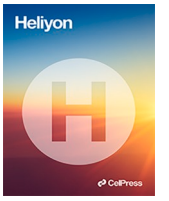
Heliyon 9 (2023) e17581

Contents lists available at ScienceDirect

Heliyon

journal homepage: www.cell.com/heliyon



Chemical extraction and its effect on the properties of cordleaf burbark (*Triumfetta cordifolia A. rich*) fibres for the manufacture of textile yarns

Anny Geraldo Soppie [a,b], Achille Desire Omgba Betené [a,c,*], Pierre Marcel Anicet Noah [a,c], Abel Emmanuel Njom [a,c], Fabien Betené Ebanda [a,c], Atangana Ateba [a,c], Armel Mewoli [a], Didimus Nkemaja Efeze [a], Roger Moukené [a]

[a] *Laboratory of Mechanics, Doctoral Training Unit in Engineering Sciences (UFD-SI), University of Douala, P.O. Box: 1872, Douala, Cameroon*
[b] *Department of Textiles and Clothing Industry, ENSET, University of Douala, P.O. Box: 1872, Douala, Cameroon*
[c] *Department of Mechanical Engineering, ENSET, University of Douala, P.O. Box: 1872, Douala, Cameroon*

ARTICLE INFO

Keywords:
Triumfetta cordifolia
Alkaline extraction
Textile fibres
Fibre characteristics
Fick's model

ABSTRACT

Tropical *Triumfetta cordifolia* (TC) fibre extracted from the equatorial region of Cameroon has been characterized as a potential fibre for textiles. An investigation of extraction parameters to soften this fibre is crucial to use it as a biobased material in the spinning process. To obtain textile quality fibres, 34 sodium hydroxide extraction tests were carried out to study the effect of extraction conditions on its characteristics. Thus, three levels of concentrations (0.5, 1.0 and 1.5 wt%), temperatures (80, 100 and 120 °C) and durations (120, 180 and 240min) were used for extraction by cooking, and at room temperature, durations of 120, 150 or 180 min with three concentrations (2.5, 3.0 and 3.5 wt%) were considered. Only 6 combinations produced fibres that were clear and soft to the touch, without defects (corrugations, stuck fibres) and without residual bark epidermis at the macroscopic scale. For these fibres, the dissolution of non-cellulosic substances, morphological, physical, thermal and mechanical properties depended on the austerity of the alkaline retting. Under mild conditions, the SEM surfaces of the fibres showed large residues of the middle lamella, which made the lignin content (10 wt%) and hydrophilic function higher. Under medium conditions, the fibre surfaces were clean and slightly wrinkled (at 80 °C; 120min). Under severe conditions, heterogeneous transverse shrinkage and wrinkling were observed and accompanied by cellulose degradation (39 wt%) with a significant reduction in tenacity at 16cN/tex. The medium extraction conditions were considered more effective, and their fibres showed cellulose content up to 49 wt%, density up to 1.39 g cm^{-3}, "Fickian" moisture absorption kinetics with saturation up to 11 wt%, thermal stability up to 237 °C, Young's modulus up to 3.7 GPa, tensile strength up to 113 MPa and tenacity up to 40cN/tex. These new results were compared with lignocellulosic textile fibres in the literature, showing similarity with banana, sisal and jute fibres.

* Corresponding author. Laboratory of Mechanics, Doctoral Training Unit in Engineering Sciences (UFD-SI), University of Douala, P.O. Box: 1872, Douala, Cameroon.

E-mail addresses: achille-desire.betene-omgba@insa-lyon.fr, achilbetene@gmail.com (A.D.O. Betené).

https://doi.org/10.1016/j.heliyon.2023.e17581
Received 3 January 2023; Received in revised form 21 June 2023; Accepted 21 June 2023
Available online 23 June 2023

1. Introduction

Yarns made from plant fibres are increasingly used to weave and knit a wide range of objects in many fields such as clothing, decoration, health, packaging industry or sports and leisure. These yarns combine softness, stretchability, tenacity, and lightness, but their performance depends mainly on the performance of the fibres [1,2]. Plant fibres are a biosourced raw material that is very abundant in nature. They are renewable, biodegradable, antistatic, porous and moisture-regulating, making them an attractive alternative to synthetic polyester and polyamide textile fibres. They generally have good specific properties. These are still rather dispersed, difficult to control and generally inferior to synthetic fibres, which limits their use [3,4]. However, the diversification of low environmental impact textile fibres is a key issue to replace synthetic fibres. The most used vegetable textile fibres (VTF) are cotton, flax, hemp and jute [5–7]. Some banana, sisal, pineapple and coconut fibres are also used. The low availability of these fibres due to a very low extraction yield (5–15 wt%) [8] is prompting researchers to investigate the possibility of using other lignocellulosic fibres. In addition, the idea of offering other types of lignocellulosic fibres such as *Triumfetta cordifolia*, *Triumfetta pentadra*, okra and *Urena lobata* is to reduce costs and greenhouse gas emissions due to the transportation of raw materials. However, the use of new VTF for the development of value-added textiles requires a better understanding and control of the extraction process and the properties induced by this process. In the case of chemical extraction, the effect of several parameters on fibre performance needs to be well controlled and understood [8–11].

Alkaline extraction with sodium hydroxide (NaOH) is the most used chemical extraction method [12,13]. In contrast to mechanical and biological extraction, chemical extraction allows for rapid extraction of the fibres, which represents a considerable energy saving. Specifically, the NaOH process allows for the simultaneous dissolution of non-cellulosic components such as hemicellulose, pectin and lignin, as well as other substances present in the epidermis of the raw material (bark or leaves). The dissolution of lignin is even more efficient when the NaOH solution is heated to a temperature above 75 °C [12,14,15]. This seems to be an interesting solution to produce spinnable and much less stiff textile fibres [5]. Among other things, extraction is considered efficient when significant removal of non-cellulosic material occurs without significant degradation of cellulose. To avoid cellulose degradation during the extraction process, the NaOH concentration, temperature and time should be defined according to the type of plant [4,5]. Elseify et al. [7] showed that, the alkaline extraction process effectively extracts long textile fibres with a low proportion of impurity in the date palm vein. The extraction was performed with NaOH concentrations of 1, 3 and 5%, with three temperature levels (25, 75 and 100 °C) and three-time levels (1, 2 or 3 h). The fibres were light (1.324 g cm^{-3}), and could withstand temperatures up to 226 °C and tensile loads of about 453 MPa. In Hasan et al. [8], 3 NaOH concentration levels (4, 7 and 10% w/v), 4 temperature levels (70, 80, 90 and 95 °C) and 5 time levels (4, 6, 8, 10 and 12 h) were used to extract the fibres from *Typha latifolia* leaf. The authors showed that using a concentration of 7% NaOH, a temperature of 90 °C and a time of 10 h resulted in fibres with a higher strength (168 MPa) and a lower moisture absorption capacity (8% by weight). Vinod et al. [13] reported that increasing the concentration of NaOH between 3 and 5 wt% during the chemical extraction of fibres from the Yucca elephantine plant resulted in a progressive decrease in cellulose content from 66 wt% to 57% and in tenacity from 5.7 cN/dTex to 3.8 cN/dTex. This decrease shows that NaOH degraded the crystalline structure of the fibre and altered its ability to withstand loads [16]. All these results on chemical extraction with NaOH are very instructive and show that fibre performance depends fundamentally on extraction conditions and the nature of the plant.

Triumfetta cordifolia (TC) or cordleaf burbark fibre is a bast fibre that belongs to the *Tiliaceae* family. Congo is the largest producer of TC fibre with about 260 kg/ha, followed by Equatorial Guinea [17]. The TC plant is grown in savannahs, fallows and riverbanks in the humid regions of tropical Africa. It is grown from its seeds or from leafy stem cuttings. The plant is an erect, slightly branched shrub with fragrant leaves, reaching 2.5–5 m in height [18]. All parts of the plant are used in traditional African medicine to treat burns, muscle pain, lung and stomach infections [18]. Its leaves are eaten as a vegetable and the wood from its stem is used in house construction or as fuel [18,19]. The fibres extracted from its stem bark are used in handicrafts to make strong ropes and twine, bowstrings, fishing lines and belts used for climbing trees and palms. They are also used to make baskets, coffee bags, mats, hammocks, and traditional dance costumes [17]. Like all lignocellulosic fibres already used in textile development, TC fibre is composed of cellulose (44 wt%), hemicellulose (31 wt%), lignin (9–19 wt%), pectin (3.3 wt%), extractives (3 wt%), wax (0.5 wt%), minerals (2 wt%) and water (8 wt%). This high proportion of non-cellulosic substances is likely to make it difficult for TC fibre to be spun and for dyes to attach to its surface [20,21].

Studies have been conducted to extract fibres from TC bark to identify their potential for reinforcement in composites. Senwitz et al. [19] immersed TC stem bark for 3 and 6 weeks in standing water to extract fibre bundles. The 6-week retting made the cellulose content higher in the fibres. Surprisingly, the tensile strength of these fibres was lower, and their stiffness was higher due to their higher lignin content. Grosser et al. [18] fabricated non-woven and unidirectional composites based on polyamide and TC bast fibres extracted after 3 weeks of water retting. It was reported that TC fibres have positive effects on the mechanical performance of composites in the same way as hemp fibres. In a previous study, Mewoli et al. [22] extracted TC fibres by immersing the stem bark in water for about 30 days. The extracted fibre bundles were processed by carding to individualise them for addition as reinforcing material in polyamide and polypropylene. It was observed that, the cross section of the fibre has an elliptical lumen, and its shape varies from circular to flat oval. In addition, the surface of the fibre is covered with a rough sheath composed of impurities, pectin and lignin, which makes the fibre rough, hard to the touch and stiff, thus seriously hindering its use in the design and development of yarns for clothing. In the literature, no studies have been done to extract the fibres from TC bark for use in common textiles.

In view of the above, this study proposes to carry out for the first-time alkaline extraction tests at room temperature and under heat. Extraction parameters that will effectively separate the fibres, making them soft to the touch without blackening or agglomerating them into a paste will be selected. The fibres associated with the selected parameters will be subjected to standard gravimetric tests to assess their density, water absorption and moisture absorption kinetics. In addition, their chemical composition will be assessed using

the industrial pulp and paper analysis technique. SEM and FTIR analysis will be used to analyse morphological and structural changes. Thermal behaviour will be analyzed by thermogravimetric tests (TGA). Mechanical properties will be determined by standard tensile tests.

