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Determination of the rupture parameters of a plant fiber by using two diameter measurement techniques

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ABSTRACT

An essential challenge for the future of composite materials is to make their manufacture and use compatible with increasingly demanding environmental expectations. In this context, the replacement of mineral or synthetic fibers by plant fibers is of great interest. However, the latter must comply with the same specifications, particularly in terms of mechanical properties. Thanks to their low density, their biodegradability as well as their abundance, the use of these fibers is interesting from Thanks to their low density, their biodegradability as well as their abundance, the use of these fibers is interesting from thanks to their low density, their biodegradability as well as their abundance, the use of these fibers is interesting from economic and environmental point of view.

The dimensions of the fiber, such as diameter, are essential elements when calculating the mechanical parameters of the plant fiber. Among the techniques used to measure the diameter, one can use the optical microscope, the software ImageJ and the density. Theoretically, similar failure parameters should be obtained when different techniques are used. However, the experimental data show various parameters according to different used techniques. In this work, we carried out several experiments on natural fibers in order to determine the fracture parameters in static traction by calculating the diameter of the fiber by two ways: density method and optical method. The results reveal that the failure parameters are dependent on the diameter, hence the suitable technique plays a crucial role.

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1. Introduction

In the past years, plant fibers have been relied on as a reinforcement for composite materials such as: jute, flax, hemp and kenaf [1], and they have shown environmental and economic advantages over synthetic fibers. The structural and functional properties of natural fibers have stimulated the industrial sectors and nominated these fiber as environmentally safe products [2]. The physical, chemical, mechanical, and structural properties of above fibers are varied based on climatic conditions, types of soil, and availability of water resources.

The suitability for industrial applications is determined based on the physical properties of the fibers. The density, length and diameter of the fibers in addition to their basic properties and the availability of standards are taken into account to determine their use in various industrial works [3–5]. There is no specific scale for measuring plant fiber dimensions, which makes the prospects open to many homemade solutions [6]. Various methods have been used, such as fiber density technique [7], optical microscopy [8], fiber size analyzers [9], 2D high-resolution scanners [10], and scanning electron microscopy [11]. These techniques are a recent study that was used to determine the diameter of the fibers to give the most accurate results.

There is a consensus that the measurement of the geometrical characteristics of lignocellusic fibers is a tough operation, this is because multiple aspects can directly influence the measurement result. Among these ones are the used preparation technique of the samples, image processing approaches, fundamental

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limitations of the measurement technique, and the used statistical method [6].

The extraction method, the plant characteristics such as age or maturity, climatic conditions of weather circumstances and the position where the cross sectional area of fiber is determined can also affect the mechanical properties of tested fiber. For example, several researchers have worked on flax fiber properties, and from the results, it is worth to point out that there is a large scatter in mechanical properties. The cause of the scattering has been partly attributed to uncertainties in the measurement of the fiber cross sectional area. The cross sectional area of fiber is usually calculated from the average of width measurements of the fibers along the fiber length in view of the fiber as a cylinder neglecting the presence of the cavity. (and ignoring the lumen space) [12].

In other hand, the method used for cross-sectional area measurement can also lead to large variation in tensile properties of natural fibers. Manimaran et al. [8] used Carl Zeiss optical microscope to calculate the average diameter of fibers extracted from the *Dracaena reflexa leaves*. A number of 25 samples of *Dracaenareflexa* fibers were identified. The diameter of each fiber was measured at five different places and the mean value was used for statistical analysis. It is very challenging to decide diameter of the plant fibers because the fiber is uneven in shape, so it is essential to calculate the average diameter of the fiber. The same technique is used by Belouadah et al. [13]. fiber diameters of *Atriplexhalimus L* plant were measured by an image processing software (Image J) using SEM images. The mean equivalent diameter of fibers of *Agave Americana L.* and *Hyphaenethebaica L.* fibers, assuming a cylindrical shape, were evaluated from density measurements by Msahal et al. [14] and Rokbi et al. [7] respectively, Berzin et al. [9] was also used to calculate the diameter of the *Cannabis sativa L* fibers using a soxhlet device and analyze their volumes using an automated analyzer, and Ching-carrasco et al was also used to measure the diameters of the centrifugal process, and samples were analyzed using a scanner [10]. Aslan et al. [12] investigated the variability in tensile properties of flax fibers using two different methods for cross-sectional area measurement. They found that the inaccuracy in the determination of the cross sectional area of the fibers was one major reason for the variability in calculated fiber properties.

