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Tensile mechanical properties and surface chemical sensitivity of technical fibres from date palm fruit branches (*Phoenix dactylifera* L.)

Salah Amroune [a,b], Abderrezak Bezazi [a], Ahmed Belaadi [a], Chenchen Zhu [c,d], Fabrizio Scarpa [c,d,*], Sameer Rahatekar [c,d], Abdellatif Imad [e]

[a] *Laboratoire de Mécanique Appliquée des Nouveaux Matériaux (LMANM), Université 8 Mai 1945, Guelma, Algeria*
[b] *Université de M'sila, B.P 166 ICHBELIA, M'sila 28000, Algeria*
[c] *Advanced Composites Centre for Innovation and Science (ACCIS), University of Bristol, BS8 1TR Bristol, UK*
[d] *Bristol Centre for Nanoscience and Quantum Information (NSQI), University of Bristol, BS8 1FD Bristol, UK*
[e] *Laboratoirede Mécanique de Lille, Université Lille1, CitéScientifique, Avenue Paul Langevin, 59655 Villeneuve d'AscqCedex, France*

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ABSTRACT

The paper describes the manufacturing process and the characterization of the tensile mechanical properties of treated and untreated palm dates long technical fibres. The fibres extracted from Fruit Bunch Branch of Palm Date (FBBPD) have been subjected to alkaline treatment with different NaOH concentrations at room temperature. The experimental results show that the chemical technical fibre treatments provide an increase of the mechanical properties (tensile strength and Young's modulus) under quasi-static tensile loading. A specific treatment leads a threefold increase of the failure stress. An analysis of stress at failure has been performed over a population of 630 samples using Weibull statistics with two and three-parameters, together with a one-way analysis of variance (ANOVA). FBBPD technical fibres show stiffness and strength performance comparable to the ones of agave Americana L fibres, and higher failure at strength than okra fibres.

1. Introduction

Natural fibres extracted from plants provide reinforcement to composites used in transport and constructions. A typical example is constituted by automotive components (interior trim doors, records of seats, carpet supports, bonnets) that are reinforced by plant fibres [1], like kenaf, jute, sisal, hemp and flax [2–10]. To obtain acceptable mechanical properties for the fibres it is necessary to use an adequate extraction method that allows the production reinforcements with minimal defects. There is abundant evidence in open literature that the production process has a significant impact on the chemical composition of the fibres, upon their lengths and general mechanical properties [11,12]. The most popular fibre extraction techniques consist in mechanical decortication, seawater retting, burial in earth and chemical methods using soda at low concentrations, to avoid the degradation of cellulose [13–16]. Physical properties like density, tensile strength and Young's modulus are linked to the internal structure and chemical composition, which vary from one fibre to another [17]. Belkhir et al. [18] investigated the effect of the harvest season and climate over the chemical composition of Alfa fibres. Alawar et al. have examined the effect given by a chemical treatment on the surface of the fibres surrounding the date palm leaves to improve the adhesion at the interface between fibre and matrix [19]. A study presented by Tahaet et al. [20] has identified the mechanical properties of fibres extracted from the stem spadix of palm date DPSS from the region of Ezbet El-Nakhl in Egypt. These rods have been cut into 50 cm long fibres and then chemically treated with hydroxide (NaOH) with a concentration ranging from 2% to 5% for periods varying between 2 and 24 h at a temperature of 23 °C. The most interesting results have been obtained from a solution of NaOH concentration of 2% for a treatment period of 2 h. The tensile strength of the chemically treated fibres was 600 MPa, which represents an increase of 50% compared to the case of the untreated reinforcements. Abdel-Rahmanet et al. [21] have investigated the mechanical properties of the fibres of the rods of Palms (rachis). The results of their study indicate that the tensile strength of the stem walls varies between 116 MPa and 208 MPa, while the heart of the rods provides 50% of those values. The Young's modulus of the stems ranges between 10 GPa and

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[*] Corresponding author at: ACCIS, University of Bristol, BS8 1TR Bristol, UK.
E-mail addresses: amroune_salah@yahoo.fr (S. Amroune), ar_bezazi@yahoo.com (A. Bezazi), ahmedbelaadi1@yahoo.fr (A. Belaadi), f.scarpa@bristol.ac.uk (F. Scarpa), abdellatif.imad@polytech-lille.fr (A. Imad).

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30 GPa. An experimental study on the natural mat surrounding the date palm tree subjected to tensile loading has been performed by Al-Khanbashiet et al. [22]. Palm date fibres consist primarily of 46% mass of cellulose, 20% lignin and 18% of hemicelluloses. The diameter of these fibres varies between 100 and 1000 μm, which are values generally larger than the ones present in other natural fibres. Tensile strength varies from 170 to 275 MPa, and the modulus of elasticity ranges between 5 GPa and 12 GPa, with an elongation at break of 5–10%. Treatment with a concentration of 5% NaOH at 100 °C and a duration of 2 h showed evidence of the enhancement of the tensile failure stress (496 MPa) of 45% compared to untreated fibre configurations.

Fatigue and mechanical endurance are also key parameters to characterise the behaviour of natural fibres [4,23]. Recently, Belaadi et al. [4] have investigated the behaviour under quasi-static tensile and fatigue cyclic loading of sisal fibres. Single fibres used in that work has an approximate diameter of 250 μm and a length of 0.8–1 m. The samples have been subjected to tensile loading

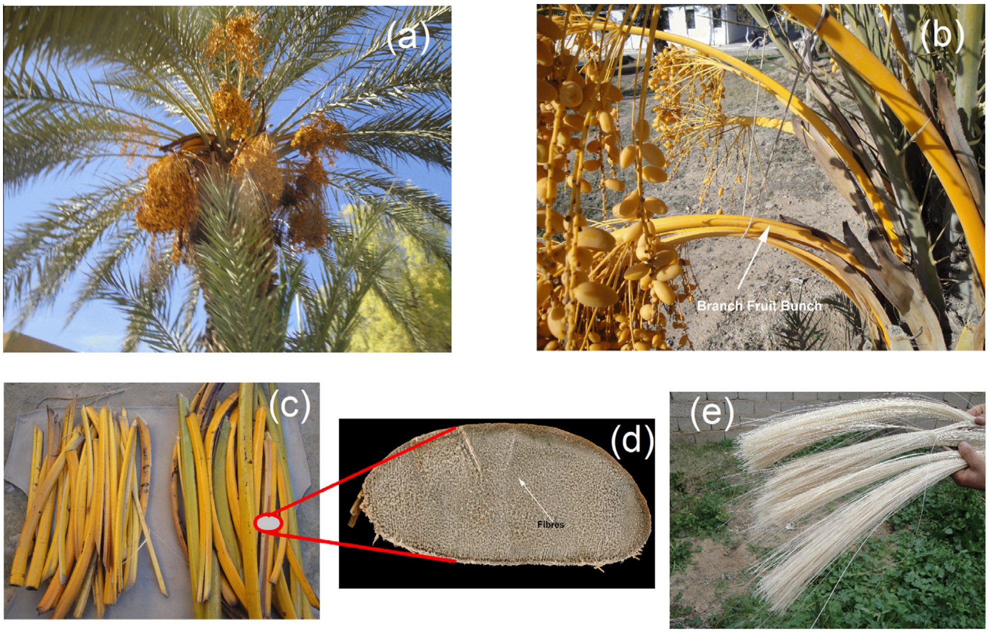

Fig. 1. Photographs of (a) date palm tree, (b) and (c) fruit bunch branch, (d) section transversal FBBPD (e) FBBPD technical fibres used in this work. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