2. Materials and methods

2.1. *Triumfetta cordifolia fibres*

2.1.1. *Plant material*

Triumfetta cordifolia (TC) stems (Fig. 1a and b), were collected in November, in the *Messock I* forest, *Mbankomo* district, Centre region (Cameroon). In this locality, the relative humidity is 85% and the average temperature is 23 °C. Alkali retting experiments by cooking and at room temperature were carried out in a 5000 ml digester, equipped with a WRN-130K thermocouple (Bastor, SS304 M27, China).

2.1.2. *Alkaline fibre extraction*

For alkaline cooking, three temperature levels (80, 100 and 120 °C) were used, with three mass concentration levels (0.5, 1 and 1.5 wt%) of sodium hydroxide solution (NaOH) and three-time levels (120, 180 and 240 min). Similarly, three-time levels (120, 150 and 180 min) and three concentrated solutions (2.5, 3 and 3.5 wt%) of NaOH were used for alkaline retting at room temperature ($T = 25 \pm 2$ °C). It is interesting to note that concentrations below 2.5 wt% had no defibrillating effect on the bark during the ambient alkaline retting. Temperature, time and concentration levels were chosen according to the processing conditions of the textile fibres [1,23].

The TC barks were placed in a freezer for 30 days, then defrosted in a conditioned medium ($T = 25$ °C, 50%RH) and cut to approximately 10 cm in length. For each extraction, a 12.5 g dry mass of bark was immersed in demineralised water for 60 min at room temperature (25 ± 2 °C) to soften the skin. The softened barks were then wiped with a cotton cloth to remove surface water, and placed in a digester containing a concentrated solution of NaOH (ratio of bark to alkaline solution is 1:40 w/v) previously heated to the target temperature. Cooking was carried out in a temperature-controlled electric water bath, while room alkaline retting was carried out in a room controlled for temperature (25 ± 2 °C) and relative humidity (50 ± 5%). The extraction was carried out at a fixed time *t*. The extracted fibres were washed in a concentrated solution of 1 wt% acetic acid for 10 min and subjected to several washes with lukewarm demineralised water (40–50 °C) to neutralise the dissolved substances and residual NaOH. The resulting fibres were dried in a vacuum oven at 80 °C for 24 h [22] and stored in sealed polyethylene bags.

2.1.3. *Fibre selection*

The physical appearances of some fibres and the fineness (2.9–6.2 tex) of all fibres are shown in Fig. 2(a–d). More details on the methodology for the determination of fineness by gravimetry are presented by Betené et al. [21].

It can be seen that the fineness and texture of the fibre depend on the extraction parameters (type, temperature, concentration, and time). The images show that the fibres are partially burnt (dark colour) and heaped (when $C = 1.5$ wt% for firing temperatures of 120 °C (Figs. 2d), and 100 °C at $t = 240$ min (Fig. 2c)), twisted, and partially composed of bark. In a textile application context, the fibres chosen (Table 1) are those that are individualised, visibly unidirectional and light in colour, and relatively soft to the touch. Table 1 also gives the extraction parameters and linear densities of these fibres. The fibres selected for alkaline room retting have a fineness in the range of 3.0–5.7 tex and the fibres obtained by alkaline firing have a fineness of 3.0–5.7 tex. The average length of these fibres is of the same order as that of the base bark (about 10 cm).

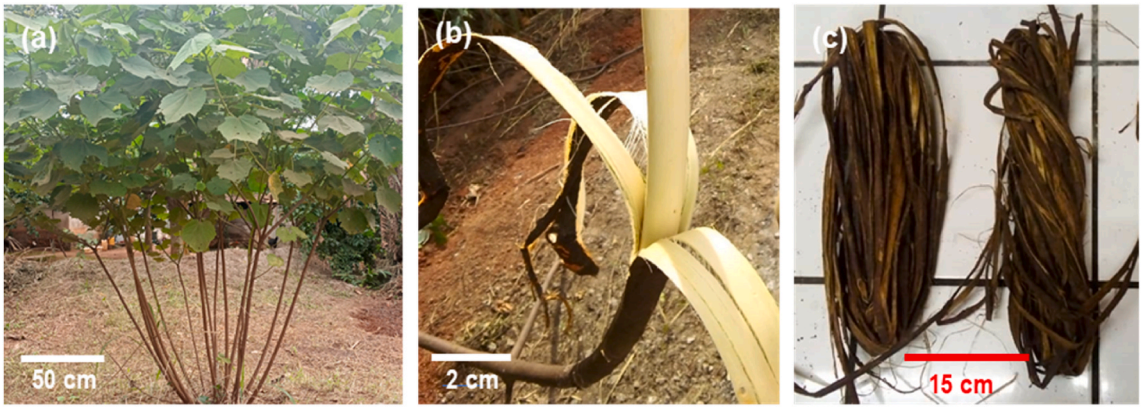


Fig. 1. (a) TC plant, (b) TC stem with partially detached bark and (c) TC bark after 30 days of freezer storage.

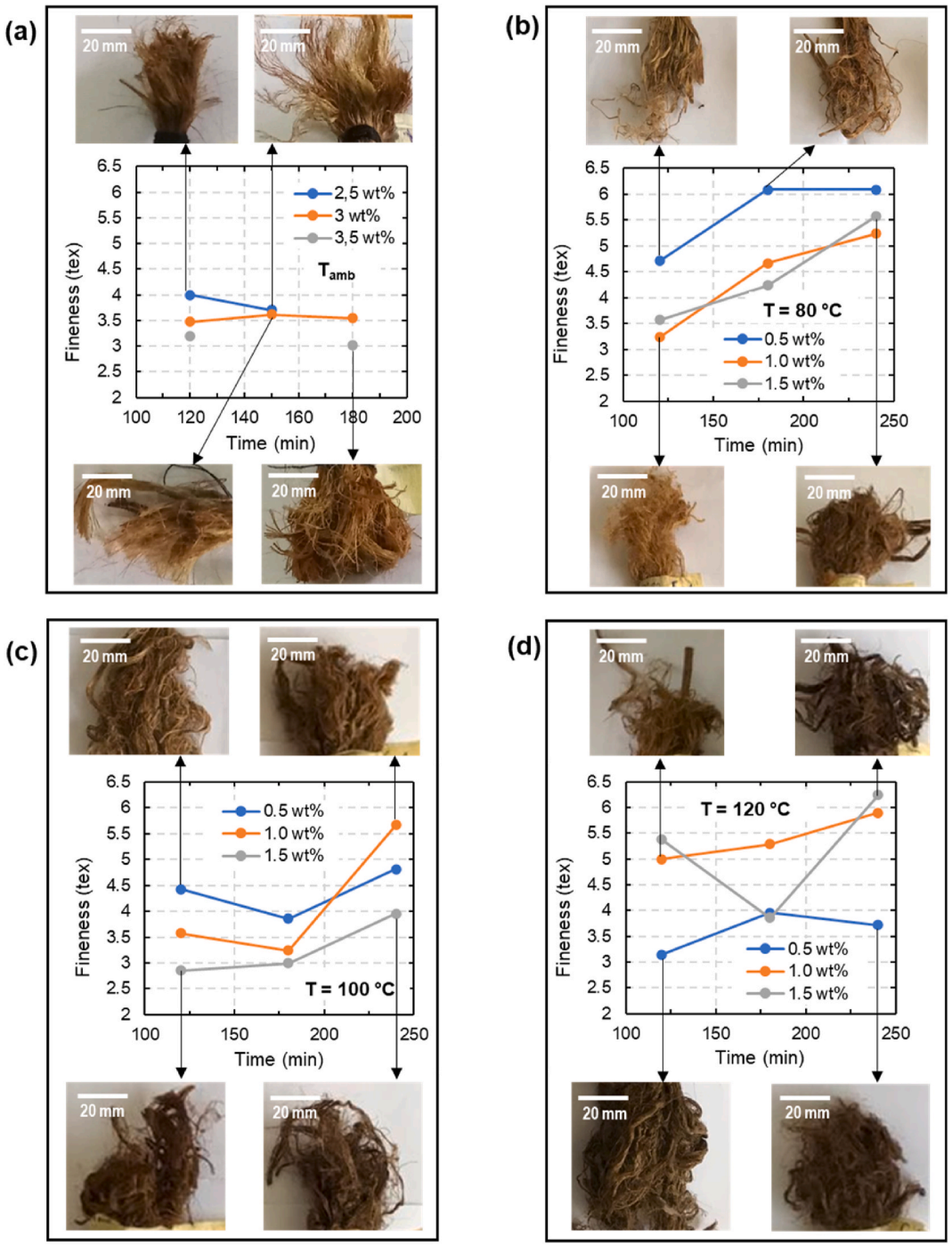


Fig. 2. Graphs showing the evolution of the fineness of TC fibres extracted by the alkaline method at (a) room temperature T_{amb} for 120, 150 and 180 min, and by firing at (b) 80 °C, (c) 100 °C and (d) 120 °C for durations of 120, 180 and 240 min. The attached images show the visual appearance of some of the resulting fibres.

2.2. SEM analysis

The morphology of the fibre surface was observed using a scanning electron microscope (HITACHI SEM, S-3500 N). Prior to observation, the fibres were coated with a thin layer of gold/palladium by sputtering. The images were taken on the longitudinal views at × 600 magnification.

Table 1
Coding, extraction parameters and fineness of the studied TC fibres.

Coding	Extraction	Austerity	Extraction parameters			Fineness (tex)
			Concentration (wt%)	Temperature (°C)	Time (min)	
F1	Ambient	Mild	3.5	25	120	3.2
F2	Ambient	Medium	2.5	25	150	3.7
F3	Ambient	Medium	3.5	25	180	3.0
F4	Cooking	Medium	1.0	80	120	3.2
F5	Cooking	Severe	1.0	100	240	5.7
F6	Cooking	Severe	1.5	100	180	3.0

2.3. Chemical composition

The estimation of the content of TC fibre constituents was carried out using the analytical technique for pulp and paper industry (TAPPI) [4,24,25]. It consists in isolating and quantifying (in mass percentage) successively the biochemical components of the fibre such as extractives, pectin, lignin, hemicellulose, and cellulose. In this analysis, 10 g/sample of ground fibres (average particle size 115 μm) were used and dried in a vacuum oven at 80 °C for 24 h. The method was performed by leaching in an ethanol-benzene solution (1:2 v/v) for 7 h to dissolve soluble extractables, such as waxes, proteins and lipids. After drying the resulting powder at 50 °C (for 1 h), heating under reflux with distilled water was carried out for 7 h to dissolve the residual extractables. The defatted (~3 mg) and dried powder (105 °C, 12 h) was then heated under reflux (with magnetic stirring) at 80 °C in a concentrated detergent solution of chloridric acid (2 wt%) for 4 h, resulting in the dissolution of pectin. To isolate the lignin, a few milligrams (~500 mg) of the pectin-free residue (dehydrated at 105 °C for 12 h) were treated according to the Klason method by hydrolysis in a concentrated sulphuric acid solution (72 wt%). An amount (~3 mg) of the same residue after pectin dissolution was then subjected to a detergent solution of acetic acid (1:5 v/v) and sodium chlorite (15%, v/v) to extract the holocellulose. This residue consisting mainly of holocellulose was then dissolved in an ethanol-nitric acid solution (1:4 v/v) by heating in a water bath for 1 h to remove the hemicelluloses. The residue obtained at each stage was weighed with a milligram balance to determine the mass fraction of the corresponding component. The hemicellulose content was calculated by subtracting the cellulose content from the cellulose content.