The current study concerns the reliability of two techniques that are generally used for the determination of natural fibers diameter, i.e. density technique (DM) and optical technique (OM). In practice, the cross section of the fibers show irregular shapes (non-uniformity along the length of fiber), and includes a visible central voids (the lumen spaces). We believe that the decrease in mechanical properties can be related to the increase in lumen size with increasing diameter. The influence of the measurement method of the fiber dimensions and the calculation of the cross-sectional area has been investigated and analyzed. Alfa fibers which were extracted from Stipatenacissima L. plant were used in this study. A comparative study is conducted between these two techniques when the Alfa fibers were subjected to the single fiber tensile test.

2. Materials and experimental methods

2.1. Plant material

Alfa fiber has been used in this study(Stipatenacissima L.) fibers were collected from BouSaada region (Algeria), Alfa fibers contain cylindrical stems with have a maximum height of about 1 m and a surface area of 2 m^2. The Alfa fibers consist of 45% cellulose, 24% of hemicellulose, 24% of lignin, 2% ash and 5% wax [15]. Physical and mechanical properties of Alfa fiber are listed in Table 1.

2.2. Fiber extraction

After harvesting, lignocellusic fibers were extracted from the Alfa plant represented in Fig. 1 by the following steps: The Alfa plant was washed with pine water to remove dust and excess impurities, the stems were immersed in a water tank filled with tap water for biological regeneration for 25 days to facilitate the process of separating the fibers from the stems, and then the fibers were manually removed from the stems using an iron comb and then washed with distilled water.

Finally, the fibers were dried in an oven at 70 °C for 6 h.

2.3. Fourier transform infrared spectroscopy (FT-IR)

Fourier transform infrared spectroscopy [FT-IR] was used to determine the chemical components present in the plant Alfa formation, the FT-IR test was carried out using a (Cary630KBr Engine, Agilent Technologies) apparatus at room temperature.

Infrared spectra were recorded in the wavelength range from 4000 cm^{-1} to 400 cm^{-1} with a spectral resolution of 2 cm^{-1}.

2.4. Diameter measurement technique

2.4.1. Density technique

The density of the fibers (ρ_f) was calculated by pycnometer technique, it was filled with ethanol liquid (ρ_e = 0.79 g/cm^3), the sample was cut into 10 mm, then it was dried in an oven at 60 °C for 15 min, the measurements were made on a sensitivity scale of 0.0001 g, the density of the fibers was obtained by the relation (1):

$$\rho_f = \frac{(m2 - m0)}{(m1 - m0) - (m3 - m2)}\rho_e \quad (1)$$

where m_0 is the mass of the vacant pycnometer, m_1 is the mass of the pycnometer filled with ethanol, m_2 is the mass of the pycnometer and fiber embedded, and m_3 is the mass of the pycnometer with ethanol and fiber

From the density results presented above, the equivalent diameter can be found assuming a cylindrical shape, using the following relationship (2) [14]:

$$D_e(\mu m) = \sqrt{4 * D(mtex)/\pi \cdot \rho DLF(g/cm^3)} \quad (2)$$

2.4.2. Optical technique

The fiber diameter of the outer surface of the Alfa fiber was measured using an optical microscope (Metallurgical Microscope A13.1013 BF/DF DIC) equipped with a high-resolution digital camera. Images were processed by analysis program (image J). In order to measure the diameter distribution, 20 fiber samples were cut in the lower, middle and upper part of the root, the diameter was determined by measuring on three different regions of each fiber and given the average value between them. The fiber diameters were also shown to be in the range [0.15–1 mm]. It may be difficult to accurately measure the diameter of natural fibers, because the fibers have different thicknesses, which make them take an irregular shape. Also, natural fibers contain a large number of elements surrounded by lignin and hemicellulose, and therefore its cross-section is not circular [17].