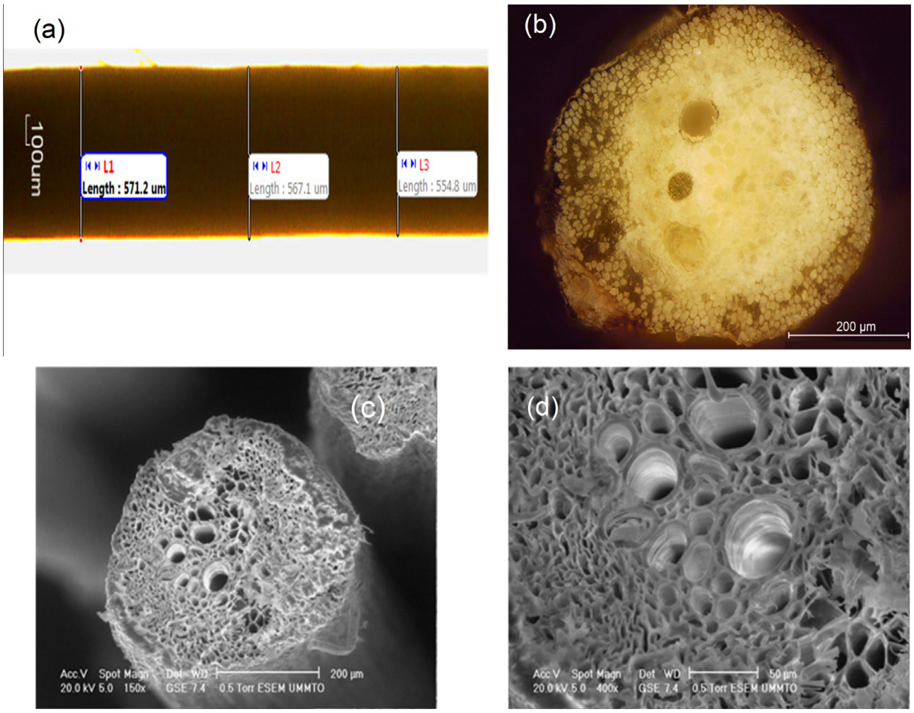

Fig. 2. (a, b) Optical microscopy image of a longitudinal and section cross. (c, d) SEM cross-sectional morphology and details of cell FBBDP technical fibre. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

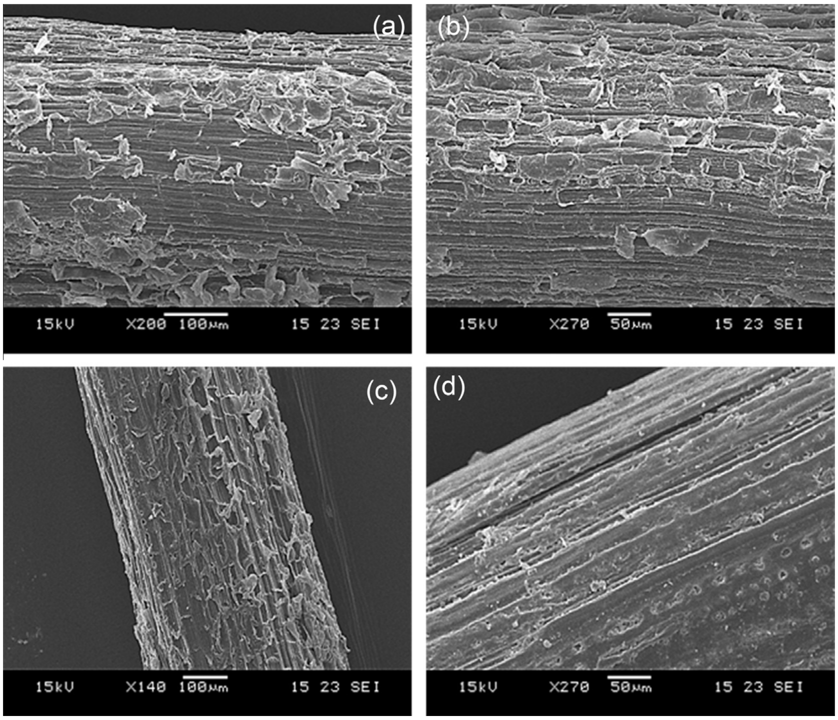

Fig. 3. (a, b) SEM images of FBBPD technical fibre untreated and treated at 0.5% for 12 h. (c, d) SEM images of FBBPD technical fibre treated at 1% and 3% for 12 h.

according to ASTM D3822-07 standard at gauge length (GL) of 20 mm. The average tensile stresses and strain at failure and the Young's modulus of 15 samples showed a significant dispersion, as already reported for other natural fibres [24–28]. It is worth mentioning that many scientists working in this field typically investigate the mechanical characteristics within the framework of Weibull statistics.

This work is devoted to the extraction and characterization of technical fibres from the fruit bunch branch of palm date (FBBPD) *Phoenix dactylifera L.* Only a limited number of works related to palm date exist in open literature [19–22,29], and this plant is particularly abundant in Algeria (over 18.6 millions of date palm trees), making the country the 5th in the world in terms of trees population [30]. These fibres are being discarded or burned each year after the harvest of the dates, generating 200,000 tonnes of waste [30], and could therefore constitute a potential source for recycling and sustainable material. The aim of this work is to determine the uniaxial tensile mechanical properties of long technical fibres FBBPD produced using a sustainable manufacturing process. The FBBPD technical fibres have been evaluated in their untreated and treated version (using different NaOH concentrations and times of treatments). The results of the mechanical properties of these novel FBBPD reinforcements have been compared against the analogous mechanical properties of other types of natural fibres. A total number of 630 specimens (21 batches of 30 samples) have been tested in this work. The mechanical properties (tensile strength and Young's modulus) are post-processed using both two and three-parameters Weibull statistics, and one-way ANOVA. To the best of the Authors' knowledge, this is the first time that this particular type of natural technical fibre has been investigated.

2. Material and technical testing

2.1. Morphology and extraction of the technical fibres

The palm date (*Phoenix dactylifera* L.) is a tree of 20–30 m high which grows in arid and semi-arid regions (Fig. 1a). The leaves of the tree are pinnate and divided, each having a length varying between 4 m and 7 m. Palm date trees has female and male inflorescences, and produce between 5 and 15 bunches of dates per tree [31]. Each bunch can weight between 6 kg and 8 kg, and can contain up to 1000 dates. A palm tree starts producing dates after 3 years of age, and can remain alive and productive for about 150 years [31]. The technical fibres used in this work come from FBBPD in the region of El-Hodna M′sila – Algeria, where the climate is semi-arid and the average temperature is 20 °C in November. During the summer (August) the shade temperature can reach 46 °C, and the average rainfall is 215 mm per year [32]. The fruit bunch branch of palm date (Fig. 1b and c) has a negligible cost because it can be retrieved during the harvest time (November, in general). The method used in this work to extract the technical fibres consists in immersing the FBBPD in a water bath for 48 h. The timescale of 48 h has been chosen after conducting several trials, during which it was found that this immersion time was sufficient to provide enough flexibility in the FBBPD branches to facilitate the extraction of the technical fibres. The branches are then peeled to remove the outer part and divided into 3–5 parts to facilitate the extraction of the fibres. The number of technical fibres obtained using this method varies between 500 and 750 (Fig. 1d), and their length can reach 1.2 m (Fig. 1e).

2.2. Treatment of the technical fibre with alkali

The technical fibres are all obtained from the same palm date to obtain statistically coherent data. After fabrication, the FBBPD technical fibres have been immersed in a solution of NaOH with concentrations of 0.5%, 1%, 2% and 3% for periods lasting 12 h, 24 h, 48 h, 72 h and 96 h at a room temperature of 23 °C. The technical fibres have been subsequently rinsed using tap water before being dipped into a 1% concentration of sulphuric acid (H_2SO_4) for 5 min. The FBBPD fibres are immersed in distilled water for 15 min to have a neutral Ph. and subsequently dried in an oven at a temperature of 70 °C for a period of 5 h. It is important to note that the technical fibres are all preserved in polyethylene bags prior to

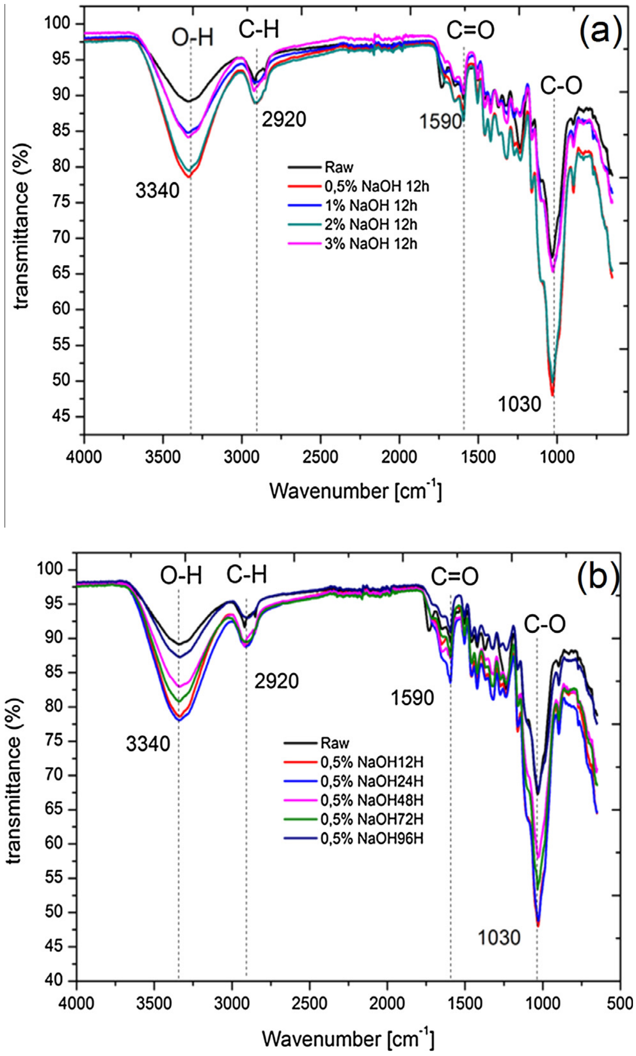

Fig. 4. (a) FTIR spectra of FBBPD technical fibres untreated and treated with (0.5%, 1%, 2% and 3% NaOH) for 12 h. (b) FTIR spectra of FBBPD technical fibres untreated and treated with 0.5% NaOH for (12, 24, 48, 72 and 96 h). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

testing to avoid moisture absorption. It is also worth of notice that the alkaline treatment of natural fibres has been also used by other authors [2,5,11,19,20,22].