2.4. Determination of fibre diameter

A Bresser optical microscope (model Biolux NV 20x-1280×, France) was used to record images (magnification) of the longitudinal view of 10 mm long fibres. These images were used to measure the diameters of the fibres with the ImageJ software. Three measurements were made in the transverse direction for each of the fibres. This allowed the diameter distributions to be plotted, and the mean diameter to be determined using the normal and Weibull distributions.

2.5. Determination of density

A gravimetric method using a pycnometer, a 0.1 mg precision balance and toluene (density $\rho_T = 0.866$ g cm^{-3} at 25 °C) as immersion liquid was applied to estimate the density of the walls of each TC fibre. The fibres were first dried at 105 °C in a vacuum oven for 24 h [3,26], then cut into 5 mm long pieces and introduced into the pycnometer which will finally be filled by toluene. After 2 h of rest, the microbubbles were no longer visible on the surface of the fibres. By noting: m_0 the mass of the empty pycnometer, m_1 the mass of the pycnometer filled with the chopped fibres, m_2 the mass of the pycnometer filled with toluene, m_3 the mass of the pycnometer filled with chopped fibres and toluene solution, equation (1) [27] can be used to calculate the density of the fibres:

$$\rho_f = \rho_T \times \frac{m_1 - m_0}{(m_2 - m_0) - (m_3 - m_0)} \quad (1)$$

2.6. Evaluation of water absorption

A gravimetric method based on NF EN ISO 1097-6 was used to determine the water absorption of TC fibres. Before testing, the fibres were cut to 15 mm length and dried in an oven at 105 °C for 6 h. Three bundles of fibres of initial mass $m_0 = 1 \pm 0.1$ g were prepared and placed in a pycnometer. The pycnometer was then filled with distilled water. After 24 h of immersion, the samples were wiped with a cotton cloth to remove the water from the surface of the fibres. The final mass m_{24h} of the sample is measured and the water absorption is determined using equation (2).

$$W_{Abs} = 100 \times \frac{m_{24h} - m_0}{m_0} \quad (2)$$

2.7. Monitoring of moisture uptake

The monitoring of moisture uptake by TC fibres was carried out using a gravimetric method according to NF EN ISO 3344. The TC

Table 2
Chemical composition and physical properties of chemically retted TC fibres and other fibres from the literature.

Fibres	Chemical composition					Physical properties					
	Cellulose (wt %)	Hemicellulose (wt %)	Lignin (wt %)	Pectin (wt %)	Extractive (wt %)	Diameter (μm)		Density (g. cm^{-3})	Water absorption (wt%)	Moisture content (wt%)	References
						Normal	Weibull				
F1	49.5	26.6	10.0	3.7	2.4	235.3 ± 52.5	257 (4.8)[a]	1.26 ± 0.07	284.5 ± 61	10.1 ± 0.7	This study
F2	48.7	24.5	9.0	2.7	3.0	250.6 ± 53.2	273 (4.7)[a]	1.36 ± 0.06	222.0 ± 10	8.1 ± 0.4	This study
F3	46.4	23.2	8.0	2.5	1.8	226.3 ± 59.8	250 (4.1)[a]	1.39 ± 0.12	217.5 ± 52	9.7 ± 1.1	This study
F4	41.3	22.5	7.6	2.4	1.0	145.3 ± 61.8	164 (2.5)[a]	1.31 ± 0.10	203.9 ± 13	8.0 ± 0.8	This study
F5	39.5	18.0	5.0	2.2	1.2	203.3 ± 73.2	228 (2.8)[a]	1.32 ± 0.06	166.6 ± 19	7.9 ± 0.3	This study
F6	38.8	16.9	6.0	2.0	1.0	245.4 ± 76.0	294 (3.5)[a]	1.32 ± 0.07	150.3 ± 11	7.5 ± 0.6	This study
Triumfetta C.	44.4	30.8	18.9	3.3	2.6	20–210	–	1.48	342.3	8.1	[22]
Triumfetta P.	61.1	14.3	17.7	5.7	0.9	10–120	–	–	183.3	11.2	[31]
Cola lepidota	54.2	14.3	15.3	10.1	3.7	65–120	–	1.72	172	6.5	[25]
Rhecktophyllum C.	65.2	7.4	16.2	3.5	5.2	251–449	339	0.75	198.2	9.4	[24]
Ananas comosus	68.1	4.9	12.0	4.2	3.1	30–330	92	1.25	188.6	12.2	[24]
Megaphrynium M.	56–69	10–17	7–11	4–6.5	–	77–275	162 (3.9)	1.01–1.47	63–77	–	[4]
Cotton	85–90	4–6	0.5–1.0	–	0.4	11–22	–	1.5–1.6	–	8–25	[32]
Ramie	76.0	17.0	1.0	–	6.0	–	–	1.5	–	–	[11]
Banana	37.5	28	14.7	6	13	40–140	–	1.1–1.5	–	–	[11]
Sisal	60–78	10–14	8–14	0.8	1.2	122–135	–	1.33–1.5	190–250	5–10	[33]
Kenaf	31–72	20.3–21.5	8–19	–	–	–	–	1.45	–	–	[34]
Jute	62.1	5.4	16.9	–	–	–	–	1.3–1.48	281	12.3	[11]
Flax	64.1	16.7	2.0	3.9	1.5–3.3	20–250	–	1.52	136 ± 25	12.0	[11]
Hemp	68.0	15	10	5.3	–	20–240	–	1.46	158 ± 30	10.5	[11]

[a] The values in brackets represent the Weibull modulus which characterises the diameter dispersion of TC fibres.

fibres, previously dried at 105 °C for 6 h, were used to make samples (bundle of fibres of length 15 mm) of initial mass $m_0 = 0.5$ g. Three samples from each batch of fibres were placed in cups on a wire mesh inside a hygroscopic tray. A saturated ammonium nitrate solution was introduced into the tray 24 h prior to testing to create an atmosphere with a humidity of 65% at 23 °C [28]. In addition, a chemical solution based on thymol was placed directly in the hygroscopic tank to reduce the risk of alteration of the fibres by microbial attack. From the beginning of the conditioning process, weighing was carried out for increasing times with a milligram scale until a quasi-constant mass variation was obtained. The moisture content *MC* (equation (3)) and the moisture absorption ratio *MR* (equation (4)) were calculated for each measurement point to characterise the kinetics of fibre moisture uptake.

$$MC = 100 \times \frac{m_t - m_0}{m_0} \tag{3}$$

$$MR = 100 \times \frac{m_t - m_0}{m_s - m_0} \tag{4}$$

where m_t is the mass of the wet sample at measurement time t, and m_s is the mass of the moisture-saturated sample.

2.8. Fourier transform infrared (FTIR) spectroscopy analysis

The identification of the functional chemical groups in the alkaline retted TC fibres at room temperature and during firing was carried out using a Bruker Alpha-P spectrometer equipped with an ATR module. The spectra were recorded by crushing a few milligrams of the 110 μm milled fibre sample in transmittance mode in the spectral range 400–4000 cm^{-1} with a resolution of 4 cm^{-1}.

2.9. Thermogravimetric analysis

Thermal degradation of TC fibres was performed in a TA Q50 thermal analyser with an open platinum crucible. For these thermogravimetric analyses, a sample of fibre ($m = 4 \pm 0.2$ mg) previously ground to a size of 115 μm was heated at a constant rate of 10 °C.min^{-1}, in a temperature range of 25–600 °C, under a nitrogen atmosphere at 20 ml min^{-1}.

2.10. Tensile tests on individual fibre bundles

NF T25 501-2 standard was used to prepare the TC fibre bundle specimens. The randomly selected fibre bundle from a batch (F1, F2, F3, F4, F5 and F6) was glued with an adhesive to a paper frame with a window cut to have a gauge length of 10 mm. This length was chosen to limit the likelihood of defects (creases, bands) being present [29]. Prior to the tensile test, the gauge length of each specimen was observed under an optical microscope to remove the specimens containing the fibre bundles. In addition, three transverse measurements were taken every 3 mm along the gauge length of the specimen and used to calculate the average diameter of the fibre bundle under test. Each validated specimen was gently placed on the LDW-5 universal mechanical testing machine, equipped with a 100 N load cell. With the fibre bundle aligned with the axis of movement of the jaws, it was stretched to failure at a speed of 2 mm min^{-1} [30]. 25 specimens were tested in batches to determine their average mechanical properties, i.e., Young's modulus (in the 0.1%–0.25% deformation range), tensile strength, elongation at break and toughness (ratio of breaking strength to thickness). Specimen preparation and testing was carried out in a temperature-controlled room of 25 °C and 65% relative humidity.

3. Results and discussions

3.1. Chemical composition of the fibres

The mass percentages of the chemical constituents of the studied TC fibres are given in Table 2. Cellulose, hemicellulose and lignin represent at least 77.6 wt% and 61.7 wt% for the fibres extracted at room temperature and by cooking, which is reassuring. However, they are lower than those obtained for cotton (>85 wt%), hemp (>80 wt%) and flax (>80 wt%), but close to the percentages of banana (~75 wt%) and jute (~78 wt%) fibres [1,4,11,26].