2.5. Single fiber tensile test

The tensile strength of Alfa fibers was tested by adopting standard unit D3322-01 for tensile properties, and the process was conducted using (micro traction). The mechanical properties of the Alfa fibers (Young modulus, tensile strength, and failure mod-

Table 1
Physical and mechanical properties of Alfa fiber [16].

Materials	Physical properties				Mechanical properties			
	Diameter of fibre bundles (μm)	Diameter of ultimate fibre (μm)	Density (g/cm^3)	Linear density (Tex)	Tensile strength, (MPa)	Strain at break ξ (%)	Tensile modulus (GPa)	Stress at break (MPa)
Alfa fiber	50–120	6–22	0.89–2.10	13.3–21.9	134–264	1.5–5.8	11–22	134–220

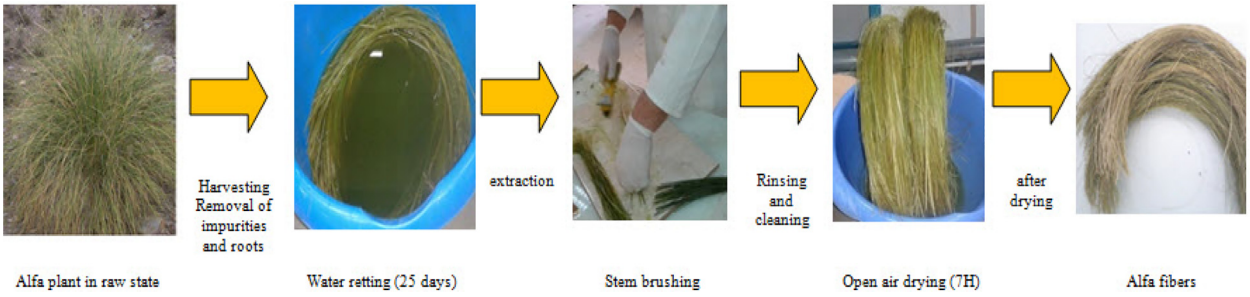

Fig. 1. Alfa fiber extraction techniques.

ulus) were calculated using 25 single fibers with a length of 40 mm and a transverse velocity of 2.5 mm/min Equipped with a 2.5 kN load cell.

3. Results and discussion

3.1. Fourier transform infrared spectroscopy (FT-IR)

Fig. 2 represents the analytical spectra of Alfa fibers obtained by ATR-FTIR, the broadband at 3296 cm^{-1} was assigned to the O-H hydroxyl group of cellulose [8], and the C-H saturated expansion vibrations of CH and CH_2 were observed at 2845 cm^{-1} and 2919 cm^{-1} [7], the range of 1734 cm^{-1} corresponds to the hemicellulose carbonyl ester group C=O [7], the range of 1560 cm^{-1} is attributed to the expansion of the C=C bonds [18], and that around 1456.13 cm^{-1} is due to the symmetric curvature of cellulose CH_2 [13], and it appears As well as the bending of the C - O - C alkyl bonds at 1419.32 cm^{-1} and 1371.47 cm^{-1} [13],the resulting band of lignin-acetylated C-O group was determined at 1236.38 cm^{-1} [13], the band at 1032 cm^{-1} corresponds to the stretching patterns C-O for hydroxyl [13], the small band at 897 cm^{-1} is assigned with β-glycosidic bonds [7]. The different peaks assignments corresponding to various groups are summarized in Table 2.

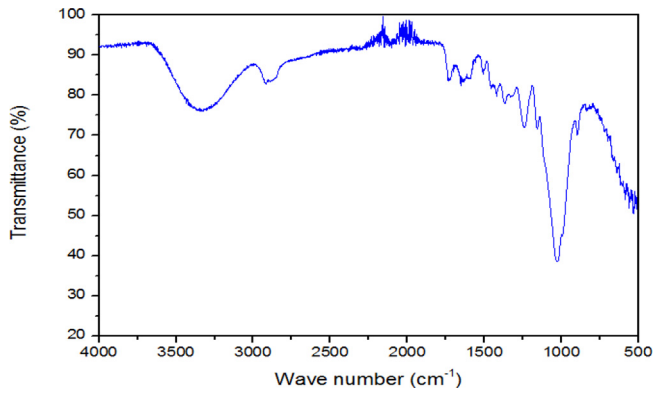

Fig. 2. ATR–FTIR spectrum of fibers extracted from the Alfa fiber.

