1.48	0.32	0.696	2.09	69.36	13.38	0.503
10%	1.57	1362.66	234.57	0.384	1.88	1.16	0.50	0.441	2.42	90.56	14.74	0.791
20%	1.66	1447.29	185.87	0.513	1.58	1.35	0.34	1.026	2.20	84.44	23.50	0.420

Characteristic strength (σ_0). Characteristic strain (ε_0). Characteristic Young's modulus (E_0). Weibull modulus (m_s, m_ε, m_E). Anderson-Darling (AD)

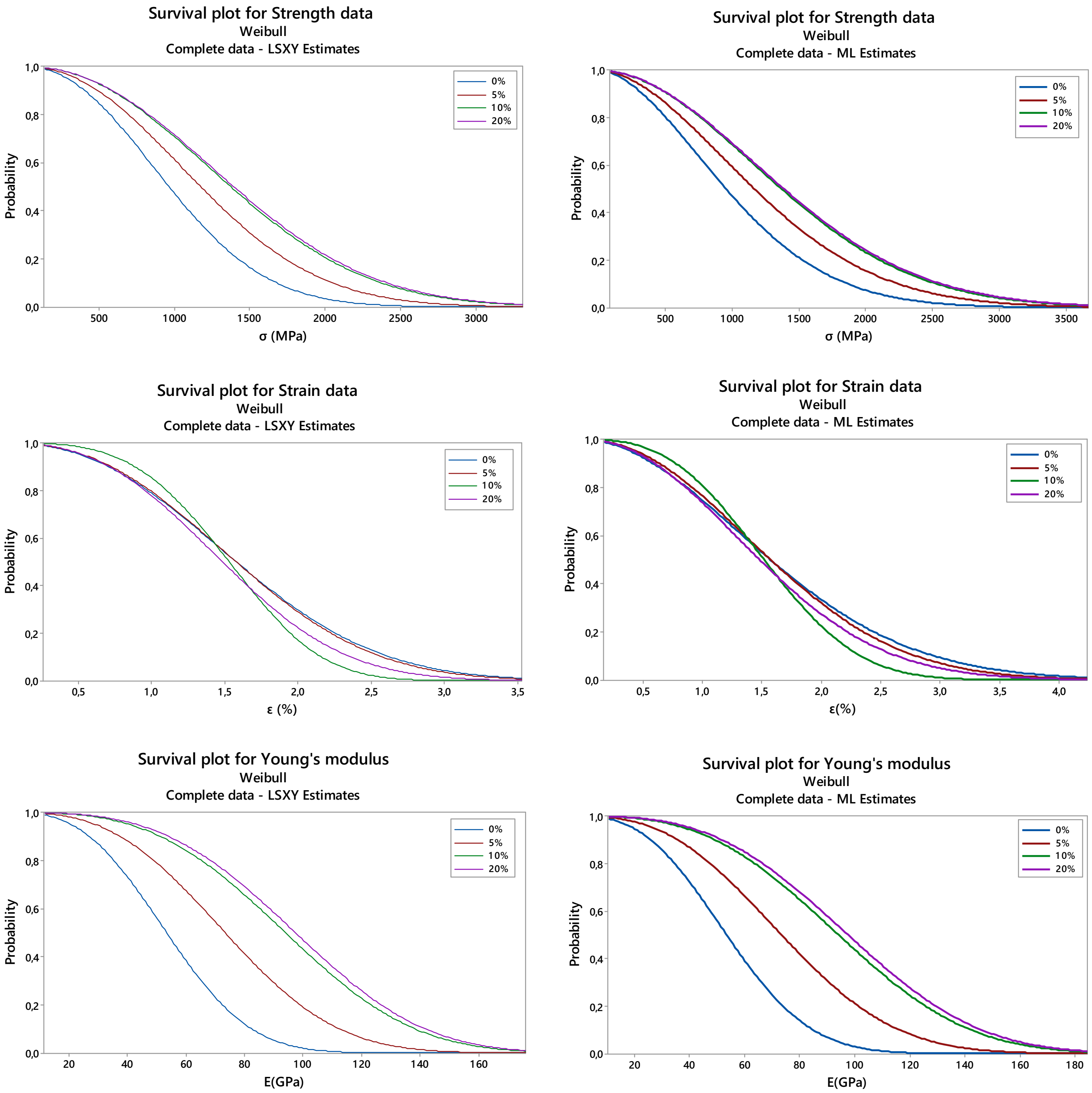


Fig. 13 Probability of survival graphs (with 2P-Weibull) of mechanical properties of the untreated and treated with different concentrations of $NaHCO_3$ of the flax fibres for *LS* and *ML* methods

2.14 and $\sigma_0 = 1393.19$, $m_\sigma = 2.19$ and $\sigma_0 = 1624.2$, and $m_\sigma = 2.28$ and $\sigma_0 = 1648.51$. Similarly for strain at failure and Young's modulus, it should also be noted that the values of the Weibull parameters obtained are close to those available in the literature in the case of cellulosic fibres [17, 19, 53] with a Weibull modulus that ranges from 1.5 to 4.5. Figure 13 a confirms that at 50% ($P(\sigma) = 0.5$) specimen survival for an untreated flax fibre population data, the ultimate tensile strength is estimated at 989 MPa, which coincides with the mean experimental value determined ($\sigma = 1036$ MPa). However, we obtain values of 1.58% and 55 GPa (for $P(\varepsilon) = P(E) = 0.5$) for strain at failure and Young's modulus for untreated flax fibre, respectively.