It is also observed that fibres extracted at room temperature (F1, F2 and F3) have a higher content of extractables (1.8–3.0 wt%), pectin (up to 3.7 wt%), lignin (up to 10 wt%) and hemicellulose (23.2–26.6 wt%) than fibres extracted by cooking (F4, F5 and F6). Furthermore, the percentages of these non-cellulosic materials, especially lignin and hemicellulose, tend to decrease with increasing temperature, time and applied NaOH concentration. This result shows that alkaline extraction by cooking is more effective in removing non-cellulosic materials on the surface of TC fibres. Furthermore, the hemicellulose content of the studied fibres is at least 17 wt%, which is about 3 and 8 times higher than that (Table 2) of jute and cotton, respectively. This high hemicellulose content could make the fibres hydrophilic, which is interesting for the development of absorbent textile fabrics [23], but a major drawback for the interlocking of the fibres with polymer matrices [11,33]. For the lignin content, which makes the fibres stiff, the values found in this study are interesting, as they are lower than those found for fibres already used for the manufacture of placemats such as soda degummed jute [1] and banana [11]. The lower cellulose contents of F4, F5 and F6 fibres indicate that cellulose oxidation occurs partially during alkaline cooking. Such an effect could damage the fibre structure and consequently reduce its strength and stiffness [21,35,36]. The oxidation of cellulose could be avoided by adding a reducing agent such as sodium dithionite to the NaOH solution [37]. Despite this oxidation of cellulose, it is observed that (Table 2), the values found are in the same order as those for banana fibres and are within the

wide range reported for kenaf fibres by Ramesh [11].

Compared to TC fibres extracted by water retting [22], F1, F2 and F3 fibres contain slightly more cellulose and slightly less lignin, pectin, and hemicellulose. This may improve the flexibility and probably the strength of the fibres [8,34]. Therefore, the extraction of TC fibres by alkaline retting at room temperature could be interesting for the manufacture of textile fabrics and yarns.

3.2. FTIR spectra of the fibres

In order to analyse the influence of the alkaline extraction conditions and parameters on the chemical functional groups of TC fibres, FTIR spectra were plotted between 400 and 4000 cm^{-1} (Fig. 3). These spectra show characteristic peaks of the biochemical components of the plant fibres, such as cellulose, hemicellulose, pectin, and lignin [13,16,21].

The broad band visible between 3600 and 3200 cm^{-1}, with a major peak at 3330 cm^{-1}, corresponds to the stretching vibrations of the O–H and –OH bonds [12,15] of hemicelluloses and cellulose. The intensity of this broad band is clearly low for F2, and F3 fibres, and lower for all fibres extracted by cooking, indicating dissolution of hydroxyl (OH) groups. The peak at 2920 cm^{-1} signals the asymmetric stretching of C–H groups in cellulose, hemicellulose, and lignin [38,39]. Due to the lower intensity observed for the F6 fibre, it is possible that cellulose degradation occurred under the combined effect of the temperature (100 °C) and concentration (1.5 wt%) applied during its extraction process. The absence of the peak at 1728 cm^{-1} that was observed in a previous study [22] for TC fibre extracted by water retting shows that alkaline extraction is effective in removing the carboxyl (C–H) and carbonyl (C═O) groups from pectin or the acetyl and uronic ester from hemicellulose.

However, the presence of the peak at 1625 cm^{-1} in the spectra indicates that there are still water molecules trapped in the structure of the TC fibres studied. Similarly, the small peak at 1594 cm-1 indicates that there are still small traces of fatty acids and wax in the different fibres, but much less for the fibres extracted by cooking. These water molecules and waxy and fatty materials can be completely removed by treatment in a concentrated NaOH solution [26,40], or by a bleaching process with hydrogen peroxide [1] or sodium chlorite [4,21]. In addition, the peak at 1424 cm^{-1} is attributed to C–O bond stretching and C–H or O–H bond bending in hemicellulose [2,41]. The transmittance of this peak is significantly low for F2 and F3 fibres and tends to disappear for fibres extracted by alkaline cooking due to temperatures above room temperature. The strongest peak in the spectra is at 1027 cm^{-1} and is associated with the stretching vibration of the –OH and C═O bonds of cellulose and lignin.

Due to the partial removal of lignin, the intensity of this peak decreased as the time and NaOH concentration decreased for the room temperature extraction, but also as the temperature increased up to 100 °C for the alkaline firing extraction. The small peak at 899 cm^{-1} is attributed to the β-glycosidic lysis that links the saccharide carbon atoms in the complex structure of cellulose [42]. The reduction of this peak is evident for fibre extracted in a concentrated 1.5 wt% NaOH solution penda

The reduction of this peak is evident for the fibre extracted in a concentrated 1.5 wt% NaOH solution for 180 min at 100 °C, clearly indicating that the cellulose on the F6 fibre has been partially degraded. This result correlates with the one presented in Table 2 when the mass percentage of cellulose on the F6 fibre was determined.

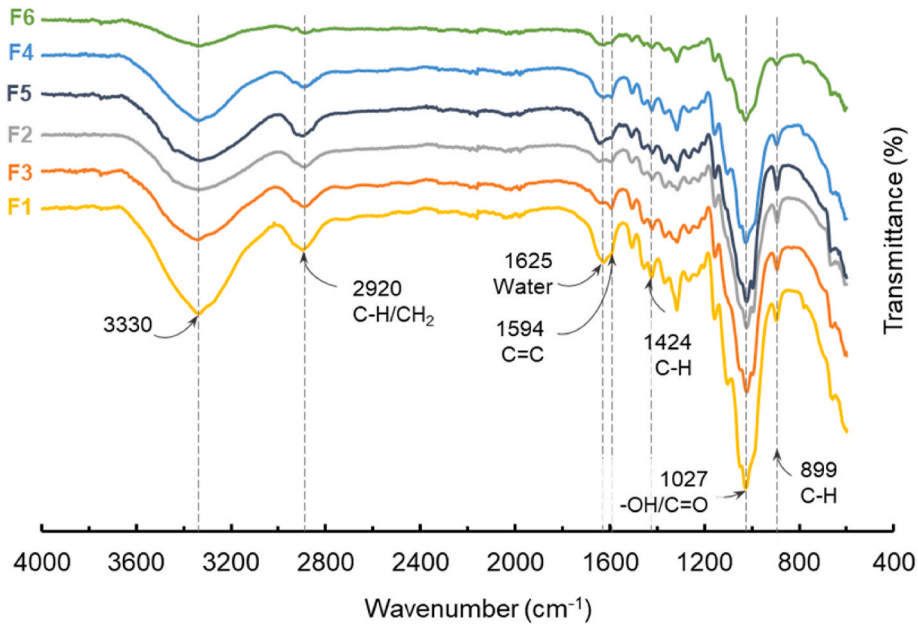


Fig. 3. Graph showing the FTIR spectra of the TC fibres designated F1, F2, F3, F4, F5 and F6.

3.3. Morphology of the fibre surface

SEM micrographs reveal that F2 (Fig. 4b) and F3 (Fig. 4c) fibres have cleaner and smoother surfaces compared to F1 (Fig. 4a), F4 (Fig. 4d), F5 (Fig. 4e) and F6 (Fig. 4f) fibres. The differences between the SEM micrographs confirm those observed on the FTIR spectra in terms of dissolution of non-cellulosic substances and degradation of cellulose.

In a previous study [22], it was observed that the surface of TC fibres extracted by water retting is covered by a sheath composed of non-cellulosic materials such as hemicellulose, pectin, lignin, and wax. Thus, the micrographs in this study show that alkaline retting (ambient and cooking) is effective in dissolving non-cellulosic substances. Room temperature extraction with a concentration of 3.5 wt % NaOH for 120 min (Fig. 4a) resulted in partial degumming of the sheath. This can be justified by the high chemical resistance of the C–C bonds and aromatic groups present in the lignin [42]. However, firing with alkaline NaOH solution at 1 wt% ($T = 100$ °C, $t = 240$ min: Figs. 4e) and 1.5 wt% ($T = 100$ °C, $t = 180$ min: Fig. 4f) stripped the sheath and seemed to shrink the fibres, but produced rough, wrinkled fibres. This is very important for the adhesion between the fibre and the polymer matrices. The shrinkage of the fibre may be explained by the removal of pectin from the middle lamella and the reduction of the empty spaces between the fibrils. This could

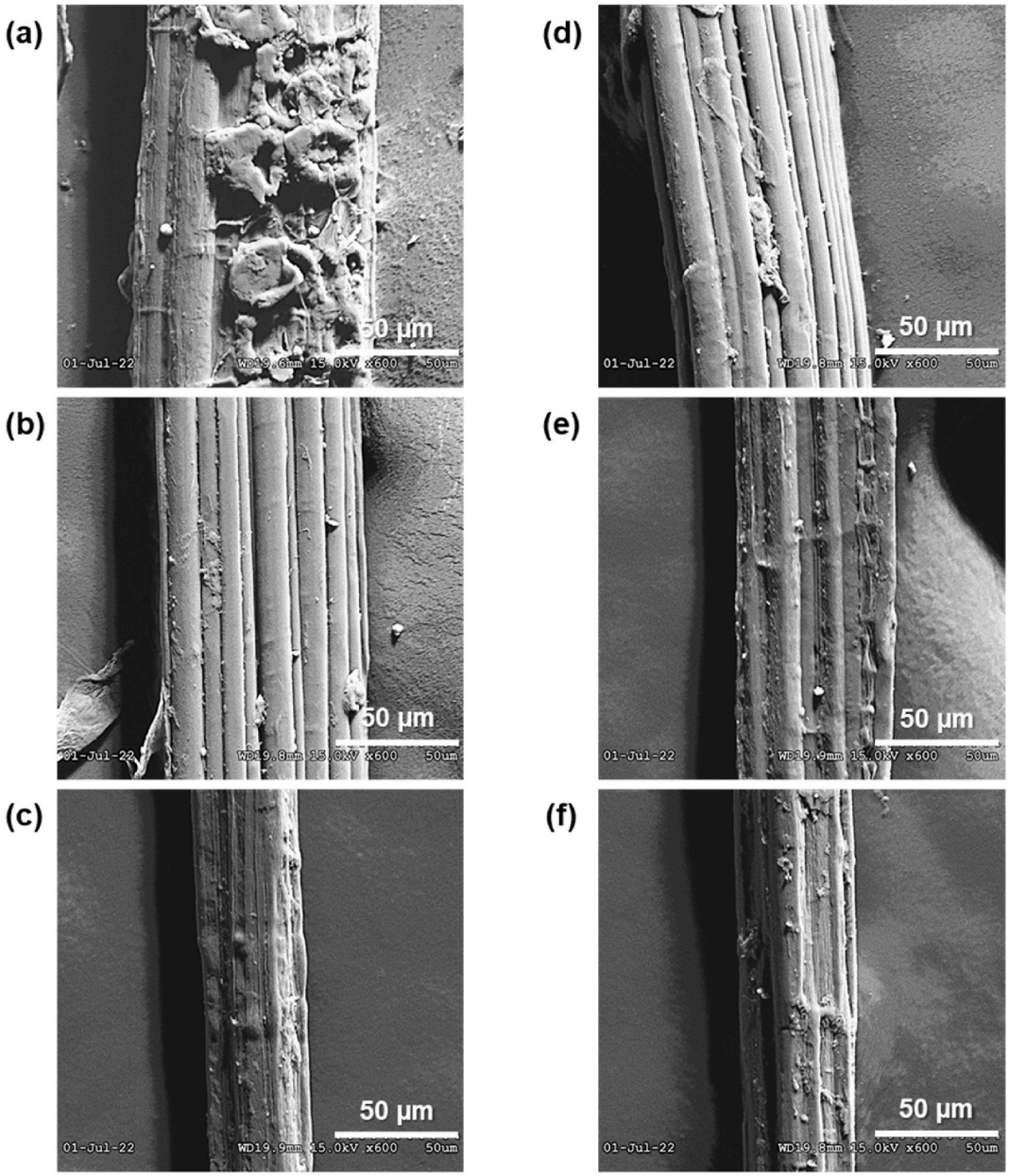


Fig. 4. SEM micrographs showing longitudinal views of the TC fibres designated: (a) F1, (b) F2, (c) F3, (d) F4, (e) F5 and (f) F6.