Table 2
Identification of peaks ATR-FTIR spectra of Alfa fiber.

Wave number (cm^{-1})	Vibrations mode (s)	Source (s)	References
3296	O-H stretching	Cellulose,	[8]
2919–2845	C–H stretching	Hemicellulose	[7]
1734	C=O stretching	Cellulose,	[7]
1560	C=C stretching	Hemicellulose	[18]
1456.13	C–H stretching	Hemicellulose, lignin and Extractives	[13]
1419.32–1371.47	C-O-C stretching	Lignin	[13]
1236.38	C-O stretching	Lignin	[13]
1032	C–O bridge	Lignin	[13]
895	Stretching ß-glycosidic linkage	Cellulose, Hemicellulose Cellulose, Hemicellulose	[7]

3.2. Diameter measurement technique

3.2.1. Density technique

One of the most important characteristics that draw attention to researchers developing composites is the low density. The lightness of composite materials can contribute to lower energy consumption. The average value was taken using relationship (1), where a value of 1.13 g/cm^3 was obtained for Alfa fibers. The fiber density is shown along with the published values in Table 3. Thus the density of Alfa fibers appears to be higher than that of *Dracaena reflexa* fibers (0.79 g/cm^3) and almost equal to that of *Juncuseffusus L* fibers (1.139 g/cm^3) and lower than that of *Doum* fibers (1.19 g/cm^3), *Kigelia Africana* (1.316 g/cm^3) and *Lygeum Spartum L*

Table 3
Comparison of some physical properties of Alfa fiber.

Fibers type	Density (g/cm^3)	References
Alfa fiber	1.13 ± 0.04	Present work
Doum fiber	1.19 ± 0.05	[7]
Dracaena reflexa	0.79	[8]
LygeumSpartum L	1.4997	[19]
Kigelia Africana	1.316	[20]
Juncuseffusus L	1.139	[21]

(1.4997 g/cm^3). This makes the use of Alfa fibers as a reinforcement suitable for lightweight composite materials.

3.2.2. Optical technique

The process of measuring the diameter of plant fibers with an optical microscope is complex because the diameter of the fibers is opaque in nature. The fibers are assumed to be circular in shape to calculate the stress ratio at break and Young's modulus. After calculation, the diameter of the Alfa fibers ranges between (120–300 μm).

3.3. Single fiber tensile test

Fig. 3 represents the evolution of the stress/strain curves for the Alfa fibers that were tested for the different techniques adopted (optical method (Fig. 3a) and density method (Fig. 3b)). The tensile properties of tested Alfa fiber, using both techniques, are compared and are shown in the Table 4. It is noteworthy that the tested Alfa fibers have the same behavior for both techniques once they are under tensile loads. Like most natural fibers subjected to mechanical testing, a large scattering in the tensile results of Alfa can be seen for both techniques. This dispersion can be explained by many different factors that influence the quality of fibers such as [7,8]:

- Plant characteristics (maturity, climatic conditions),
- Extraction methods,
- Test parameters/conditions,

Added to these are the variation of the number of fiber cell from one bundle to another [19], and the stress conditions of the fibers (fiber tightening).

The tear parameters of the tensile test of Alfa fibers are shown for the density technique (394.98 ± 163.86 MPa) and for Young's modulus (21.66 ± 3.8 GPa), while the optical technique shows (300.63 ± 182.43 MPa) for the stress and (15.32 ± 4.07 GPa) for the Young's modulus.

Fig. 4 illustrates of the stress/strain curves for the Alfa fibers (diameter ≈ 0.3 mm). From this Figure, it seem that the values of the mechanical properties in tensile appear to be sufficiently preserved for the same diameter when using the density method. In contrast, a large dispersion is observed in the case of optical method. In other words, the mechanical properties measurements in tensile are strongly dependent on the used technique for determining the diameter of fiber.