2.3. Technical fibre quality analysis

The diameter of the technical fibres is measured using ZEISS optical microscope equipped with a Moticam2500 camera digitally controlled by Motic Images Plus V2.0 image processing program (Fig. 2a). Nine measurements have been taken all along the technical fibre (length of technical fibre 120 mm) in different places (three for each fibre end, and three in the middle). The cross-section of the fibres has been considered as circular (Fig. 2b and c). The structure of the fibre is composed of technical fibres (also called fibre bundles [33]) bonded together in part by a weak pectin and lignin interphase. The topology of the cross-section of these technical fibres is also similar to the one observed by Bendahou et al. in the leaflets of *Phoenix Dactifera*-L. [34], with average nominal diameters varying between 349 μm and 577 μ treated and untreated fibres surfaces were also examined using a scanning electron microscope (SEM) JSM-5600 with 15 kV accelerating voltage. All technical fibres have been covered with a thin layer of gold to make them conductive.

2.4. FTIR spectroscopy

To identify the influence provided by the treatment on the chemical composition of the fibres, Fourier transform infrared (FTIR) spectra were measured using an Elmer Perkin Spectrum 100 with proprietary quantitative analysis software. The spectra of different technical fibre samples have been measured with a scanning speed of 32 acquisitions between 500 and 4000 cm^{-1} and resolution of 2 cm^{-1}.

2.5. Tensile test

The mechanical properties (stress and strain at failure and Young's modulus) of the technical fibres at gauge length (GL) of 50 mm have been determined following the ASTM D 3822-01 standard, using a Zwick/Roell Z005 universal test machine with a 5 KN load cell. The clamps used during the tests have self-concentric alignment and are manually adjusted by mechanical springs. The tensile static tests were performed at a constant speed of 1 mm/min. All tests were conducted at a room temperature of 23 °C and a relative humidity of approximately 55%. The results have been analysed using two and three-parameters Weibull models and analysis of variance (ANOVA) using the Minitab software (version 15). The Weibull distribution provides a reasonable approximation of the experimental data to assess the mechanical properties of natural technical fibres. ANOVA has been performed to understand if the chemical treatment provides a meaningful statistical contribution to the mechanical properties of these

Table 1
FTIR spectral data of untreated and treated FBBPD technical fibres for (24, 48, 72 and 96 h).

Raw	Transmittance (cm^{-1})																		Possible assignments
	24 h				48 h				72 h				96 h				Ref. [20]	Ref. [19]	
	0.5%	1%	2%	3%	0.5%	1%	2%	3%	0.5%	1%	2%	3%	0.5%	1%	2%	3%			
	NaOH				NaOH				NaOH				NaOH						
3343	3344	3349	3328	3338	3347	3349	3348	3351	3346	3344	3338	3347	3341	3344	3338	3347	3360	3500	O—H stretching
2918	2904	2899	2910	2920	2915	2917	2945	2882	2910	2904	2915	2892	2912	2904	2915	2892	2920	2900	C—H stretching vibration in cellulose and hemicelluloses
1536	1586	1594	1592	1592	1598	1596	1597	1584	1590	1586	1586	1594	1582	1586	1586	1594	1605	1740	C═O stretching of carboxylic acid and ester
1031	1029	1030	1025	1027	1030	1042	1039	1037	1033	1029	1029	1027	1032	1029	1029	1027	1045	–	C—O stretching of acetyl (lignin)

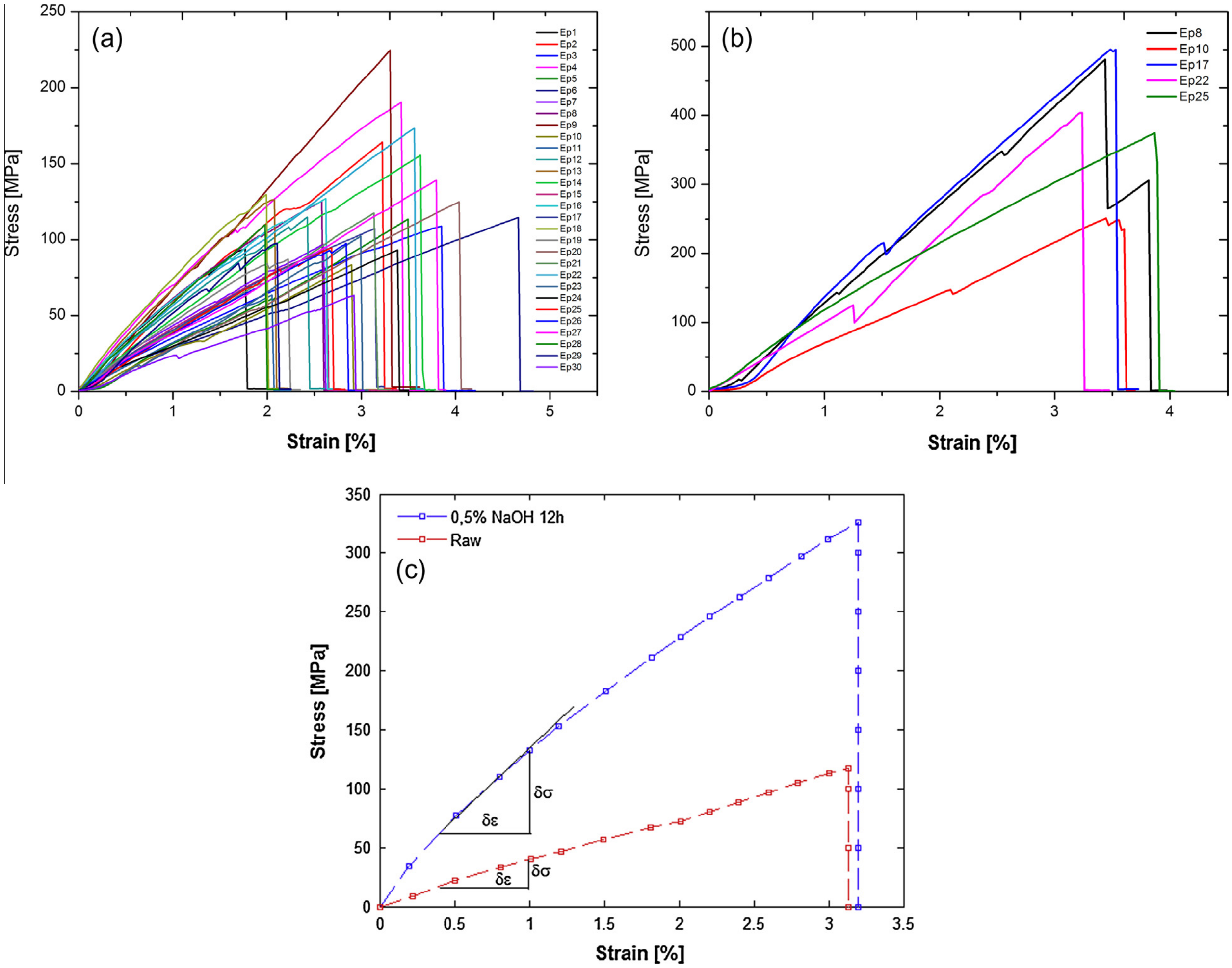

Fig. 5. Stress–strain curves (a) thirty samples testes and (b) stairs behaviour of FBBPD technical fibres untreated. (c) Comparison of stress–strain curves FBBPD technical fibres untreated and treated at 0.5% NaOH 12 h. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 2
Mechanical properties and statistical Weibull parameter for untreated and treated of the extracted FBBPD technical fibres.