4.1.3 ANOVA test of variability statistics of tensile properties

The statistical analysis of the data allows the interpretation of the experimental results and to discuss correctly their meaning according to very precise methods. The analysis chosen in this study is a statistical method such as ANOVA analysis of

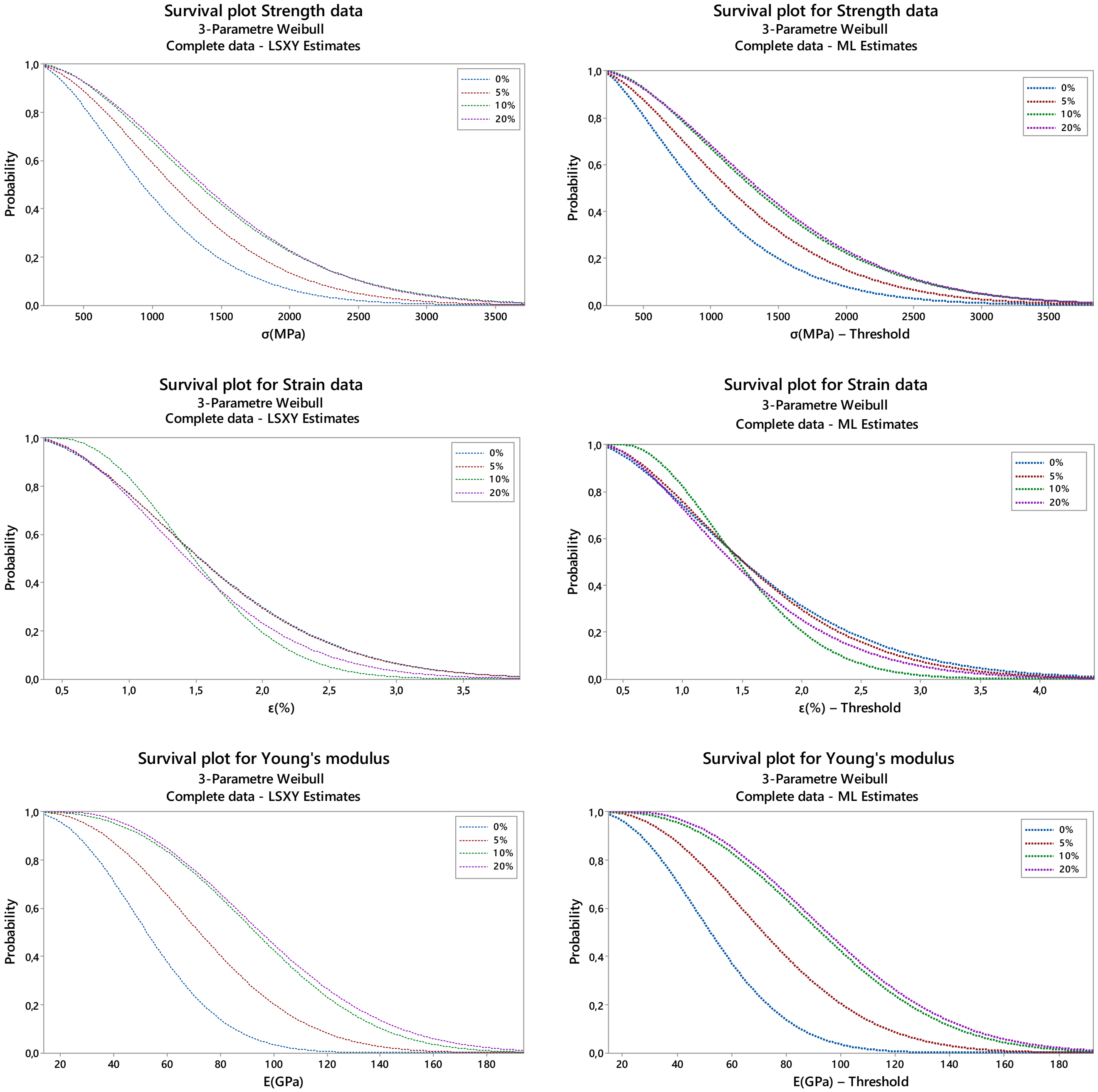


Fig. 14 Probability of survival graphs (with 3P-Weibull) of mechanical properties of the untreated and treated with different concentrations of $NaHCO_3$ of the flax fibres for *LS* and *ML* methods

variance (one-way ANOVA Scheffé post hoc). In order to perform tests of statistical significance, a model is assumed ($y = \alpha i + \beta i$) or the dependent variable y (usually named response variable in ANOVA) which is quantitative, as in the regression but that the independent variable α (or the factor) is qualitative, i is the actual average value of the dependent variable for the i^{eme} population and finally βi is the random error in the non-imputable response to the independent variable. As in the regression, the error is assumed to be normally distributed with a constant variance.