As known, that an increasing diameter of the fibers leads to a decrease in the mechanical properties of the fibers. The lumen size and the presence of porosity affect directly the physical properties of lignocellulosic fiber such as fiber diameter [22]. Added to this the irregular shape of the fiber and the presence of lignin and hemicelluloses surrounded the fiber surface. Hence, It is well recognized the difficulty of measuring with precision the diameter of the natural fiber [8]. If we compare the two techniques used, the measurement of the diameter using the density technique would appear closer to the real dimension of the fiber diameter.

3.4. Statistical analysis

The cumulative distribution function for the three-parameter Weibull distribution is defined by (3):

$$\rho_f = 1 - \exp\left[-\left(\frac{\chi - \chi_\mu}{\chi_0}\right)^{m_x}\right] \quad (3)$$

Three parameter PDF

$$f(x) = \frac{\gamma}{\alpha}\left(\frac{(x-\mu)}{\alpha}\right)^{\gamma-1} exp\left[-\left(\frac{x-\mu}{\alpha}\right)^{\gamma}\right]$$

μ=0

Two parameter PDF

$$f(x) = \frac{\gamma}{\alpha}\left(\frac{x}{\alpha}\right)^{\gamma-1} exp\left[-\left(\frac{x}{\alpha}\right)^{\gamma}\right]$$

μ:location parameter *γ:shape parameter* *α:scale parameter*

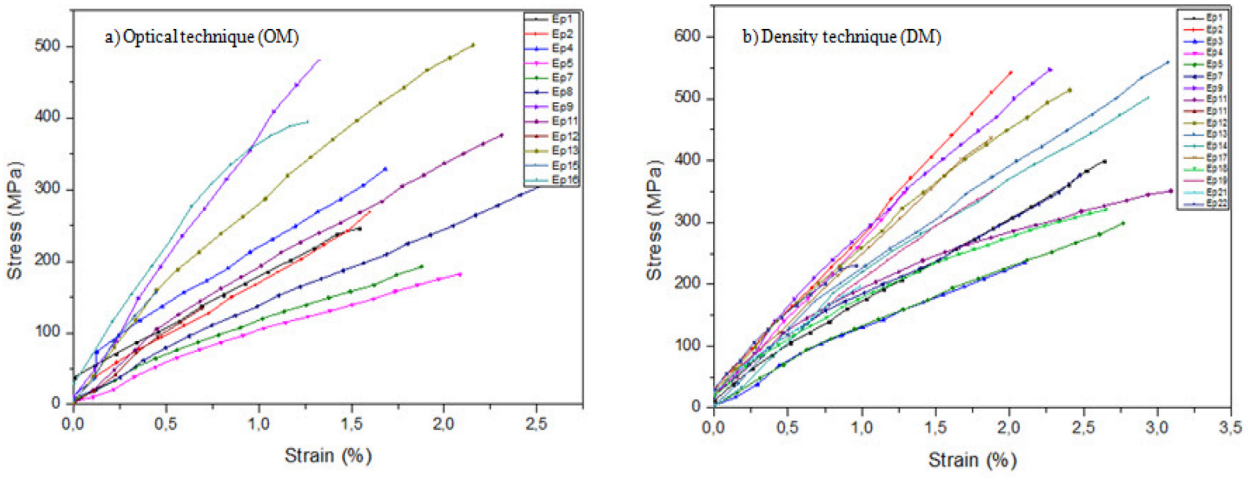

Fig. 3. Typical tensile stress/strain curve for Alfa fiber.

Table 4
Comparison of tensile properties of Alfa fiber with other natural fibers.

Fibers type	Gauge length (mm)	Diameter (μm)	Tensile strength (MPa)	Young's modulus (GPa)	Strain failure (%)	References
Alfa (DM)	40	208–324	394.98 ± 163.86	21.66 ± 3.8	0.93–3.07	Present work
Alfa (OM)	40	120–300	300.63 ± 182.43	15.32 ± 4.07	1.09–3.22	Present work
Doum leaf fibers	40	137.02–220.42	124.84–448.1	4.06–19.59	0.81–2.86	[7]
Dracaena reflexa	70	176.2	829.6	46.37	2.95	[8]
LygeumSpartum L	40	180–433	64.63–280.03	4.47–13.27	1.49–3.74	[19]
Kigelia Africana	40	582 ± 204	52.68 ± 11.97	36.84 ± 33.68	0.22 ± 0.11	[20]
Juncuseffusus L	40	280 ± 56	113 ± 36	4.38 ± 1.37	2.75 ± 0.6	[21]