	Experimental results				Weibull 2P-UTS		Weibull 3P-UTS			Weibull 2P-E		Weibull 3P-E		
	D_{moy} (μm)	σ (MPa)	ε (%)	E (GPa)	m	σ_0 (MPa)	m	σ_0 (MPa)	σ_u (MPa)	m	E_0 (GPa)	m	E_0 (GPa)	E_u (GPa)
Raw	577 ± 83	117 ± 35	3.13 ± 0.7	4.3 ± 1.4	4.45	128.23	2.42	80.73	45.68	3.73	4.78	1.76	2.85	1.82
0.5 NaOH 12 h	349 ± 73	328 ± 119	3.33 ± 0.6	11.6 ± 4.8	2.71	359.71	2.85	372.11	11.97	2.21	13.28	4.25	20.16	0.73
0.5 NaOH 24 h	468 ± 130	178 ± 78	3.95 ± 1.0	5.2 ± 3.0	2.67	198.16	1.70	149.30	45.20	1.47	5.90	2.28	7.05	1.12
0.5 NaOH 48 h	423 ± 87	199 ± 87	3.98 ± 1.9	5.4 ± 3.6	2.83	220.93	1.97	174.03	43.87	1.37	6.06	1.68	6.51	0.43
0.5 NaOH 72 h	441 ± 120	206 ± 88	3.52 ± 0.8	7.5 ± 3.8	2.65	230.74	2.49	221.73	8.58	2.29	8.40	2.24	8.29	0.11
0.5 NaOH 96 h	387 ± 91	233 ± 94	3.26 ± 0.7	8.8 ± 3.8	3.00	258.49	1.35	155.14	93.32	2.31	9.96	2.54	10.54	0.55
1 NaOH 12 h	357 ± 75	245 ± 89	3.13 ± 0.7	9.5 ± 3.3	3.36	270.77	1.99	189.80	76.79	2.96	10.62	5.93	17.16	0.48
1 NaOH 24 h	419 ± 99	276 ± 115	3.73 ± 0.7	9.2 ± 4.5	2.44	312.40	3.10	366.03	51.57	2.36	10.37	2.27	10.13	0.22
1 NaOH 48 h	390 ± 68	229 ± 72	3.88 ± 0.9	6.7 ± 3.7	3.47	254.67	4.11	287.69	32.45	1.44	7.78	2.47	9.72	1.90
1 NaOH 72 h	443 ± 87	227 ± 98	3.32 ± 0.6	7.9 ± 3.2	2.60	253.71	1.72	196.83	52.72	2.84	8.86	2.34	7.77	1.04
1 NaOH 96 h	394 ± 79	222 ± 87	3.76 ± 1.0	7.8 ± 2.7	3.07	246.89	1.50	154.89	84.49	3.25	8.70	2.38	7.00	1.62
2 NaOH 12 h	383 ± 69	234 ± 90	4.19 ± 1.6	7.2 ± 3.4	2.41	261.70	1.55	200.40	55.37	2.72	9.14	1.13	5.47	3.22
2 NaOH 24 h	431 ± 113	235 ± 117	3.41 ± 0.7	8.2 ± 3.6	3.38	242.45	2.56	199.60	41.23	2.91	8.79	1.98	6.83	1.84
2 NaOH 48 h	407 ± 138	231 ± 100	3.76 ± 1.0	8.1 ± 3.9	2.37	260.84	2.15	244.57	15.05	1.59	9.40	3.37	13.58	4.10
2 NaOH 72 h	423 ± 133	229 ± 117	3.33 ± 0.9	8.2 ± 4.2	2.55	252.73	1.22	158.20	82.02	2.15	9.23	1.82	8.36	0.79
2 NaOH 96 h	431 ± 99	220 ± 76	3.36 ± 1.0	8.0 ± 3.1	2.34	267.74	5.47	476.73	106.11	2.01	8.25	2.27	8.83	0.55
3 NaOH 12 h	336 ± 69	292 ± 105	3.73 ± 0.7	9.4 ± 3.4	3.66	320.02	1.94	201.67	111.36	3.26	10.48	2.91	9.69	0.77
3 NaOH 24 h	385 ± 88	237 ± 105	3.67 ± 1.0	7.7 ± 3.4	2.48	267.41	2.22	250.22	16.30	2.39	8.72	2.26	8.44	0.27
3 NaOH 48 h	354 ± 52	224 ± 101	4.04 ± 1.0	7.1 ± 3.5	2.44	251.54	1.81	209.96	38.52	1.72	8.22	3.64	12.73	1.35
3 NaOH 72 h	336 ± 70	268 ± 88	3.55 ± 1.1	10.1 ± 3.4	4.08	293.02	1.66	152.80	131.47	3.85	11.10	1.92	6.76	4.12
3 NaOH 96 h	405 ± 86	220 ± 78	3.09 ± 0.7	9.2 ± 3.6	3.46	243.76	3.14	227.26	16.01	2.58	10.38	4.10	13.97	1.53

technical fibres. The ANOVA analysis has been performed with a level of significance $\alpha \leqslant 0.05$ to discriminate between the different fibre groups [35].

3. Results and discussions

3.1. Morphology analysis (SEM)

Some observations by scanning electron microscope (SEM) of the longitudinal topographic surface of the palm technical fibres before and after the alkali treatment are shown in Fig. 3a–d. There is an evident difference in morphology of the surface between treated and untreated reinforcements. The untreated technical fibre surface is covered with a white organic material (lignin), created mainly during the extraction process (Fig. 3a). Fig. 3b and c are related to the technical fibres treated with 0.5% and 1% NaOH for 12 h. The surfaces of the treated technical fibres appear to be smoother than in the untreated case, with less asperities provided by the lignin formations. The technical fibre treated with 3% NaOH for 12 h shows an even more uniform surface finish (Fig. 3d). This particular state of surface of the technical fibre has been already observed by Alawar et al. [19] in natural palm date fibres surrounding the date palm leaves and treated with a 2.5% NaOH solution.

3.2. Infrared spectroscopy (FTIR)

Spectra from the Fourier transforms of untreated and treated technical fibres are shown in Fig. 4a and b. The main IR bands corresponding to the vibrations of different groups are reported in Table 1. Fig. 4a show a broadband spectrum around 3340 cm^{-1} mainly due to the OH groups existing in the structure of the technical fibres. There is a noticeable dependence of the transmittance on the concentration of the chemical treatment, and the peaks of untreated technical fibres are the largest, while the ones corresponding to a 3% NaOH treatment have the lowest amplitude, confirming that the chemical treatment reduces the effect and number of OH links. The peak at 2920 cm^{-1} corresponds to the vibrations of the aliphatic chains C—H. One can also observe the presence of peaks at 1590 cm^{-1} and 1030 cm^{-1}, which indicate the existence of the double bonds C═O and simple bond C—O, respectively. The concentration of NaOH has practically no effect on the position of the peaks, with very little variation in intensity (Fig. 4b). Taha et al. [20] have already observed that palm date fibres the spadix stem treated with a solution of soda (5% during 24 h at 23 °C) have a similar spectra to those found in this study. Also Alawar et al. [19] show that natural fibres surrounding the palm date leaves and treated by a soda solution with different concentrations does not exhibit a significant variation of their FTIR bands before and after treatment.

3.3. Technical fibre testing

The FBBPD technical fibres tested under tensile static loading have been grouped into 21 series of 30 specimens each. Fig. 5a represents the stress–strain curves of 30 tests carried out on untreated FBBPD fibres. The curves show a quasi-linear variation of the stress