Table 5 groups all the one-way ANOVA test values at a 95% confidence interval (CI) for the groups studied in this work (0%, 5%, 10% and 20%), which represent the mechanical properties namely the tensile strength and strain at failure and Young's modulus. ANOVA analysis of variance can be evaluated according to the following statistical criteria: the *F-Fisher* test, confidence interval (CI), coefficient of determination (*R-sqr*) and standard deviation (SD). In the sample experiment comprising 120 tests on four levels, 0%, 5%, 10% and 20%, which corresponds to $k = 4$ with a sample size $n = 480$,

Table 5 One-way ANOVA test for mechanical properties of the treated and untreated flax fibres

a) ANOVA test for ultimate tensile strength data (120 samples)					
Source	DF	SS	MS	*F*-value	*p* value
BG	3	15329183	5109728	9.23	0.000
WG	476	263551799	553680		
Total	479	278880983			
Factor	Sample	Mean	SD	At 95% CI	$R^2 = 0.945$
0%	120	1037.0	621.4	(903.50; 1170.4)	R^2 adjusted = 0.9510
5%	120	1259.6	737.1	(1126.1; 1393.0)	R^2 predicted = 0.962
10%	120	1459.1	795.5	(1325.6; 1592.6)	
20%	120	1478.6	807.7	(1345.1; 1612.1)	
b) ANOVA test for strain at failure data (120 samples)					
Source	DF	SS	MS	*F*-value	*p* value
BG	3	1.689	0.5630	0.87	0.459
WG	476	309.494	0.6502		
Total	479	311.183			
Factor	Sample	Mean	SD	At 95% CI	$R^2 = 0.893$
0%	120	1.6791	0.9483	(1.5345; 1.8237)	R^2 adjusted = 0.990
5%	120	1.6538	0.8500	(1.5091; 1.7984)	R^2 predicted = 0.992
10%	120	1.5427	0.5772	(1.3980; 1.6873)	
20%	120	1.5565	0.8036	(1.4118; 1.7011)	
c) ANOVA test for Young's modulus (120 samples)					
Source	DF	SS	MS	*F*-value	*p* value
BG	3	148049	49350	48.92	0.000
WG	476	480210	1009		
Total	479	628258			
Factor	Sample	Mean	SD	At 95% CI	$R^2 = 0.839$
0%	120	54.52	22.70	(48.82; 60.21)	R^2 adjusted = 0.842
5%	120	74.82	30.91	(69.13; 80.52)	R^2 predicted = 0.884
10%	120	95.00	35.53	(89.31; 100.70)	
20%	120	98.28	36.08	(92.58; 103.98)	

and until that, we depend on the degree of freedom that is n-1 = 479, whereas the explanatory variations and residues are respectively $BG = k\text{-}1 = 3$ and $WG = n\text{-}k = 476$ and total deviations total = n-1 = 479.

The values of the sum (SS) and the averages (MS) of the squares between the values of the deviations are intended to estimate the parameters of the mechanical properties. As an example, we take the values of Young's modulus. In this case, we consider the value $F = MS_{\mathrm{BG}} / MS_{\mathrm{WG}} = 48.92$ with p value = 0.000 ($p < 0.001$) in these results, and the null hypothesis states that the average values of four different factors (0%, 5%, 10% and 20%) are equal. Since the p value is less than the significance level of 0.05, we can reject the null hypothesis and conclude that some factors have different averages. In the results obtained with Tukey's method (at 95% CI), the confidence intervals in Table 5c indicate that for the difference between two factors of concentration (0% and 20%), there is a range from 92.58 to 103.98. This range does not include a value of zero, which indicates that the difference is statistically significant. R^2 represents the percentage change in the model response. The high R^2 indicates that the model satisfies the hypotheses. The predicted R^2_{pr} shows the ability of our model to predict the response of new observations. The models with high R^2_{pr} values have better predictive ability. However, in the case of the values of the tensile strength presented in Table 5a, we have $R^2_{pr} = 0.962$ and $R^2 = 0.945$ that is to say that $R^2_{pr} > R^2$, and this is valid for other properties (strain at failure (Table 5b) and Young's modulus (Table 5c). A value of slightly higher expected R^2_{pr} than R^2 can be a sign of a good fit of the model.