increase connectivity between individual fibres in fibre bundles and lead to shear stresses when the fibre bundle is subjected to tension due to deformation of the fibre-fibre interfaces [43,44]. Due to their smooth surface, like those of *Ananas comosus* [21] and jute [1] fibres, F2 and F3 fibres are expected to provide a better dye fixation effect for the manufacture of dyed fabrics compared to F1, F4, F5 and F6 fibres.

3.4. Fibre diameter distribution

The diameter distributions presented in Fig. 5a reveal a high variability in the diameters (coefficient of variance *CV* between 22 and 43%) of the TC fibres. This indicates irregularities along the length of the TC fibres due to their natural character and extraction defects visible on the SEM micrographs (Fig. 4). These distributions follow a normal distribution (Fig. 5b), which made it possible to calculate the mean values reported in Table 2. It can be noted that the baking process shrinks the TC fibres more than the room temperature extraction, which correlates well with the SEM observations (Fig. 4).

Furthermore, the experimental data correlate with the Weibull distribution ($0.92 < R^2 < 98$) as shown in Fig. 5b for the F2 fibre ($R^2 = 0.97$). The dispersion coefficients found are 3.5, 4.7, 4.1, 2.5, 2.9 and 4.8 for F1, F2, F3, F4, F5 and F6, respectively. The presence of size defects that can impact early failure and variability of mechanical properties of the fibres increases with the decrease of this coefficient [30,45]. Thus, an interesting ranking that informs about the effect of the treatment on the appearance of diameter irregularity defects induced by extraction can be proposed: F6 < F2 < F3 < F1 < F5 < F4. However, the theoretical mean values generated by Weibull's law using OriginPro software for fibres F1, F2, F3, F4, F5 and F6 increased by 20%, 8%, 9%, 12%, 11% and 4%, respectively, compared to the experimental values. Compared to other plant fibres (Table 2), which are already used in clothing textiles, TC fibres have average diameters that are in the range of flax, hemp, and pineapple comosus fibres.

3.5. Fibre densities

The densities of the fibres are presented in Table 2. It can be noted that the density tends to increase when the extraction time is extended (from 120 to 160 min) at room temperature but is almost constant for extraction by cooking. Compared to literature values for the same fibre, the values in this study are lower than the one (1.48 g cm^{-3}) found by Mewoli et al. [22], but very close to the one (1.26 g cm^{-3}) given by Senwitz et al. [19]. Furthermore, the use of TC fibres could contribute to the manufacture of lighter textile fabrics and composite structures compared to tropical Cola lepidota fibres and commercial fibres such as cotton, flax, kenaf and hemp.

3.6. Water absorption of fibres

Water absorption values by immersion for 24 h (Table 2) reveal that TC fibres extracted at room temperature can absorb between 217% and 285% of their dry mass. Similarly, fibres extracted by cooking absorb between 150 and 204% of their dry mass. An overall decrease in water absorption capacity was observed with increasing time, temperature and NaOH concentration. Fibres extracted at room temperature were more hydrophilic than fibres extracted by cooking due to their higher hemicellulose content. The maximum water uptake of 285 wt%, found for the F1 fibre, is similar to that of jute, while the lowest value 150.3 wt%, obtained for F6, is similar to that of sisal and *Ananas comosus* and hemp (Table 2). Furthermore, all the values found are lower than those in the literature [22] for the same fibre. This result indicates that, alkaline retting is effective in reducing the hydrophilic function of TC fibres compared to fresh water retting.

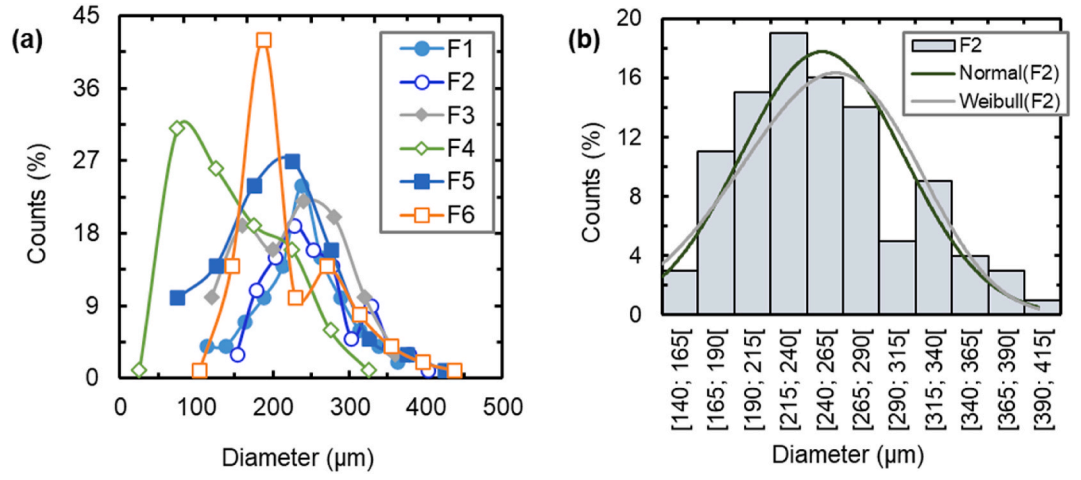


Fig. 5. (a) TC fibre diameter distributions, (b) Fitting the fibre diameter distribution with the normal distribution and the Weibull distribution.

3.7. Fibre moisture uptake kinetics

The moisture uptake curves (Fig. 6a) of TC fibres, obtained by conditioning at 65%RH/23 °C for increasing times, show that they can absorb up to 11.9% of their dry mass following simple exponential kinetics (Equation (5)) of the "Fickian" type.

$$y = y_0 - ae^{-b.t} \tag{5}$$

This two-phase moisture absorption behaviour is commonly described by natural fibres [29,46,47]. The first phase is linear and materialises a rapid absorption that occurs due to the porosity of the fibres and the many branches of the hemicelluloses. Its duration varies from 80 to 90 min, and its resistivity (or slope) is between 0.013 and 0.015 s^{-1}. These parameters are less pronounced for fibres extracted at room temperature F5 and F6. The second absorption phase is non-linear and converges as the fibre becomes saturated with moisture. This convergence is due to the swelling of the fibre [40,45]. This ability to hold moisture without dripping leads to an interesting classification that provides information on porosity and structural variations induced by extraction conditions and parameters: F1 > F2 > F3 > F4 > F5 > F6.

The highest moisture content (11.9 wt% for F1) in this study is lower than that of fibres already used in textile yarn production such as sisal (13.6 wt%), Typha latifolia (13.0 wt%) and jute (12.3 wt%) [8,48,49]. However, it is similar to that of flax (12.0 wt%). Moreover, this content is higher than that given for the same fibre by Mewoli et al. [22]. This discrepancy can be attributed to the higher extractive content [49] for this fibre in the literature. It is important to mention that in this comparative study, the literature values were not obtained with the same conditioning parameters, which has an influence on the moisture uptake and the diffusion coefficient as presented in Fig. 7.

The constants y_0, a and b in equation (6) were calculated using the Levenberg-Marquardt iteration algorithm [21] implemented in the OriginPro software. It can be seen in Table 3 that the theoretical values found and the experimental values agree with a very satisfactory R^2 correlation coefficient.

To estimate the diffusion coefficients D, the moisture absorption ratios obtained using equation (6), were plotted in Fig. 6b, and Fick's law (equation (6)) [28,48] was used.

$$D = \frac{\pi}{4}\left(\frac{r}{MC}\right)^2\left(\frac{H_2 - H_1}{\sqrt{t_2} - \sqrt{t_1}}\right)^2 \tag{6}$$

where r is the radius of the fibre, MC is the moisture content of the saturated fibre, H_2 and H_1 are the moisture content at the instants t_2 and t_1 belonging to the linear region of the curves in Fig. 6a.

The moisture diffusion coefficient values found are presented in Table 3. An overall correlation between the evolution of the diffusion coefficient and the moisture content is clearly observed. Diffusion and moisture retention are higher for fibres extracted at room temperature, and seem to increase with increasing NaOH concentration, temperature, and cooking extraction time. This result confirms the previous ones (Table 2) and consolidates the fact that the shrinkage of the fibres induced by the cooking process decreased the volume of voids in its structure.

3.8. Thermal degradation of the fibres

The TG and DTG thermograms of the TC fibres in this study are presented between 30 and 600 °C in Fig. 8a and b. The evolution of these thermograms confirms the presence of several constituents in the fibres and is commonly described for other types of plant textile fibres modified with NaOH [1,8,32].