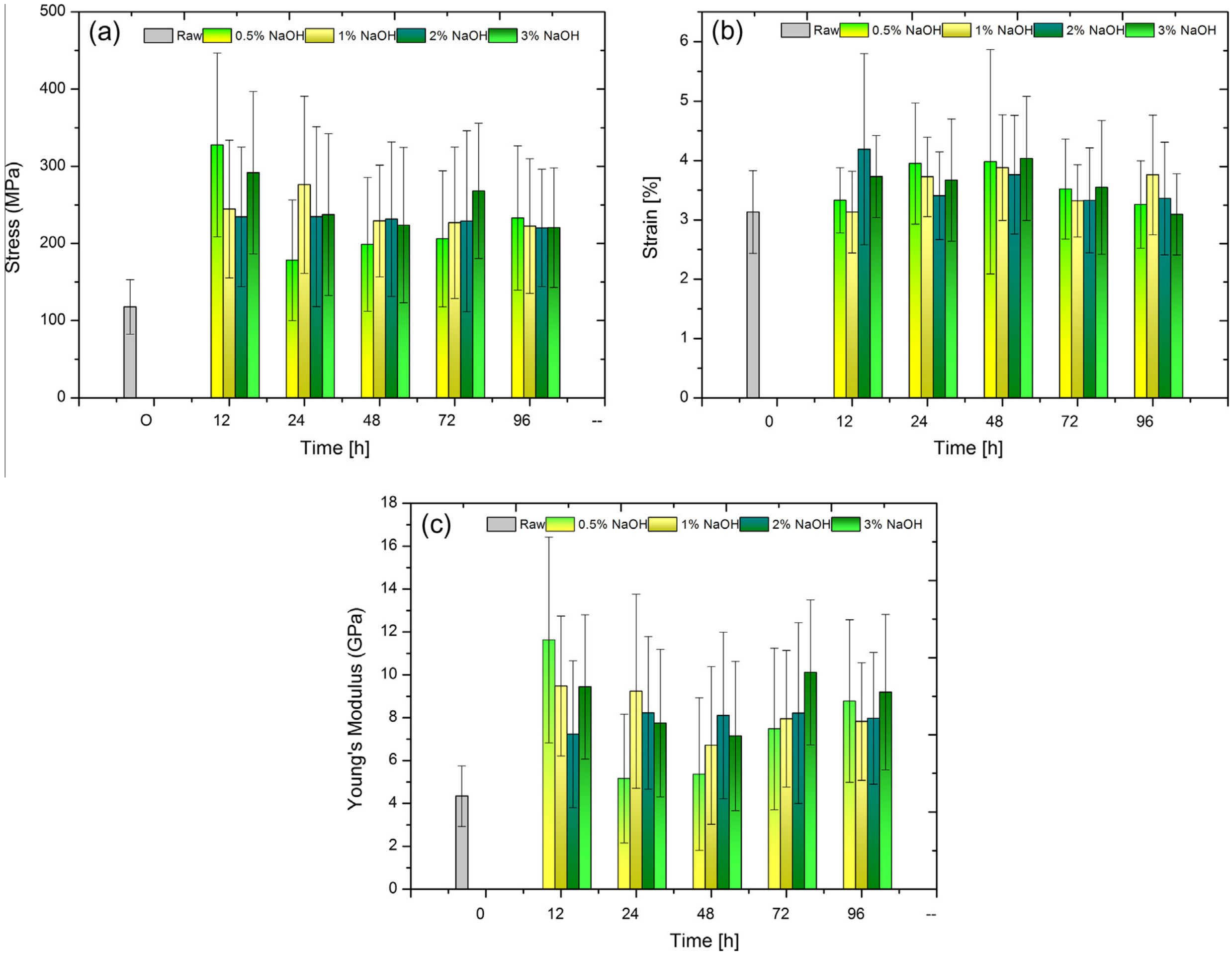

Fig. 6. Effect of NaOH treatment on (a) stress at failure, (b) strain at failure and (c) Young's modulus of FBBPD technical fibres. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

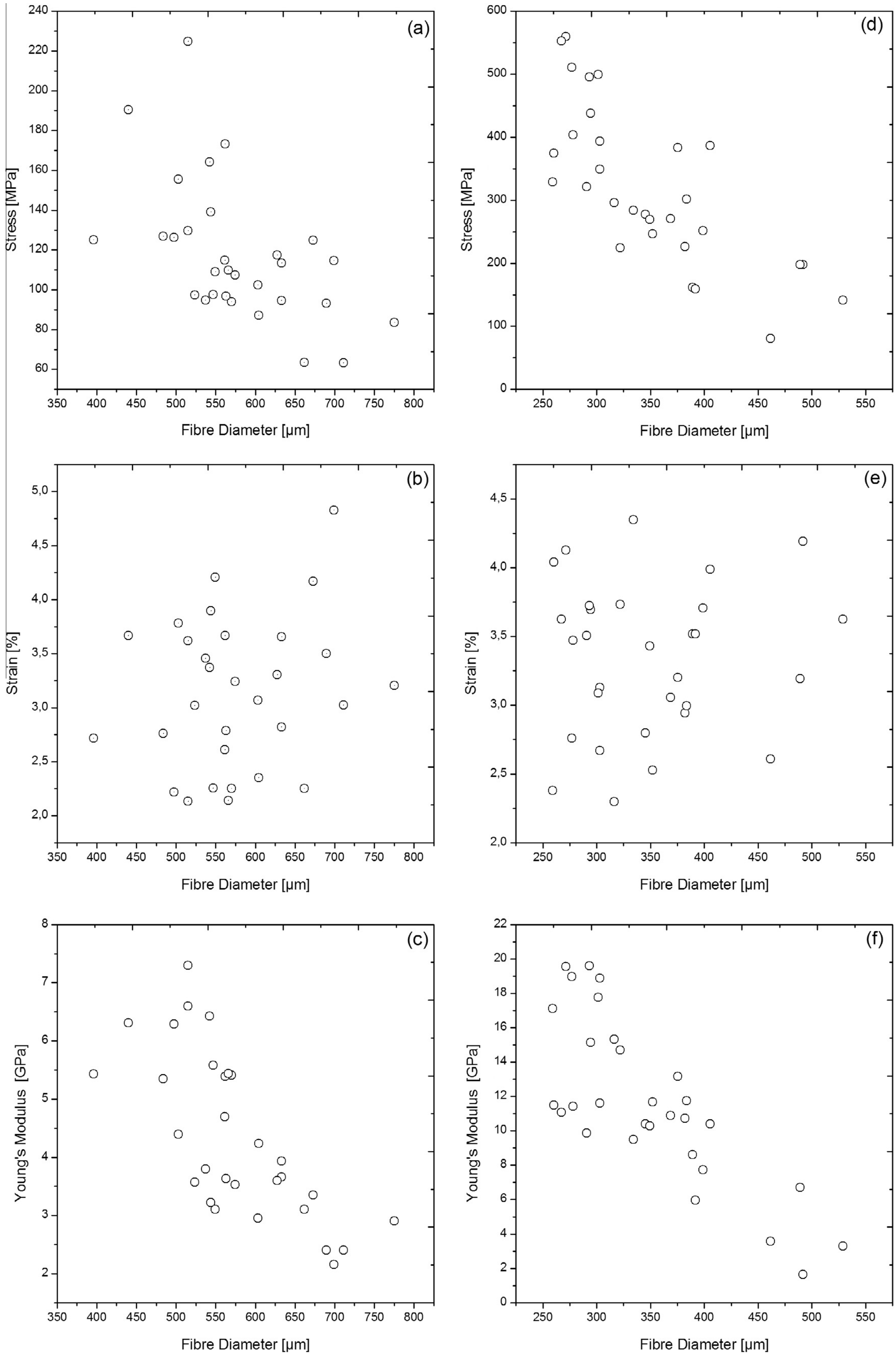

Fig. 7. Dependence of the tensile properties of the FBBPD technical fibres extracted (a–c) untreated and (d–f) treated at 0.5% NaOH for 12 h.

for increasing strains until a maximum value corresponding to a catastrophic failure. It is also important to note that for certain technical fibres the behaviour of stress–strain curves assumes a staircase form (Fig. 5b). This phenomenon is characterized by a sharp fall of the stress without complete failure of the sample that can occur one or several times during the load history before failure. Silva et al. [36] already described this type of behaviour as being caused by the collapse of the weak walls of primary cells in the technical fibre structure, as well as by the delamination between the fibrils. The stress–strain curves of untreated and treated FBBPD technical fibres (the latter with a concentration of 0.5% NaOH for 12 h) are represented in Fig. 5c. We notice that the treated fibres have higher modulus and stress at failure compared to the untreated ones, however the strain at failure is not affected by the type (or lack) of chemical treatment.

Table 2 illustrates the mechanical properties of the technical fibre series tested in this work. The significant dispersions observed in the mechanical data could be ascribed to three main factors [36]: (i) the testing parameters, (ii) the fibre characteristics and (iii) the measurement of the fibres sections. The test parameters that could influence the results are the accuracy of the instrumentation used, the length measurement, deformation speed, type of machine grips used and overall setup of the tensile equipment. The characteristics of the fibre (age, type of treatment and microstructure configuration) do also play a role. The effect of the presence of the alkali treatment is clearly visible (Table 2), and gives a noticeable increase of the mechanical properties in all the treated specimens, however the enhancement is dependent on the concentration of the chemicals used (NaOH), and on the duration of the treatment. Untreated technical fibres have an average Young's modulus of 4.33 GPa and tensile strength of 117 MPa. On the other hand, treated technical fibres having the best increase in the mechanical characteristics are obtained for technical fibres treated with 0.5% NaOH for 12 h and the recorded increases for the stress and Young's modulus are respectively 178% and 167%. It is also important to note that the dispersions are more important for the treated fibres.