5 Conclusion

This work is devoted to the characterization of vegetal flax fibre. The choice of chemical treatment requires the definition of the concentration and the chemical structure of the coupling agents, as well as the consideration of the shape, size and species of the cellulosic fibre. This study allows highlighting the influence of chemical treatment with sodium bicarbonate

($NaHCO_3$) at different concentrations (5%, 10% and 20%) on mechanical performance such as tensile strength and strain at break and Young's modulus. The obtained experimental results show that the surface treatment of flax fibres with a 20% solution of $NaHCO_3$ concentration is the best treatment for this type of fibre. In other words, the chemical treatment of the fibres with $NaHCO_3$ for a duration of 120 h allows an increase of 43% and 81% in the tensile strength and Young's modulus respectively compared to the untreated fibres. Finally, the obtained experimental results on a single flax fibre, under tensile quasi-static loading, are analysed by the Weibull distribution with two and three parameters using least-squares and maximum likelihood methods. The values of the Weibull modulus (2P-Weibull-*LS*) of the untreated flax fibre tested at a gauge length (*GL*) of 20 mm for the strength and Young modulus are equal to 2.23 and 2.76, respectively. However, slightly higher values ($m_\sigma = 2.28$ and $m_E = 3.20$) are found for the treated fibre at a concentration of 20% $NaHCO_3$. ANOVA one-way analysis of variance has also been used which concluded that the mechanical properties of flax fibres are influenced by different concentrations of sodium bicarbonate.

References

1. David-West OS, Banks WM, Pethrick RA (2011) A study of the effect of strain rate and temperature on the characteristics of quasi-unidirectional natural fibre reinforced composites. Proc Inst Mech Eng Part L J Mater Des Appl 225:133–148. https://doi.org/10.1177/0954420711404635
2. Joshi SV, Drzal LT, Mohanty AK, Arora S (2004) Are natural fiber composites environmentally superior to glass fiber reinforced composites? Compos Part A Appl Sci Manuf 35:371–376. https://doi.org/10.1016/j.compositesa.2003.09.016
3. Sanjay MR, Madhu P, Jawaid M, Senthamaraikannan P, Senthil S, Pradeep S. Characterization and properties of natural fiber polymer composites: a comprehensive review. vol. 172. Elsevier B.V.; 2018. doi:https://doi.org/10.1016/j.jclepro.2017.10.101.
4. Väisänen T, Das O, Tomppo L (2017) A review on new bio-based constituents for natural fiber-polymer composites. J Clean Prod 149:582–596. https://doi.org/10.1016/j.jclepro.2017.02.132
5. Wang KF, Wang BL (2018) A mechanical degradation model for bidirectional natural fiber reinforced composites under hydrothermal ageing and applying in buckling and vibration analysis. Compos Struct 206:594–600. https://doi.org/10.1016/j.compstruct.2018.08.063
6. De Rosa IM, Kenny JM, Puglia D, Santulli C, Sarasini F (2010) Morphological, thermal and mechanical characterization of okra (Abelmoschus esculentus) fibres as potential reinforcement in polymer composites. Compos Sci Technol 70:116–122. https://doi.org/10.1016/j.compscitech.2009.09.013
7. Resende LM, Franca AS, Oliveira LS (2019) Buriti (Mauritia flexuosa L. f.) fruit by-products flours: Evaluation as source of dietary fibers and natural antioxidants. Food Chem 270:53–60. https://doi.org/10.1016/j.foodchem.2018.07.079
8. Dutta S, Kim NK, Das R, Bhattacharyya D (2019) Effects of sample orientation on the fire reaction properties of natural fibre composites. Compos Part B Eng 157:195–206. https://doi.org/10.1016/j.compositesb.2018.08.118
9. De Rosa IM, Kenny JM, Puglia D, Santulli C, Sarasini F (2010) Tensile behavior of New Zealand flax (Phormium tenax) fibers. J Reinf Plast Compos 29:3450–3454. https://doi.org/10.1177/0731684410372264
10. Asgarinia S, Viriyasuthee C, Phillips S, Dubé M, Baets J, Van Vuure A et al (2015) Tension-tension fatigue behaviour of woven flax/epoxy composites. J Reinf Plast Compos 34:857–867. https://doi.org/10.1177/0731684415581527
11. Baets J, Plastria D, Ivens J, Verpoest I (2014) Determination of the optimal flax fibre preparation for use in unidirectional flax-epoxy composites. J Reinf Plast Compos 33:493–502. https://doi.org/10.1177/0731684413518620
12. Andersons J, SparniņŠ E, Poriķe E (2009) Strength and damage of elementary flax fibers extracted from tow and long line flax. J Compos Mater 43:2653–2664. https://doi.org/10.1177/0021998309345035
13. Virk AS, Hall W, Summerscales J (2009) Multiple Data Set (MDS) weak-link scaling analysis of jute fibres. Compos Part A Appl Sci Manuf 40:1764–1771. https://doi.org/10.1016/j.compositesa.2009.08.022
14. Dobah Y, Bourchak M, Bezazi A, Belaadi A, Scarpa F (2016) Multi-axial mechanical characterization of jute fiber/polyester composite materials. Compos Part B Eng 90:450–456. https://doi.org/10.1016/j.compositesb.2015.10.030
15. Belaadi A, Bezazi A, Bourchak M, Scarpa F (2013) Tensile static and fatigue behaviour of sisal fibres. Mater Des 46:76–83. https://doi.org/10.1016/j.matdes.2012.09.048
16. Belaadi A, Bezazi A, Maache M, Scarpa F (2014) Fatigue in sisal fiber reinforced polyester composites: Hysteresis and energy dissipation. Procedia Eng 74:325–328. https://doi.org/10.1016/j.proeng.2014.06.272
17. Belaadi A, Bezazi A, Bourchak M, Scarpa F, Zhu C (2014) Thermochemical and statistical mechanical properties of natural sisal fibres. Compos Part B Eng 67:481–489. https://doi.org/10.1016/j.compositesb.2014.07.029
18. Amroune S, Bezazi A, Belaadi A, Zhu C, Scarpa F, Rahatekar S et al (2015) Tensile mechanical properties and surface chemical sensitivity of technical fibres from date palm fruit branches (Phoenix dactylifera L.). Compos Part A Appl Sci Manuf 71:95–106. https://doi.org/10.1016/j.compositesa.2014.12.011
19. Bezazi A, Belaadi A, Bourchak M, Scarpa F, Boba K (2014) Novel extraction techniques, chemical and mechanical characterisation of Agave americana L. natural fibres. Compos Part B Eng 66:194–203. https://doi.org/10.1016/j.compositesb.2014.05.014
20. Fiore V, Valenza A, Di Bella G (2011) Artichoke (Cynara cardunculus L.) fibres as potential reinforcement of composite structures. Compos Sci Technol 71:1138–1144. https://doi.org/10.1016/j.compscitech.2011.04.003
21. Placet V (2009) Characterization of the thermo-mechanical behaviour of Hemp fibres intended for the manufacturing of high performance composites. Compos Part A Appl Sci Manuf 40:1111–1118. https://doi.org/10.1016/j.compositesa.2009.04.031
22. Reddy KO, Maheswari CU, Dhlamini MS, Mothudi BM, Kommula VP, Zhang J, Zhang J, Rajulu AV (2018) Extraction and characterization of cellulose single fibers from native African napier grass. Carbohydr Polym 188:85–91. https://doi.org/10.1016/j.carbpol.2018.01.110
23. Manimaran P, Senthamaraikannan P, Murugananthan K, Sanjay MR (2018) Physicochemical properties of new cellulosic fibers from Azadirachta indica Plant. J Nat Fibers 15:29–38. https://doi.org/10.1080/15440478.2017.1302388
24. Sandeep NC, Raghavendra Rao H, Hemachandra RK (2017) Extraction and characterization of physicochemical and tensile