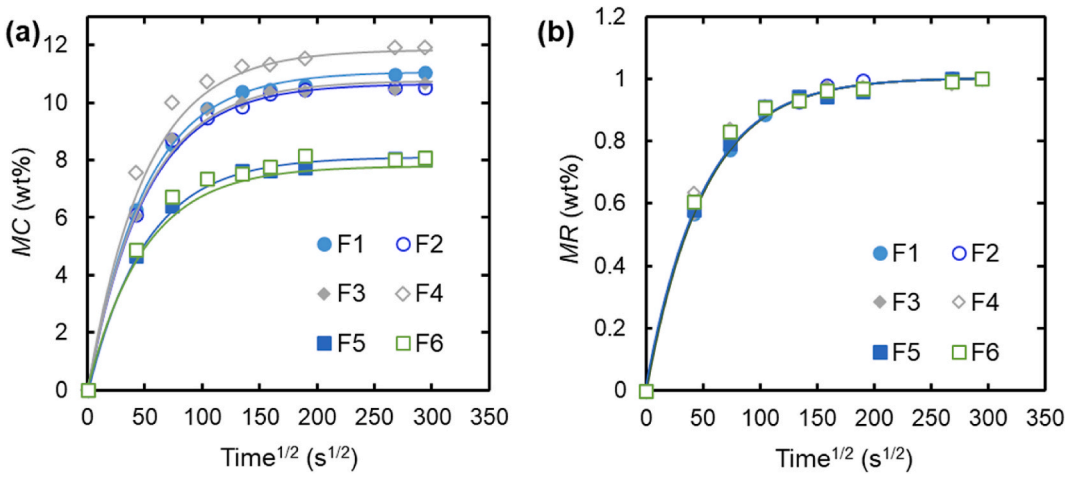


Fig. 6. Modelling curves for (a) moisture content and (b) moisture absorption rate.

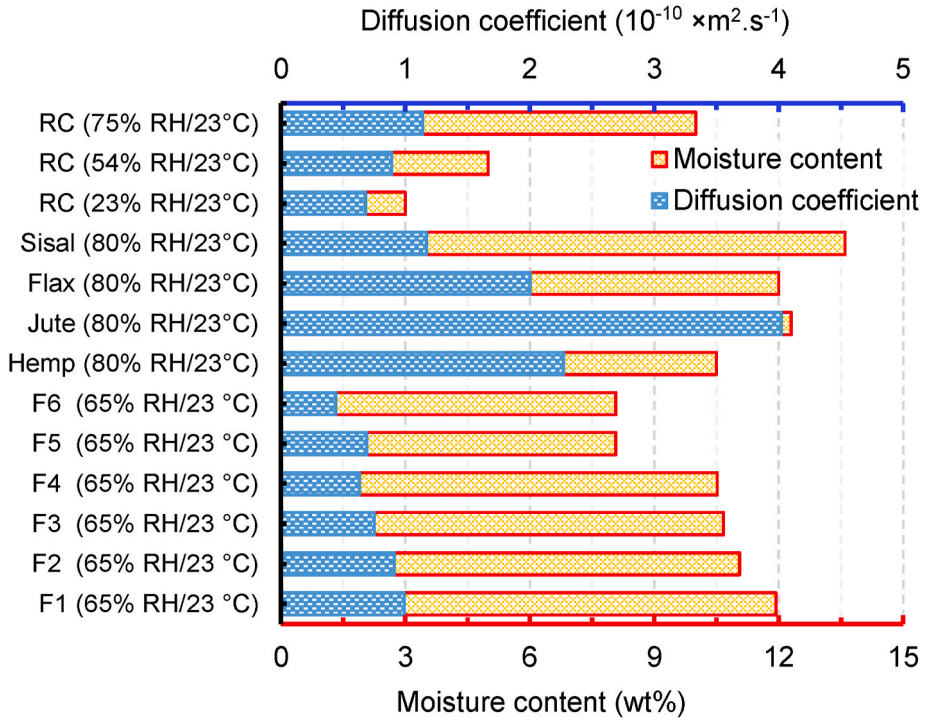


Fig. 7. Diffusion coefficients and moisture content of TC and other plant fibres from the literature [46,49]. RC stands for *Rhecktophyllum camerunense*.

Table 3
Moisture absorption properties of *Triumfetta cordifolia* fibres extracted by chemical retting.

Fibres	*MC* (wt%)	*r* (10^{-4} m)	*D* (10^{-11} m^2 s^{-1})	Fick model parameters			
				y_0	*a*	*b*	R^2
F1	11.9 ± 0.3	1.17	0.99	12,2	11.8	0.02	0.998
F2	11.1 ± 0.7	1.25	0.91	11,3	11.0	0.02	0.999
F3	10.7 ± 1.1	1.13	0.75	11,1	10.7	0.02	0.997
F4	10.5 ± 0.7	0.73	0.63	10,9	10.6	0.02	0.998
F5	8.1 ± 0.8	1.01	0.69	8,4	8.2	0.02	0.998
F6	8.1 ± 0.4	1.22	0.54	8,7	7.7	0.02	0.996

r: Fibre radius, *D*: Scattering coefficient.

It is observed (Fig. 8a) that, the first mass loss (of about 6% occurs) between 30 and 120 °C and is manifested by an initial peak on the DTG curve (blue arrow, Fig. 8b). This initial loss is related to the evaporation of water and low molecular weight constituents from the fibres. After this dehydration, we can note a thermal stability of the fibres up to 236.7 °C, indicating the level of temperature resistance of the fibres. Thereafter, three phases of thermal decomposition (mass loss between 69 and 76%) are observed: (i) the first phase manifests itself as a small shoulder peak (green arrow, Fig. 8b) between 236 and 310 °C (mass loss up to 22%), and corresponds to depolymerisation of hemicellulose and cleavage of glycosidic bonds in amorphous cellulose; (ii) the second phase occurs from 310 to 427 °C (mass loss up to 50%), and is associated with the strongest peak (red arrow, Fig. 8b) and to damage of α-cellulose; (iii) the last phase of mass loss is attributed to an oxidation reaction of the carbonised products or residues.

The highest residue content is 15.3% for the F1 fibre, which is 41%, 24%, 28%, 27% and 5% higher than the F2, F3, F4, F5 and F6 fibres, respectively. It is interesting to note that lignin decomposition occurs slowly over the whole temperature range (30 °C–600 °C) due to its aromatic rings [4,26].

Table 4 presents a comparative study of the results.

The dehydration of the fibres occurs up to 125 °C for F1, 118 °C for F2, 115 °C for F3 and F5, 110 °C for F4 and 118 °C for F6. This result indicates that drying is faster for the fibres extracted by cooking in general, and in particular for the F4 fibre. This thermo-regulatory property makes it an ideal fibre for the manufacture of summer clothing [2,5]. In addition, this fibre showed the lowest mass loss, although its lignin content (Table 2) was 1.25 and 1.33 times higher than F5 and F6, respectively. Thermal stability indicated a service temperature range of 231.7 ± 5.2 °C with a coefficient of variation (CV) of 2.2% for all fibres studied. For the batch of fibres

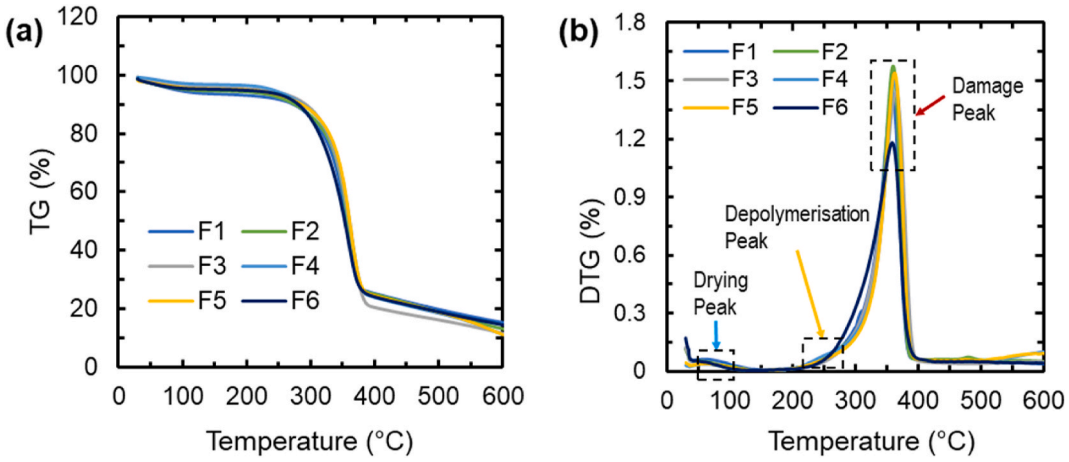


Fig. 8. (a) TG and (b) DTG curves of TC fibres.

Table 4
Thermal degradation properties of *Triumfetta cordifolia* fibres extracted by chemical retting and other fibres from the literature.

Fibres	Température de dégradation			Teneur des composants		References
	Initiale (°C)	Maximale (°C)	Final (°C)	Volatile (wt%)	Residue at 600 °C (wt%)	
F1	236.7	352	412.5	5.3	15.3	This study
F2	237.1	359	397.8	3.1	9.1	This study
F3	233.1	367	408.9	2.5	11.6	This study
F4	223.6	365	427.0	2.2	11.0	This study
F5	228.2	365	426.9	2.4	11.1	This study
F6	231.5	362	412.3	2.6	14.5	This study
Triumfetta C.	235	420	420	–	12.7	[22]
Triumfetta P.	220	380	380	2.35	24	[31]
Cola lepidota	230	325	490	7.27	10.2	[25]
Rhecktophyllum C.	220	318	495	8.2	2.3	[24]
Ananas comosus	210–230	360–394	450	2.6–6.5	5–21	[21]
Neuropeltis A.	211–230	317–368	382	3–6.7	13–21	[26]
Copernicia P.	267.3	353	414	9.6	11.6	[50]
Megaphrynium M.	235	–	393	1.1	7.4	[4]
Strelitzia reginae	240	295	377	8.4	19.0	[51]
Sisal	220	–	415	–	5.0	[52]
Cotton stalk	222	293	319	5.3	17.1	[5]
Okra	220	359	390	8.4	7.6	[53]
Bagasse	222	299	314	5.3	20.4	[54]
Banana	200	298	400	6.0	6.0	[5]
Kenaf	219	284	309	8.2	18.1	[11]
Jute	230	283	442	2.8	25.2	[1]
Flax	220	340	–	–	–	[11,53]
Hemp	220	390	400	5	30	[11]

extracted at room temperature, it was 235.6 ± 5.2 °C (CV = 0.9%), which is about 3.3% higher than that of the fibres extracted by firing (CV = 1.7%). These results show a very small difference in the temperature resistance of the fibres. Furthermore, these thermal stability temperatures are very close to those of other fibres already used in the production of weaving and knitting yarns. For example, the fibres F1, F2, F3, F4, F5 and F6 can undergo heat bleaching treatments in the same way as hemp, cotton, linen, jute and banana (Table 4). Similarly, the maximum average temperature of thermal degradation was 316.7 ± 5.5 °C (CV = 1.5%), and the final average temperature was 414.2 ± 11.2 °C (CV = 2.7%). Mass losses at this stage were also similar (CV<3%). These results clearly show that the thermal stability and degradation of the main fibre components are not affected by the mode (ambient and hot) and parameters (NaOH concentration, temp and temperature) of extraction.