Fig. 6a–c represents the variation of the mechanical properties (stress and strain at failure and Young's modulus) of the FBBPD technical fibres untreated and treated for the different concentrations and times of treatment considered in this work. The technical fibres treated for a period of 12 h with 0.5% of NaOH show the best mechanical behaviour, with an average value of the tensile strength equal to 327.7 MPa and Young's modulus of 11.6 GPa. These values represent increase of 178% and 167% against the corresponding values of the untreated fibres. It should be also noted that in the work of Al-Khanbashi et al. [22] the treatment of the mesh (natural mat) surrounding the date palm tree fibres with NaOH at concentration of 5% at 100 °C and for a duration of 2 h has shown an improvement of the tensile strength against the untreated fibre case, but limited to 45% only. The stress at failure and Young's modulus appear to be strongly dependent upon the diameter, and both engineering constants decrease with the increase of the fibre cross-section. It is also important to note that the chemical treatments allow decreasing the diameters of the fibres.

Fig. 7a–f represents the distribution of stresses and strains at failure and the Young's modulus of the untreated FBBPD technical fibres against the diameter. The data associated to the analogous best results obtained using chemical treatment (0.5% NaOH for 12 h) are also presented. These figures show clearly that the mechanical properties are strongly influenced by the diameter of the technical fibre, similarly to other natural reinforcements such as hemp [37], flax [28] and coir fibre [38]. These data suggest, however, that a statistical analysis is also necessary to better understand the dependence of the mechanical properties against the geometry and manufacturing parameters considered in this work.

Table 3 shows a comparison between the mechanical properties of the technical fibres obtained in this work and data related to natural reinforcements available in open literature [4,19,20,22,24,25,39–42]. The average mechanical properties of the FBBPD technical fibres developed in this work with gage length (*GL*) equal to 50 mm are 117 MPa for the tensile strength and 4.3 GPa for the Young's modulus. The elastic stiffness of the FBBPD considered in this work compares well with the one of sisal fibre at 20 mm *GL* [4], but it is 4 times lower than the one observed for sisal fibre for *GL* 20 mm [4]. Artichoke fibres with *GL* = 10 mm have 55% higher strength than the fibres considered in this work [25]. However, the FBBPD technical fibres have similar tensile strength to the one measured by Bezazi et al. in agave Americana L fibres at *GL* = 40 mm produced via earth burying (132 MPa) [42]. The strain to failure is also almost equal to 1.6 times to the one measured by De Rosa et al. for GL of 10 mm in okra fibres [39,40].

3.4. Statistical analysis

The mechanical properties have been statistically analysed using the Minitab software (version 15) using the Weibull statistics with two and three-parameters. The cumulative distribution function for the three-parameters Weibull distribution is defined by:

Table 3
Comparison values of mechanical properties and Weibull 2 parameters results obtained in this work and data available in open literature.

Fibres	Type	GL (mm)	Experimental results				Weibull distribution parameters				Refs.
			D_{moy} (μm)	σ (MPa)	ε (%)	E (GPa)	m_σ	σ_0 (MPa)	m_E	E_0 (GPa)	
FBBPD	Raw	50	577	117	3.13	4.3	4.45	128.23	3.73	4.78	Present work
FBBPD	0.5% NaOH 12 h	50	349	328	3.33	11.6	2.71	359.71	2.21	13.28	
DPF[a]	Raw	50	100–1000	58–203	5–10	2–7.5	–	–	–	–	[19]
DPSS	2% NaOH 2 h	20	–	650	5.4	14	–	–	–	–	[20]
DPSS	5% NaOH 2 h	20	–	540	4	17	–	–	–	–	[20]
DPF[b]	Raw	10	100–1000	170–275	5–10	5–12	–	–	–	–	[22]
Sisal	Raw	20	240	462	7.83	7.47	–	–	–	–	[4]
Hemp	Raw	10	40	285	2.2	14.4	2.86	260	–	–	[24]
Artichoke	Raw	10	300	182	2.8	10.7	3.71	201	4.47	11,62	[25]
Okra	Raw	10	88.3	270	1.9	12.8	1.90	281.68	2.02	16.5	[39,40]
P. tenax	Raw	40	120	465	2.26	27.59	2.23	469.19	–	–	[41]
Agave Americana L.	Earth	40	265	142	25.60	2.14	2.35	133.43	2.00	3.28	[42]
Agave Americana L.	Water	40	239	132	33.29	1.83	1.93	128.37	2.02	2.47	[42]

(Fibres from the mesh (natural mat) surrounding the date palm tree) – DPSS (fibres extracted from the stem spadix of palm date) – earth (the agave leaves are buried in earth for 90 days) – water (immersing in water for 10–13 days in a container) – sisal (fibreobtained using industrial method: decortications) – raw: untreated fibres.

[a] FBBPD (technical fibres extracted from fruit bunch branch of date palm) – DPF.

[b] (Fibres surrounding the date palm leaves) – DPF.

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

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 ($\chi_\mu = 0$) [43].

Figs. 8 and 9 show respectively the distribution for the stress and strain at failure and the Young's modulus of the technical fibres untreated and treated at 0.5% NaOH for a period of 12 h. The Weibull modulus m of two and three-parameter is determined by a linear equation (Least Square estimation *LS*), which is the slope of the curve. We note that the correlation coefficient R^2 obtained by the probability of the three-parameter Weibull models is best compared to the two parameters Weibull representations. For example, when observing Fig. 8e and f one can notice that the probability of the two parameters Weibull for the Young's modulus of the untreated technical fibre ($R^2 = 0.969$) is close to the one observed in the three parameters representation ($R^2 = 0.990$). In a similar way it is possible to determine the module (m), as well as making a comparison between the different hypotheses related to the nature of the experimental results. Figs. 8 and 9 allow comparing the two types of probabilities (2-parameters and 3-parameters). The module and the stress characteristic σ_0 of a

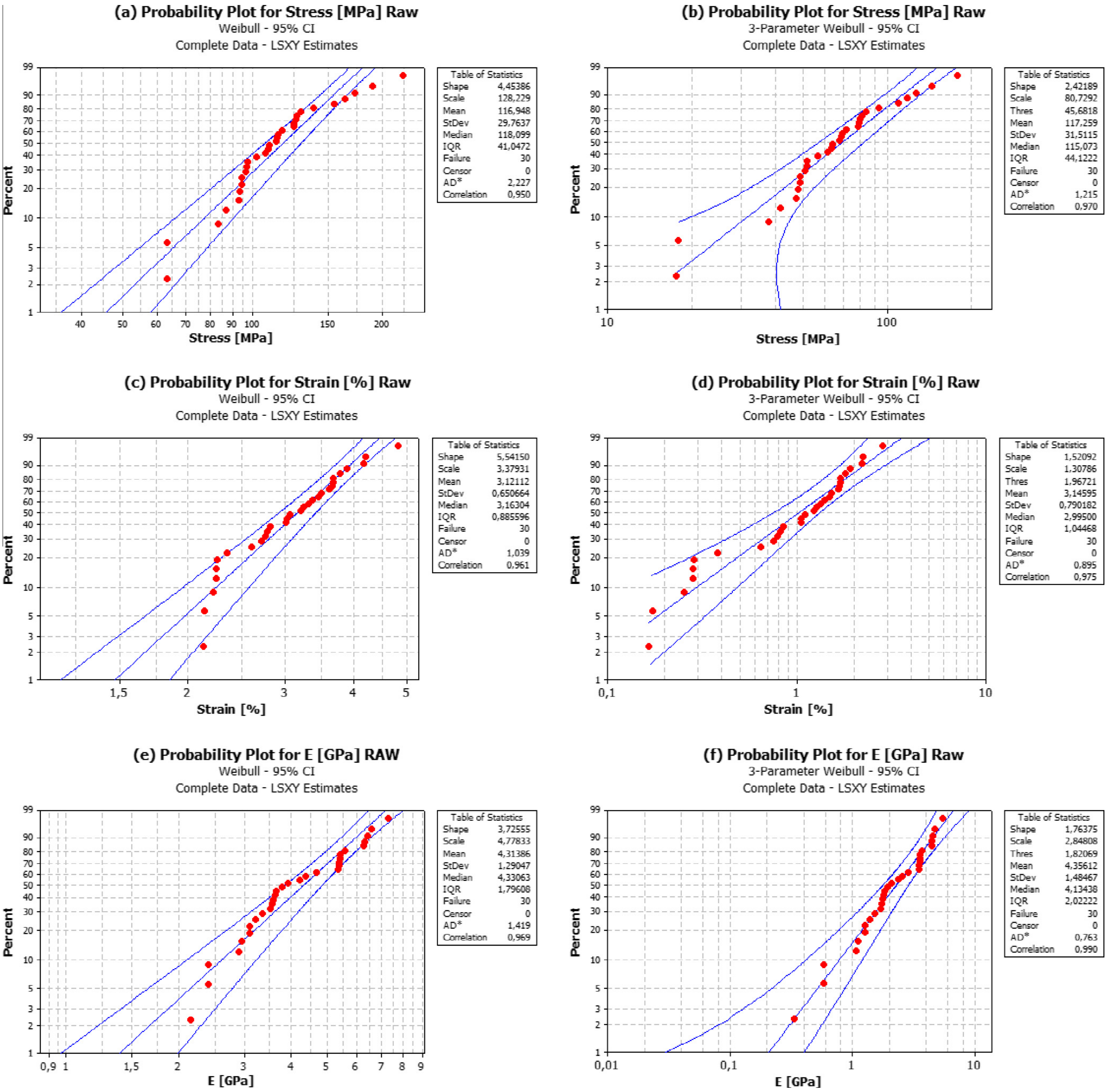

Fig. 8. Weibull distribution of mechanical proprieties of FBBPD technical fibres untreated (a, c, e) two-parameter and (b, d, f) three-parameter. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