properties of Aegle Marmelos fiber. Mater Today Proc 4:3158–3165. https://doi.org/10.1016/j.matpr.2017.02.200

25. Van De Velde K, Kiekens P (1999) Wettability of natural fibres used as reinforcement for composites. Angew Makromol Chem 272:87–93. https://doi.org/10.1002/(SICI)1522-9505(19991201)272:1<87::AID-APMC87>3.0.CO;2-Q
26. Bledzki AK, Gassan J (1999) Composites-reinforced-with-cellulose-based-fibres_1999_Progress-in-Polymer-Science.pdf. Prog Polym Sci 24:221–274. https://doi.org/10.1016/S0079-6700(98)00018-5
27. Fiore V, Scalici T, Nicoletti F, Vitale G, Prestipino M, Valenza A (2016) A new eco-friendly chemical treatment of natural fibres: effect of sodium bicarbonate on properties of sisal fibre and its epoxy composites. Compos Part B Eng 85:150–160. https://doi.org/10.1016/j.compositesb.2015.09.028
28. dos Santos JC, Siqueira RL, Vieira LMG, Freire RTS, Mano V, Panzera TH (2018) Effects of sodium carbonate on the performance of epoxy and polyester coir-reinforced composites. Polym Test 67:533–544. https://doi.org/10.1016/j.polymertesting.2018.03.043
29. Chaitanya S, Singh I (2018) Sisal fiber-reinforced green composites: effect of ecofriendly fiber treatment. Polym Compos 12:4310–4321. https://doi.org/10.1002/pc.24511
30. Fiore V, Scalici T, Valenza A (2018) Effect of sodium bicarbonate treatment on mechanical properties of flax-reinforced epoxy composite materials. J Compos Mater 52:1061–1072. https://doi.org/10.1177/0021998317720009
31. Baley C, Le Duigou A, Bourmaud A, Davies P (2012) Influence of drying on the mechanical behaviour of flax fibres and their unidirectional composites. Compos Part A Appl Sci Manuf 43:1226–1233. https://doi.org/10.1016/j.compositesa.2012.03.005
32. Lefeuvre A, Bourmaud A, Baley C (2015) Optimization of the mechanical performance of UD flax/epoxy composites by selection of fibres along the stem. Compos Part A Appl Sci Manuf 77:204–208. https://doi.org/10.1016/j.compositesa.2015.07.009
33. Bourmaud A, Ausias G, Lebrun G, Tachon ML, Baley C (2013) Observation of the structure of a composite polypropylene/flax and damage mechanisms under stress. Ind Crop Prod 43:225–236. https://doi.org/10.1016/j.indcrop.2012.07.030
34. Baley C, Bourmaud A (2014) Average tensile properties of French elementary flax fibers. Mater Lett 122:159–161. https://doi.org/10.1016/j.matlet.2014.02.030
35. Baley C, Davies P, Grohens Y, Dolto G (2004) Application of interlaminar tests to marine composites. A literature review. Appl Compos Mater 11:99–126. https://doi.org/10.1023/B:ACMA.0000012902.93986.bf
36. Gassan J, Bledzki AK (1999) Possibilities for improving the mechanical properties of jute/epoxy composites by alkali treatment of fibres. Compos Sci Technol 59:1303–1309. https://doi.org/10.1016/S0266-3538(98)00169-9
37. Azwa ZN, Yousif BF, Manalo AC, Karunasena W (2013) A review on the degradability of polymeric composites based on natural fibres. Mater Des 47:424–442. https://doi.org/10.1016/j.matdes.2012.11.025
38. Liang S, Guillaumat L, Gning PB (2015) Impact behaviour of flax/epoxy composite plates. Int J Impact Eng 80:56–64. https://doi.org/10.1016/j.ijimpeng.2015.01.006
39. Marson GA, Frollini E, Arantes GM, El Seoud OA, Regiani AM (2002) Some aspects of acylation of cellulose under homogeneous solution conditions. J Polym Sci Part A Polym Chem:1357–1363. https://doi.org/10.1002/(sici)1099-0518(19990501)37:9<1357::aid-pola16>3.0.co;2-y
40. Kalia S, Kaith BS, Kaur I (2009) Pretreatments of natural fibers and their application as reinforcing material in polymer composites-a review. Polym Eng Sci. https://doi.org/10.1002/pen.21328