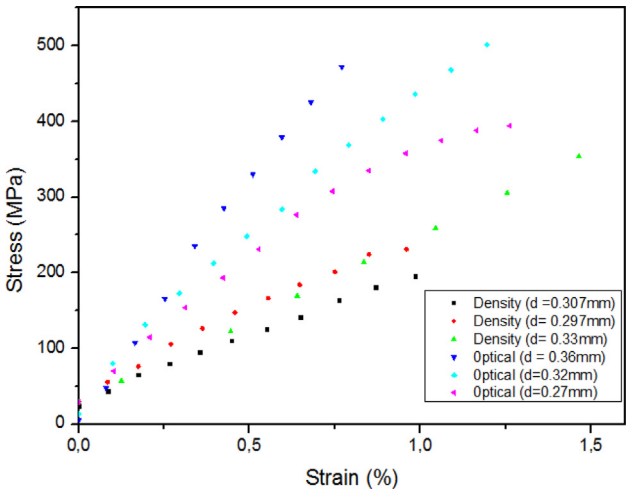

Fig. 4. Typical tensile stress/strain curves for Alfa fiber for a diameter ≈ 0.3 mm.

Where χ_0 is called scale or characteristic value, m_x indicates the shape or Weibull modulus (m_σ, m_E) and χ_μ is the threshold (location, minimum life, origin, guaranteed minimum life, shift). P_f is the probability of survival of the strength and Young's modulus for the parameter χ (σ, E) in a test specimen. If χ_μ is positive, it provides a guaranteed failure free period from 0 to χ_μ. A non-zero threshold parameter should not be used unless it is anchored in the physics of the failure process. The two-parameter Weibull model is obtained by assuming that the threshold is equal to zero (χ_μ = 0) [23].

The mechanical properties are analyzed by the 2-parameter Weibull method to see the most adequate distribution in terms of mechanical properties, namely, the tensile strength, the strain and the Young's modulus of the two methods used. Fig. 5 shows the Weibull distribution for stress and strain at break and Young's modulus of Alfa fibers. The 2-parameter Weibull's modulus is determined graphically by a linearized equation (the least square estimate LS method), which is the slope of the curve. Likewise, form and scale factors can be determined. This method makes it possible to determine modulus (m), as well as the comparison between the various hypotheses linked to the nature of the experimental results obtained. Fig. 5c and Fig. 5d compare the two assessment methods. For example, if we take the modulus (m) and the characteristic stress (σ0) of Weibull with two parameters for the method DM which are respectively equal to 1.79 and 465.56 MPa. For the Weibull modulus of the OM technique, we find that: m = 1.28 and σ0 = 509.22.73 MPa. It is also noted that the data of the OM technique are adequate compared to the DM technique.