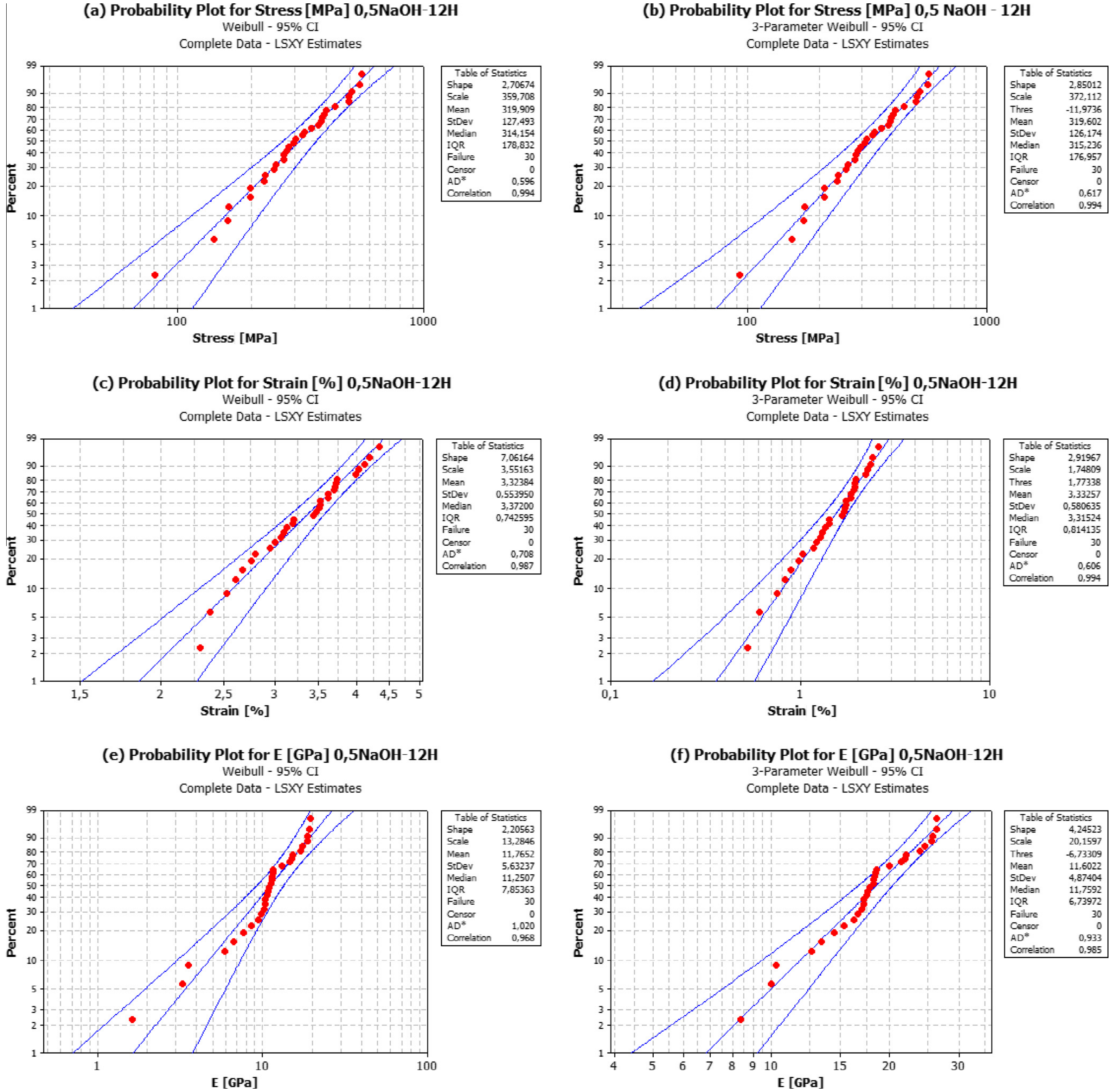

Fig. 9. Weibull distribution of mechanical proprieties of FBBPD technical fibres treated with a 0.5% NaOH for period 12 h (a, c, e) two-parameter and (b, d, f) three-parameter. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

two-parameter Weibull for the untreated FBBPD technical fibres are equal to 4.45 and 128.23 MPa respectively. For the three-parameter Weibull module, we find that m is 2.42 and $\sigma_0 = 80.73$ MPa. It is worth noticing that the two-parameter Weibull distribution is the most appropriate for the estimation of the stress of the FBBPD untreated and treated (0.5% NaOH 12 h) technical fibres, since the results obtained by this method (128 MPa and 359 MPa respectively) are closer to those obtained experimentally (117 MPa and 328 MPa) (Table 2). FBBPD technical fibres also show significantly higher values of the Weibull parameters ($m_\sigma = 4.45$) compared to the ones from other sources like artichokes ($m_\sigma = 3.7$) [25]. P. Tenax fibres have m_σ values ($m_\sigma = 2.23$) twice lower than the FBBPD technical fibres presented in this work [41]. Results from the analysis of variance are shown in Table 4. The independent variable in the analysis is the concentration of the NaOH groups, while dependent variables are stress at failure and the Young's modulus. The F (Fisher) factor for the stress at failure and Young's modulus is equal to 5.515 and 6.844, respectively. However, the critical values of the factors F are equal to $F_{crit} = 1.588$ and 1.587; therefore in this case, the average of the different groups are not equal. It is also worth of notice that the F factor for the strain at failure is 3.339, against a value of $F_{crit} = 1.588$. Because $F > F_{crit}$ it is possible to conclude that the chemical groups do have a statistical significant impact on the stresses at failure and the Young's modulus of these technical fibres and – more in general – on their mechanical behaviour.

Table 4
One way ANOVA for mechanical properties treated and untreated FBBPD technical fibres at a 0.05 level of significance.

Source	SS	Dof	MS	F	Probability	F_{crit}
ANOVA test for stress of 21 samples						
BG	987831.448	20	49391.5724	5.51534953	3.8738E−13	1.58782606
WG	5453773.58	609	8955.29324			
Total	6441605.03	629				
ANOVA test for strain of 21 samples						
BG	63.3649207	20	3.16824604	3.33943226	1.5083E−06	1.58782606
WG	577.781397	609	0.94873793			
Total	641.146318	629				
ANOVA test for Young's modulus of 21 samples						
BG	1723.76955	20	86.18847741	6.84434818	2.9937E−17	1.587826058
WG	7668.92352	609	12.59264947			
Total	9392.69307	629				

BG: between group; WG: within group; Dof: degree of freedom; SS: sum of squares; MS: mean square.
F: F-test for ANOVA-one way; F_{crit}: F critique.
Number of observations = 630; Number of samples = 21.

4. Conclusions

This work describes the manufacturing process and characterization of the mechanical properties of a novel class of natural technical fibres obtained from fruit bunch branches of palm dates. The extraction of these technical fibres requires some delicate handling to obtain long and undamaged technical fibres. However, the tensile tests show clearly that using post-processing NaOH-based chemical treatments does allow increasing significantly the stress at failure and the Young's modulus with a low influence on strain at failure (up to 178% for the stress and 167% for the Young's modulus compared to the pristine fibres). Similar increases have been observed in natural fibres extracted with chemical processes, and it is remarkable that in this case the technical fibres from the fruit bunch branches have been produced using a sustainable manufacturing process (water plus mechanical decortication). More significantly, the enhancement of the mechanical properties can be obtained with a small quantity of chemical, therefore reducing any possible environmental impact. It is also worth observing that the alkaline chemical treatment improves the structure of the fibre, which in turns may significantly improve the fibre/matrix bonding. The Weibull statistics performed also show that the two-parameter model leads to satisfactory estimations of the modulus and stress characteristic, and more sophisticate 3-parameters approaches are not necessary. The ANOVA analysis has also been instrumental in detecting the statistical contribution of the use of the NaOH groups on the mechanical properties of the FBBPD, with small error probabilities. The technical fibres from fruit bunch branch palm dates can therefore be potentially considered as reinforcement for thermo sets or thermoplastics and sustainable biocomposites.