41. Sarkar BK, Ray D, Bose NR, Rana AK (2001) Effect of alkali treated jute fibres on composite properties. Bull Mater Sci 24:129–135. https://doi.org/10.1007/BF02710089
42. Zafeiropoulos NE, Baillie CA, Matthews FL (2001) Study of transcrystallinity and its effect on the interface in flax fibre reinforced composite materials. Compos Part A Appl Sci Manuf 32:525–543. https://doi.org/10.1016/S1359-835X(00)00058-0
43. John MJ, Anandjiwala RD (2008) Recent developments in chemical modification and characterization of natural fiber-reinforced composites. Polym Compos. https://doi.org/10.1002/pc.20461
44. Célino A, Fréour S, Jacquemin F, Casari P (2014) The hygroscopic behavior of plant fibers: a review. Front Chem. https://doi.org/10.3389/fchem.2013.00043
45. Obi Reddy K, Uma Maheswari C, Shukla M, Song JI, Varada RA (2013) Tensile and structural characterization of alkali treated Borassus fruit fine fibers. Compos Part B Eng 44:433–438. https://doi.org/10.1016/j.compositesb.2012.04.075
46. Alawar A, Hamed AM, Al-Kaabi K (2009) Characterization of treated date palm tree fiber as composite reinforcement. Compos Part B Eng 40:601–606. https://doi.org/10.1016/j.compositesb.2009.04.018
47. Lu N, Oza S (2013) Thermal stability and thermo-mechanical properties of hemp-high density polyethylene composites: effect of two different chemical modifications. Compos Part B Eng 44:484–490. https://doi.org/10.1016/j.compositesb.2012.03.024
48. Venkateshwaran N, Elaya Perumal A, Arunsundaranayagam D. Fiber surface treatment and its effect on mechanical and visco-elastic behaviour of banana/epoxy composite. vol. 47. Elsevier Ltd; 2013. doi:10.1016/j.matdes.2012.12.001.
49. Zafeiropoulos NE, Baillie CA (2007) A study of the effect of surface treatments on the tensile strength of flax fibres: part II. Application of Weibull statistics. Compos Part A Appl Sci Manuf 38:629–638. https://doi.org/10.1016/j.compositesa.2006.02.005
50. Zafeiropoulos NE, Dijon GG, Baillie CA (2007) A study of the effect of surface treatments on the tensile strength of flax fibres: part I. Application of Gaussian statistics. Compos Part A Appl Sci Manuf. https://doi.org/10.1016/j.compositesa.2006.02.004.
51. Kompella MK, Lambros J (2002) Micromechanical characterization of cellulose fibers. Polym Test 21:523–530. https://doi.org/10.1016/S0142-9418(01)00119-2
52. Belaadi A, Bourchak M, Aouici H (2016) Mechanical properties of vegetal yarn: statistical approach. Compos Part B Eng 106:139–153. https://doi.org/10.1016/j.compositesb.2016.09.033
53. Silva F de A, Chawla N, Filho RD de T. Tensile behavior of high performance natural (sisal) fibers. Compos Sci Technol 2008;68:3438–3443. doi:https://doi.org/10.1016/j.compscitech.2008.10.001.
54. Charlet K, Baley C, Morvan C, Jernot JP, Gomina M, Bréard J (2007) Characteristics of Hermès flax fibres as a function of their location in the stem and properties of the derived unidirectional composites. Compos Part A Appl Sci Manuf 38:1912–1921. https://doi.org/10.1016/j.compositesa.2007.03.006
55. Baley C (2004) Influence of kink bands on the tensile strength of flax fibers. J Mater Sci 39:331–334. https://doi.org/10.1023/B:JMSC.0000007768.63055.ae
56. Baley C (2002) Analysis of the flax fibres tensile behaviour and analysis of the tensile stiffness increase. Compos - Part A Appl Sci Manuf 33:939–948. https://doi.org/10.1016/S1359-835X(02)00040-4
57. D3822/D3822M-14. Standard Test Method for Tensile Properties of Single Textile Fibers. ASTM Int USA 2014. https://doi.org/10.1520/D3822.
58. Weibull W (1951) A statistical distribution function of wide applicability. J Appl Mech 18:293–297