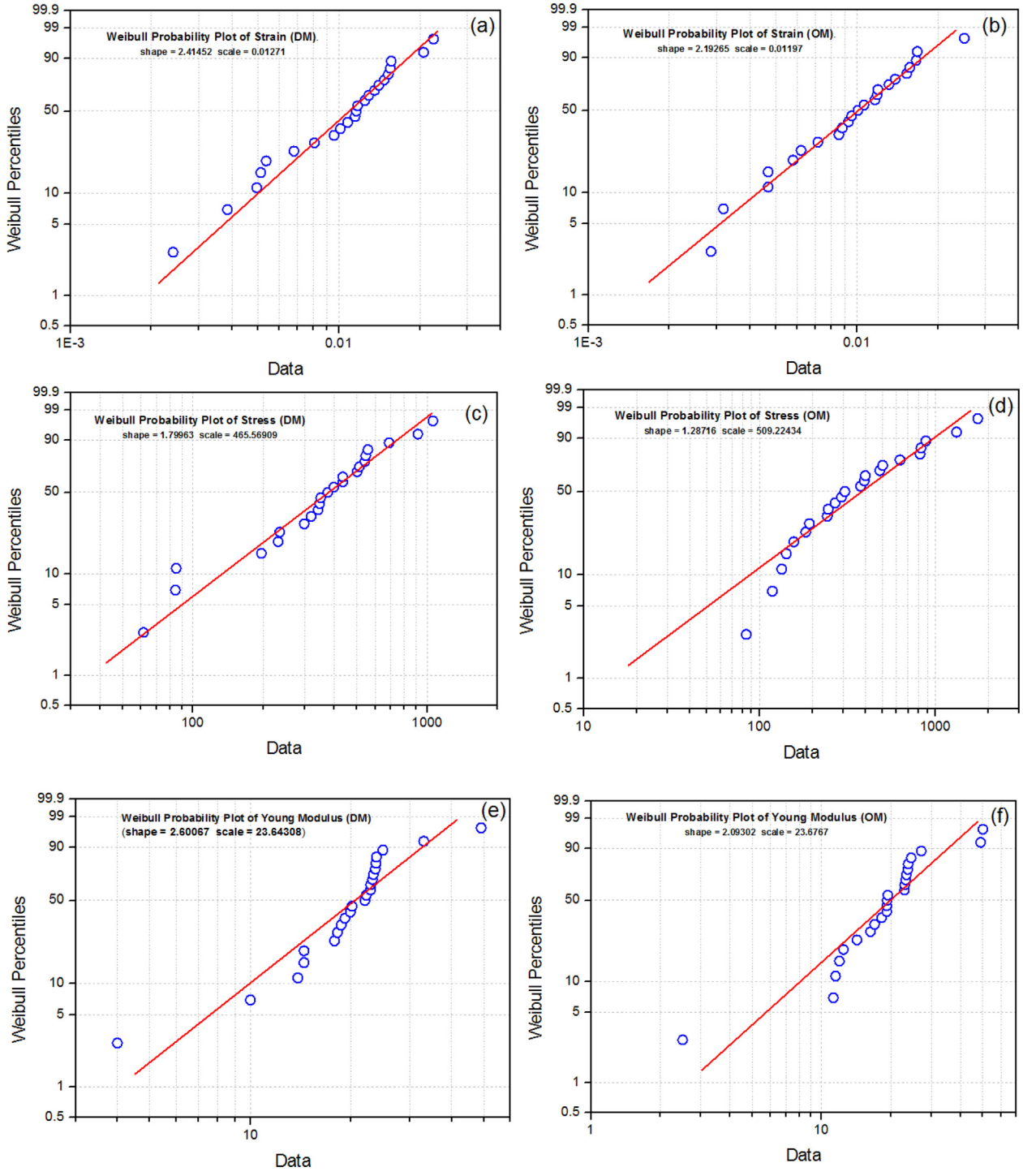

Fig. 5. Two and three Weibull distribution for the tensile strength, strain at break, and young's modulus.

4. Conclusion

In this work, two methods for calculating the diameter of the fibers were used to evaluate the mechanical properties, namely: stress, strain at break, and Young's modulus. From this study we reveal the following results:

- The diameter measurement method takes a long time in terms of preparing the sample before it is measured, but the results found are very similar compared to other studies in the literature.
- In theory, similar failure parameters should be obtained when using different techniques however experimental data show different parameters according to different techniques.
- We also note a difference in the surface of the fibers for two techniques, where we find that the surface of the fibers is empty and heterogeneous with respect to the density technique, and we note in the optical technique that the surface of the fibers is full and homogeneous.
- Finally, we can say that the analysis of the dimensions of lignocellulosic fibers is a little complex.

CRediT authorship contribution statement

Abd Raouf Khaldoune: Resources, Methodology, Data curation and Investigation, Writing - Analysis, review & editing. **Mansour Rokbi:** Resources, Methodology, Writing-original draft, original draft, Investigation & supervision, Writing - review & editing. **Salah Amroune:** Methodology, original draft, supervision and Investigation, Writing - review & editing. **Said Zergane:** Formal analysis and Investigation, Writing - review & editing. **Abdelmadjid Benadda:** Formal analysis and Investigation, Writing - review & editing. **Noureddine Nouari:** Formal analysis and Investigation, Writing - review & editing

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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