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References

[1] Satyanarayana KG, Sukumaran K, Mukherjee RS, Pavithran C, Pillai SGK. Natural fibre-polymer composites. Cement Concr Compos 1990;12:117–36.
[2] Yousif BF, Shalwan A, Chin CW, Ming KC. Flexural properties of treated and untreated kenaf/epoxy composites. Mater Des 2012;40:378–85.
[3] Shah AN, Lakkad SC. Mechanical properties of jute-reinforced plastics. Fibre Sci Technol 1981;15:41–6.
[4] Belaadi A, Bezazi A, Bourchak M, Scarpa F. Tensile static and fatigue behaviour of sisal fibres. Mater Des 2013;46:76–83.
[5] Islam MS, Pickering KL, Foreman NJ. Influence of alkali treatment on the interfacial and physico-mechanical properties of industrial hemp fibre reinforced polylactic acid composites. Composites: Part A 2010;41:596–603.
[6] Baley C. Analysis of the flax fibres tensile behaviour and analysis of the tensile stiffness increase. Composites: Part A 2002;33:939–48.
[7] El Sawi I, Fawaz Z, Zitoune R, Bougherara H. An investigation of the damage mechanisms and fatigue life diagrams of flax fiber-reinforced polymer laminates. Mater Sci 2014;49:2338–46.
[8] Hachemane B, Zitoune R, Bezzazi B, Bouvet C. Sandwich composites impact and indentation behaviour study. Composites: Part B 2013;51:1–10.
[9] Mir A, Zitoune R, Collombet F, Bezzazi B. Study of mechanical and thermomechanical properties of jute/epoxy composite laminate. Reinf Plast Comp 2010;29:1669–80.
[10] El Sawi I, Bougherara H, Zitoune R, Fawaz Z. Influence of the manufacturing process on the mechanical properties of flax/epoxy composites. Biobased Mater Bioenergy 2014;8:69–76.
[11] Sreekala MS, Kumaran MG, Joseph S, Jacob M, Thomas S. Oil palm fibre reinforced phenol formaldehyde composites: influence of fibre surface modifications on the mechanical performance. Appl Compos Mater 2000;7:295–329.
[12] Malainine ME, Dufresne A, Dupeyre D, Mahrouz M, Vuong R, Vignon MR. Structure and morphologyof cladodes and spines of Opuntiaficus-indica. Cellulose extraction and characterization. Carbohydr Polym 2003;51:77–83.
[13] Reddy N, Yang Y. Biofibers from agricultural byproducts for industrial applications. Trends Biotechnol 2005;23:22–7.
[14] Zhang LL, Zhu RY, Chen JY, Chen JM, Feng XX. Seawater-retting treatment of hemp and characterization of bacterial strains involved in the retting process. Process Biochem 2008;43:1195–201.
[15] Alvarez VA, Ruseckaite RA, Vazquez A. Degradation of sisal fibre/mater Bi–Y biocomposites buried in soil. Polym Degrad Stab 2006;91:3156–62.
[16] Amel BA, Paridah MT, Sudinb R, Anwarb UMK, Husseinc AS. Effect of fiber extraction methods on some properties of kenafbastfiber. Ind Crops Prod 2013;46:117–23.
[17] Baley C, Busnel F, Grohens Y, Sire O. Influence of chemical treatments on surface properties and adhesion of flax fibre–polyester resin. Composites: Part A 2006;37:1626–37.
[18] Belkhir S, Koubaa A, Khadhri A, Ksontini M, Nadji H, Smiti S, et al. Seasonal effect on the chemical composition of the leaves of Stipa tenacissima L. and implications for pulp properties. Ind Crops Prod 2013;44:56–61.
[19] Alawar A, Hamed AM, Al-Kaabi K. Characterization of treated date palm tree fiber as composite reinforcement. Composites: Part B 2009;40:601–6.
[20] Taha I, Steuernagel L, Ziegmann G. Optimization of the alkali treatment process of date palm fibres for polymeric composites. Compos Interfaces 2007;14:669–84.
[21] Abdei-rahman HH, Ai-juruff R, Ahmad F, Alam I. Physical, mechanical and durability characteristics of date palm frond stalks as reinforcement in structural concrete. Cem Compos Lightweight Concr 1988;10:175–81.
[22] Al-Khanbashi A, Al-Kaabi K, Hammami A. Date palm fibers as polymeric matrix reinforcement. Polym Compos 2005;26:604–13.

[23] Silva FA, Chawla N, Filho RDT. An experimental investigation of the fatigue behavior of sisal fibers. Mater Sci Eng A 2009;516:90–5.

[24] Placet V. Characterization of the thermo-mechanical behaviour of Hemp fibres intended for the manufacturing of high performance composites. Composites: Part A 2009;40:1111–8.

[25] Fiore V, Valenza A, Di Bella G. Artichoke (Cynara cardunculus L.) fibres as potential reinforcement of composite structures. Compos Sci Technol 2011;71:1138–44.

[26] Zafeiropoulos NE, Baillie CA. A study of the effect of surface treatments on the tensile strength of flax fibres: Part II. Application of Weibull statistics. Composites: Part A 2007;38:629–38.

[27] Kompella MK, Lambros J. Micromechanical characterization of cellulose fibers. Polym Test 2002;21:523–30.

[28] Andersons J, Sparnins E, Joffe R, Wallström L. Strength distribution of elementary flax fibres. Compos Sci Technol 2005;65:693–702.

[29] AL-Oqla Faris M, Sapuan SM. Natural fiber reinforced polymer composites in industrial applications: feasibility of date palm fibers for sustainable automotive Industry. Clean Prod 2014;66:347–54.

[30] ABDOUCHE F. Communication from the Botanic Unit Development at the ministry of agriculture and rural development, Algerian Press Service, Alger; 30 March 2010.

[31] Alaoui SB. Référentiel pour la conduite technique du palmier dattier. In: Si Bennasseur Alaoui et AjiroYasuhei, editor. Référentiel de conduite technique des principales cultures au maroc; 2005. p. 102–12.

[32] Agence Nationale des Ressources Hydrauliques (ANRH) M'sila Algérie. Rapport ClimatologiqueAnnuel, M'sila; 2014.

[33] Franck Robert R. Bast and other plant fibres. Woodhead Publishing Limited and CRC Press LLC; 2005.

[34] Bendahou A, Habibi Y, Kaddami H, Dufresne A. Physico-chemical characterization of palm from phoenix Dactylifera-L., preparation of cellulose whiskers and natural rubber-based nanocomposites. J Biobased Mater Bioenergy 2009;3:1–10.

[35] Scheffé H. The analysis of variance. New York: John Wiley & Sons; 1999. Wiley Classic Library Edition.

[36] Silva FA, Chawla N, Filho RDT. Tensile behaviour of high performance natural (sisal) fibers. Compos Sci Technol 2008;68:3438–43.

[37] Placet V, Trivaudey F, Cisse O, Retel VG, Boubakar ML. Diameter dependence of the apparent tensile modulus of hemp fibres: a morphological, structural or ultra-structural effect. Composites: Part A 2012;43:275–87.

[38] Chollakup R, Smitthipong W, Kongtud W, Tantatherdtam R. Polyethylene green composites reinforced with cellulose fibers (coir and palm fibers): effect of fiber surface treatment and fiber content. Adhes Sci Technol 2013;27:1290–300.

[39] De Rosa IM, Kenny JM, Maniruzzaman M, Moniruzzaman Md, Monti M, Puglia D, et al. Effect of chemical treatments on the mechanical and thermal behaviour of okra (Abelmoschus esculentus) fibres. Compos Sci Technol 2011;71:246–54.

[40] De Rosa IM, Kenny JM, Puglia D, Santulli C, Sarasini F. Morphological and thermal characterisation of okra (Abelmoschus Esculentus) fibres as potential reinforcement inpolymer composites. Compos Sci Technol 2010;70:116–22.

[41] De Rosa IM, Kenny JM, Puglia D, Santulli C, Sarasini F. Tensile behavior of New Zealand flax (Phormium tenax) fibers. Reinf Plast Comp 2010;29:3450–4.

[42] Bezazi A, Belaadi A, Bourchak M, Boba K, Scarpa F. Novel extraction techniques, chemical and mechanical characterisation of Agave Americana L. natural fibres. Composites: Part B 2014;66:194–203.

[43] Johnson NL, Kotz S, Balakrishnan N. Continuous univariate distributions: wiley series in probability and statistics, 2nd ed., vol. 1. Wiley-Interscience Publication; 1994.