59. Jihan S, Siddiquib AM, Sweet MAS (1997) Fracture strength of E-glass fibre strands using acoustic emission. NDT E Int 30:383–388. https://doi.org/10.1016/S0963-8695(97)00009-1
60. El Asloun M, Donnet JB, Guilpain G, Nardin M, Schultz J (1989) On the estimation of the tensile strength of carbon fibres at short lengths. J Mater Sci 24:3504–3510. https://doi.org/10.1007/BF02385732
61. Zureick A-H, Bennett RM, Ellingwood BR (2006) Statistical characterization of fiber-reinforced polymer composite material properties for structural design. J Struct Eng 132:1320–1327. https://doi.org/10.1061/(asce)0733-9445(2006)132:8(1320)
62. Colomban P, Herrera Ramirez JM, Paquin R, Marcellan A, Bunsell A (2006) Micro-Raman study of the fatigue and fracture behaviour of single PA66 fibres: Comparison with single PET and PP fibres. Eng Fract Mech 73:2463–2475. https://doi.org/10.1016/j.engfracmech.2006.04.033
63. Jr. FJM. The Kolmogorov-Smirnov Test for goodness of fit. J Am Stat Assoc 1951;46:68–78.
64. Elsayed EA (2008) Reliability prediction and accelerated testing. Springer Ser Reliab Eng. https://doi.org/10.1007/978-1-84800-011-7_7
65. Virk AS, Hall W, Summerscales J (2010) Physical characterization of jute technical fibers: fiber dimensions. J Nat Fibers 7:216–228. https://doi.org/10.1080/15440478.2010.504389
66. Andersons J, Sparniņš E, Joffe R, Wallström L (2005) Strength distribution of elementary flax fibres. Compos Sci Technol 65:693–702. https://doi.org/10.1016/j.compscitech.2004.10.001
67. Toasa Caiza PD, Ummenhofer T (2018) Consideration of the runouts and their subsequent retests into S-N curves modelling based on a three-parameter Weibull distribution. Int J Fatigue. https://doi.org/10.1016/j.ijfatigue.2017.09.010
68. Moothoo J, Allaoui S, Ouagne P, Soulat D (2014) A study of the tensile behaviour of flax tows and their potential for composite processing. Mater Des 55:764–772. https://doi.org/10.1016/j.matdes.2013.10.048
69. Virk AS, Hall W, Summerscales J (2009) Tensile properties of jute fibres. Mater Sci Technol 25:1289–1295. https://doi.org/10.1179/174328408x385818
70. Virk AS, Hall W, Summerscales J (2010) Failure strain as the key design criterion for fracture of natural fibre composites. Compos Sci Technol 70:995–999. https://doi.org/10.1016/j.compscitech.2010.02.018
71. Virk AS, Hall W, Summerscales J (2012) Modulus and strength prediction for natural fibre composites. Mater Sci Technol 28:864–871. https://doi.org/10.1179/1743284712y.0000000022
72. Chi Z, Chou TW, Shen G (1984) Determination of single fibre strength distribution from fibre bundle testings. J Mater Sci 19:3319–3324. https://doi.org/10.1007/BF00549820
73. Estrada M, Linero DL, Ramírez F (2013) Constitutive relationship of the fiber cluster of bamboo Guadua angustifolia, determined by means of a Weibull probability function and a model of progressive failure. Mech Mater 63:12–20. https://doi.org/10.1016/j.mechmat.2013.04.007
74. Ahmadi MV, Doostparast M, Ahmadi J (2013) Estimating the lifetime performance index with Weibull distribution based on progressive first-failure censoring scheme. J Comput Appl Math 239:93–102. https://doi.org/10.1016/j.cam.2012.09.006
75. Wolfenden A, van der Zwaag S (2009) The concept of filament strength and the Weibull modulus. J Test Eval 17:292. https://doi.org/10.1520/jte11131j
76. Abernethy RB (2004) An overview of weibull analysis. New Weibull Handb. Reliab. Stat. Anal. Predict. Life, Safety, Surviv. Risk, Cost Warranty Claims.
77. Park JM, Quang ST, Hwang BS, DeVries KL (2006) Interfacial evaluation of modified Jute and Hemp fibers/polypropylene (PP)-maleic anhydride polypropylene copolymers (PP-MAPP) composites using micromechanical technique and nondestructive acoustic emission. Compos Sci Technol 66:2686–2699. https://doi.org/10.1016/j.compscitech.2006.03.014
78. Gorjan L, Ambrožič M (2012) Bend strength of alumina ceramics: a comparison of Weibull statistics with other statistics based on very large experimental data set. J Eur Ceram Soc 32:1221–1227. https://doi.org/10.1016/j.jeurceramsoc.2011.12.010
79. Bergman B (1984) On the estimation of the Weibull modulus. J Mater Sci Lett 3:689–692. https://doi.org/10.1007/BF00719924