3.9. Mechanical properties of the individual fibre bundles

Typical stress-strain curves for TC fibre bundles extracted by alkaline retting at room temperature and by firing are shown in Fig. 9. Three types of stress-strain curves can be distinguished in each batch of fibre bundle. This difference is probably due to the location of the fibres in the stem as described by Duval et al. [55] for hemp fibres. The curve assigned to type I has a single domain of linear elastic

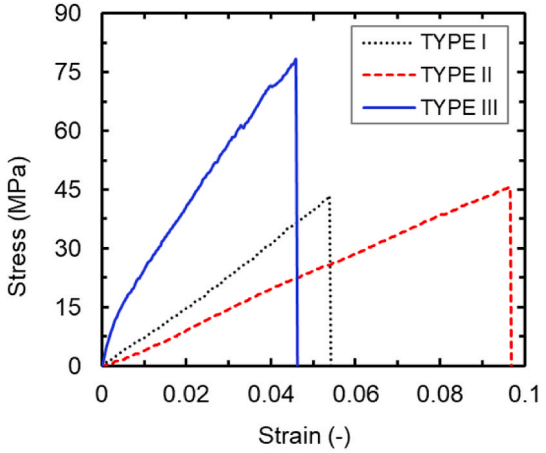


Fig. 9. Typical stress-strain curves of *Triumfetta cordifolia* fibres extracted by alkali retting at room temperature and by firing.

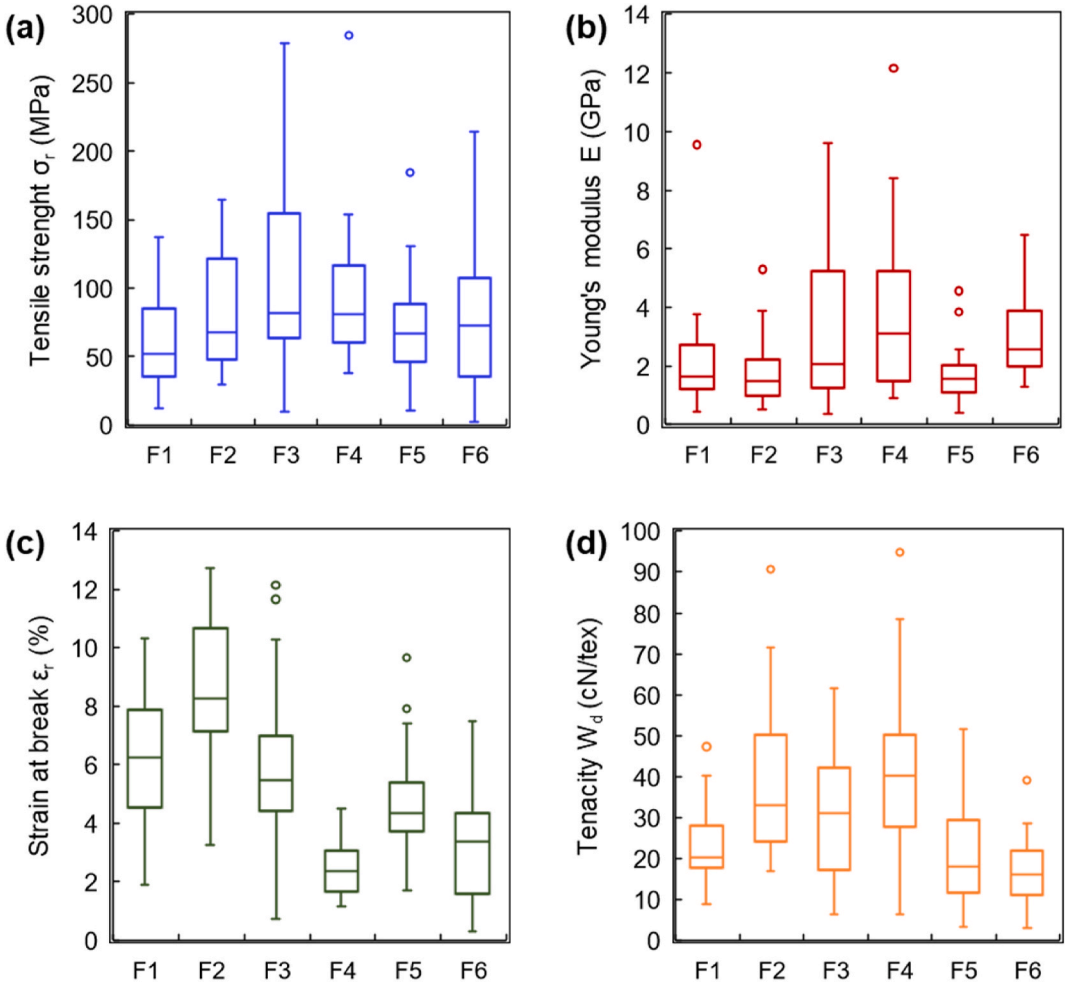


Fig. 10. Box plots showing the mechanical properties corresponding to (a) tensile strength, (b) Young's modulus, (c) elongation at break and (d) toughness of CT fibres.

behaviour (brittle failure) and the slope is the Young's modulus of the fibre. The type II behaviour consists of two linear parts separated by a very short zone of non-linear deformation due to a small slip of the microfibrils. This type of behaviour was observed for bleached *Ananas comosus* fibres [21] and raw *Cola lepidota* [56] fibres. The type III curve presents three distinct phases: the first phase is linear elastic and can extend up to 0.3% strain; the second phase (between 0.3% and 1.5% strain), non-linear is a transition zone due to significant slippage and progressive alignment of the microfibres with the fibre axis [30], while the last phase characterises the weakening (loss of stiffness) of the fibre. This type III behaviour is similar to that identified for *Furcraea foetida* [42], *Agave americana* [57] and *Rhecktophyllum camerunense* [30] fibre.

The overall evolution of the mechanical properties represented in Fig. 10 and the variations of the average values found (Table 5) show a sensitivity that seems to be significant when the conditions (room temperature and by firing) and the parameters (temperature, concentration and time) of extraction are modified. The tensile strength of fibres extracted at room temperature (F1, F2 and F3) varies from 60 to 112 MPa and increases by 27% and 47% when the retting time is increased from 120 to 150 min and from 120 to 180 min, respectively. For the same time interval, the Young's modulus, which provides information on stiffness, increases from 2.2 (for F1) to 3.2 MPa (for F3). On the other hand, the elongation at break varies from 5.8 to 8.4%, and the tenacity varies between 23 and 40 cN/tex. For this extraction condition, the F3 fibre gave the highest strength (Fig. 10a), but the F2 fibre is the softest (Fig. 10b and c) and the strongest (Fig. 10d) due to its high cellulose content, and rather low lignin and pectin content. Similarly, fibres extracted by firing (F4, F5, and F6) gave ranges of 69–112 MPa, 1.7–3.7 GPa, 2.3–4.7%, and 21–26 cN/tex for tensile strength, Young's modulus, elongation at break, and toughness, respectively. For this extraction mode, the toughest (Fig. 10a) and strongest (Fig. 10d) fibres are those extracted

Table 5
Summary of the mechanical properties of TC fibres extracted by the alkaline method and other fibres in the literature.

Fibres	Standard	Speed (mm. min^{-1})	Gauge lenght (mm)	Young's modulus (GPa)	Tensile strength (MPa)	Strain at breakage (%)	Tenacity (cN/tex)	Specific properties		References
								E/ρ (GPa. g^{-1}.cm^{-3})	σ_r/ρ (MPa.g^{-1}. cm^{-3})	
F1	NF T25 501-2	2.0	10	2.2 ± 1.8	60.6 ± 33.6	6.1 ± 2.2	23.6 ± 9.9	1.76	48.1	This study
F2	NF T25 501-2	2.0	10	1.8 ± 1.2	82.6 ± 42.0	8.4 ± 2.7	39.7 ± 19.3	2.38	60.9	This study
F3	NF T25 501-2	2.0	10	3.2 ± 2.6	111.6 ± 84.7	5.8 ± 2.9	31.0 ± 15.8	2.28	79.4	This study
F4	NF T25 501-2	2.0	10	3.7 ± 2.9	112.7 ± 100.4	2.3 ± 1.1	26.4 ± 9.2	1.28	53.2	This study
F5	NF T25 501-2	2.0	10	1.7 ± 1.0	69.9 ± 39.3	4.7 ± 1.8	21.6 ± 13.9	2.80	52.9	This study
F6	NF T25 501-2	2.0	10	3.1 ± 1.5	80.3 ± 59.2	3.3 ± 2.2	16.1 ± 12	1.36	60.9	This study
Triumfetta C.	NF T25 501-3	5.0	10	12.4 ± 6.9	526 ± 128	2.25	–	3.4–8.4	179–398	[22]
Triumfetta P.	NF T25 501-2	1.0	10	2.6 ± 1.2	1503.6	8.0	–	–	–	[31]
Cotton	–	–	–	5.5–12.6	287–597	3.0–10.0	28–48	3.4–7.9	179–373	[5]
Cola lepidota	NF T25 501-2	2.0	20	18–60	458–1440	2.4–2.6	28–88.3	11.6–36.8	280–883	[56]
Rhecktophyllum C.	NF T25 501-2	2.0	10	5.2–5.3	140–395	9–25	–	6.9–7.0	351–363	[30]
Ananas comosus	NF T25 501-2	2.0	10	8.6–12.0	323–572	4.0–5.1	62–91	6.9–9,6	258–458	[21]
Neurpeltis A.	NF T25 501-2	2.0	40	8.4 ± 5.9	321 ± 119	27.3 ± 7	–	6.8	261	[26]
Typha latifolia	ASTM D3822	2.0	25	6.8–15	68–169	1.16–2.0	–	–	–	[8]
Alfa	ASTM D3379-75	2.0	50	2.2–12.7	44–114	1.1–3.0	17.6	1.6–9.1	31.4–81	[23]
Esparto grass	ASTM D3379-75	5.0	40	2.1 ± 0.8	63.8 ± 16.8	3.1 ± 0.6	–	1.5	45.6	[58]
Banana	NF T25 501-2	10.0	25	20.0	500	4.5–6.5	11–34	13.3	333.3	[3]
Kenaf	–	–	–	20.0	223–930	9.1–12.3	–	13.8	154–641	[11]
Sisal	–	–	–	18.0	540–720	2.2–3.3	28.3	12.0	360–480	[11]
Jute	NF T25 704	10	20	15–30	610–780	1.0–1.9	–	10.1	233–527	[11]
Flax	ASTM D2256	1.0	10	50–70	345–1035	2.7–3.3	29–56	33–45	230–690	[11]
Hemp	ASTM D2256	1.0	10	4.8	270–900	1–3.5	–	3.3	185–616	[11]

E: Young's modulus; σ_r: Tensile strength; W_r: Tenacity, ρ: Density.

with a concentration of 1 wt% NaOH, a temperature of 80 °C and a cure time of 120 min. However, the same F4 fibres are stiffer and elongate 1.7 and 1.4 times less than F5 and F6. The high stiffness can be explained by the high lignin content, while the low elongation is probably due to a too small microfibrillar angle [27,42]. Blending these F4 fibres with soft cotton fibres can be considered to make soft, strong, and tenacious hybrid yarns [5,23].