80. Stawarczyk B, Özcan M, Trottmann A, Hämmerle CHF, Roos M (2012) Evaluation of flexural strength of hipped and presintered zirconia using different estimation methods of Weibull statistics. J Mech Behav Biomed Mater 10:227–234. https://doi.org/10.1016/j.jmbbm.2012.01.020
81. Abbasi B, Niaki STA, Khalife MA, Faize Y (2011) A hybrid variable neighborhood search and simulated annealing algorithm to estimate the three parameters of the Weibull distribution. Expert Syst Appl 38:700–708. https://doi.org/10.1016/j.eswa.2010.07.022
82. Gebizlioglu OL, Enolu B, Kantar YM (2011) Comparison of certain value-at-risk estimation methods for the two-parameter Weibull loss distribution. J Comput Appl Math 235:3304–3314. https://doi.org/10.1016/j.cam.2011.01.044
83. Ducros F, Pamphile P (2018) Bayesian estimation of Weibull mixture in heavily censored data setting. Reliab Eng Syst Saf. https://doi.org/10.1016/j.ress.2018.08.008
84. Roos M, Stawarczyk B (2012) Evaluation of bond strength of resin cements using different general-purpose statistical software packages for two-parameter Weibull statistics. Dent Mater 28:e76–e88. https://doi.org/10.1016/j.dental.2012.04.013
85. Gourier C, Le Duigou A, Bourmaud A, Baley C (2014) Mechanical analysis of elementary flax fibre tensile properties after different thermal cycles. Compos Part A Appl Sci Manuf 64:159–166. https://doi.org/10.1016/j.compositesa.2014.05.006
86. Davies GC, Bruce DM (1998) Effect of environmental relative humidity and damage on the tensile properties of flax and nettle fibers. Text Res J 68:623–629. https://doi.org/10.1177/004051759806800901
87. Hu W, Ton-That MT, Perrin-Sarazin F, Denault J (2010) An improved method for single fiber tensile test of natural fibers. Polym Eng Sci 50:819–825. https://doi.org/10.1002/pen.21593
88. Joffe R, Andersons JA, Wallström L (2003) Strength and adhesion characteristics of elementary flax fibres with different surface treatments. Compos Part A Appl Sci Manuf 34:603–612. https://doi.org/10.1016/S1359-835X(03)00099-X
89. Charlet K, Eve S, Jernot JP, Gomina M, Breard J (2009) Tensile deformation of a flax fiber. Procedia Eng 1:233–236. https://doi.org/10.1016/j.proeng.2009.06.055
90. Alix S, Lebrun L, Marais S, Philippe E, Bourmaud A, Baley C et al (2012) Pectinase treatments on technical fibres of flax: effects on water sorption and mechanical properties. Carbohydr Polym 87:177–185. https://doi.org/10.1016/j.carbpol.2011.07.035
91. Arbelaiz A, Cantero G, Fernández B, Mondragon I, Gañán P, Kenny JM (2005) Flax fiber surface modifications: effects on fiber physico mechanical and flax/polypropylene interface properties. Polym Compos 26:324–332. https://doi.org/10.1002/pc.20097
92. Biagiotti J, Puglia D, Torre L, Kenny JM, Arbelaiz A, Cantero G et al (2004) A systematic investigation on the influence of the chemical treatment of natural fibers on the properties of their polymer matrix composites. Polym Compos 25:470–479. https://doi.org/10.1002/pc.20040
93. Placet V, Trivaudey F, Cisse O, Gucheret-Retel V, Boubakar ML (2012) Diameter dependence of the apparent tensile modulus of hemp fibres: a morphological, structural or ultrastructural effect? Compos Part A Appl Sci Manuf 43:275–287. https://doi.org/10.1016/j.compositesa.2011.10.019

94. Placet V, Cissé O, Lamine BM (2014) Nonlinear tensile behaviour of elementary hemp fibres. Part I: investigation of the possible origins using repeated progressive loading with in situ microscopic observations. Compos Part A Appl Sci Manuf 56:319–327. https://doi.org/10.1016/j.compositesa.2012.11.019
95. Béakou A, Ntenga R, Lepetit J, Atéba JA, Ayina LO (2008) Physico-chemical and microstructural characterization of "Rhectophyllum camerunense" plant fiber. Compos Part A Appl Sci Manuf 39:67–74. https://doi.org/10.1016/j.compositesa.2007.09.002
96. Quinn JB, Quinn GD (2010) A practical and systematic review of Weibull statistics for reporting strengths of dental materials. Dent Mater 26:135–147. https://doi.org/10.1016/j.dental.2009.09.006
97. Amroune S, Bezazi A, Dufresne A, Scarpa F, Imad A (2019) Investigation of the date palm fiber for green composites reinforcement: thermo-physical and mechanical properties of the fiber. Journal of Natural Fibers. https://doi.org/10.1080/15440478.2019.1645791