The standard deviations range from 32 to 55% of the mean values, revealing a large variability in mechanical properties. This variability is well illustrated in the box plots in Fig. 10. The visible marks are exception values that contribute to the overestimation of the standard deviation. In Fig. 11a and b, the wide dispersion of mechanical properties of TC fibres is mainly caused by the variability of their diameter which is inherent to natural fibres. Among others, tensile strength (Fig. 11b) tends to decrease with increasing diameter, as observed for *Cola lepidota* [56], *Neuropeltis acuminatas* [26] and *Ananas comosus* [21,30] fibres, but this is clearly not the case for toughness (Fig. 11a). The greater dispersion in the toughness and tensile strength of the F6 fibre can be explained by the defects induced (surface wrinkles and cellulose oxidation) by the combined effect of the extraction temperature and the applied NaOH concentration. In addition, the location of the fibres in the mill and the overestimation of the fibre cross-section can also explain the high variability of the tensile strength and Young's modulus [19,29,59]. Indeed, the estimated cross-sectional area in the microscopic longitudinal view is larger than the area that actually bears the tensile load due to the voids [22] in the fibre. Furthermore, this lumen increases in the bast fibres from the bottom to the top of the plant [3,4,19,30].

Tenacities (Fig. 11c) and tensile strengths (Fig. 11d) show random distributions as Young's modulus increases. The distributions of F1, F3 and F6 fibres are the most sensitive to changes in stiffness. It can also be noted that batches F2 and F4 combine low stiffness, with an overall higher tenacity, indicating an interesting mechanical performance for their spinning.

Table 5 also compares the average mechanical properties of the TC fibres in this study with those of the same fibre and other fibre types in the literature. Specific mechanical properties that provide insight into the performance of the fibres were also compared.

The highest average tensile strength of the TC fibres in this study is 112.7 MPa (for the F4 fibre), which is 78.6% and 46.1% lower than the combed TC fibres extracted after 30 days of retting in fresh water [22], and those extracted by manual hulling [19], respectively. Similarly, the highest average stiffness is 3.7 GPa (for F4 fibre), which is 70% lower than that found by other authors [19, 22] for the same fibre. In contrast, the TC fibres in this study show much higher elongations, indicating that the alkaline extraction increased the microfibrillar angle. In addition to the extraction method, these high deviations can be attributed to the combing

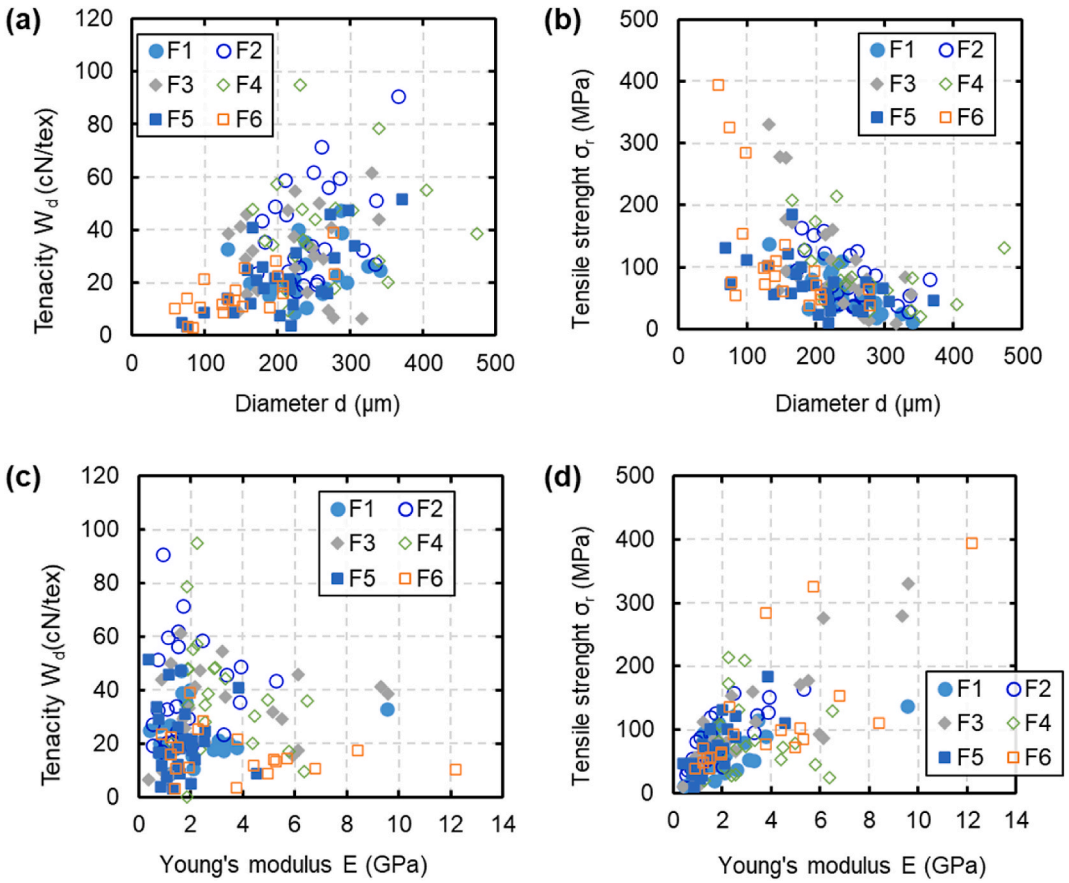


Fig. 11. Graphs showing the development of the (a,c) toughness and tensile (b,d) strength of TC fibres as a function of their (a,b) diameter and (c,d) Young's modulus.

operation, the method of determining the cross-sectional area, the initial test conditions (load cell sensitivity, device accuracy), the nature of the sample tested, the test parameters and conditions (gauge length, drawing speed, temperature and humidity) and the sample conditioning [11,26,27,29,39,59]. According to Dallel [23], the reliability of a natural fibre is possible if its tenacity is above 15 cN/tex. Thus, in addition to the low values of Young's moduli and high elongations, the allowable tenacities ($>$ 15 cN/tex) of the six fibre batches show that alkali retting proves to be an interesting process to produce stretchy and tenacious TC fibres for textile applications (yarns for fabrics and knits). Furthermore, the comparison of F2, F3 and F4 fibres shows that the specific properties of TC fibres are superior to those of *Esparto grass* (Table 5). It is important to mention that, for these literature data, the fibres were not extracted in the same way and the mechanical properties were not estimated with the same gauge length, standard and speed, which plays an important role on the discrepancies observed as reported by Baley et al. [29] and Ramesh [11]. In addition, it is found that (Table 5), the TC fibres in this study possess poor mechanical properties. Compared to other types of plant fibres, except for tenacity which is in the same range as cotton, banana and sisal textile fibres.

In order to quantify the dispersion of the tensile strength and toughness of TC fibres, a static analysis was performed using the two-parameter Weibull distribution function defined by $F(\Gamma) = 1 - exp[-(\Gamma/\Gamma_0)^m]$. In this equation, Γ is the sampling variable, Γ_0 is the characteristic value of the variable and m is the shape parameter that characterises the dispersion of the variable. The Weibull function or breakpoint probability $F(\Gamma)$ can also be written as $\ln(-ln(1-F(\Gamma))) = mln(\Gamma) - mln(\Gamma_0)$, which allows the modulus m to be determined graphically, which is the slope of an equation obtained by a linear fit [30,51,56]. Furthermore, $F(\Gamma) = (i-0.5)/n$ [26], where i is the rank of the *ith* data point and n is the number of data points corresponding to the number of samples tested.

Fig. 12 shows the Weibull distribution for the tensile strength (Fig. 12a,c) and toughness (Fig. 12b,d) of TC fibres. Fig. 12 shows the Weibull distribution for the tensile strength (Fig. 12a,c) and toughness (Fig. 12b,d) of CT fibres. The Weibull parameters m and Γ_0, as well as the correlation coefficient R^2 are summarised in Table 6. It can be seen that the correlation coefficien R^2 is greater than 0.86, which means that the experimental data fit the two-parameter Weibull model well, as shown in Fig. 12a and b. Furthermore, the characteristic values found are of the same order as those obtained from the descriptive statistics. These results indicate that the two-parameter Weibull model can be used to analyse the mechanical properties of TC fibres, at least for tensile strength and toughness. It should be noted that the dispersion of mechanical properties increases as the modulus m decreases. Consequently, the dispersions of tensile strength (resp. toughness) are higher for fibres designated F6, F3 and F4 (resp. F5, F4, F3 and F6). These dispersions are due to the high variability of the cross-section and, to a lesser extent, to structural defects (curved walls, knots) in the fibres [29,30].

4. Conclusions

The objective of this study was to extract with sodium hydroxide (NaOH) and characterise the fibres from the stem bark of *Triumfetta cordifolia* (TC) for the development of textile yarns. To this end, different concentrations (0.5, 1.0 and 1.5 wt%) of NaOH, temperatures (80, 100 and 120 °C) and times (120, 180 and 240 min) were used for alkaline extraction by cooking, while at room temperature, times of 120, 150 or 180 min with three concentrations (2.5, 3.0 and 3.5 wt%) were applied. A factorial experimental design was used to develop 34 combinations based on the extraction conditions and only six combinations produced clean, soft touch fibres. In addition, photographs proved the absence of basic bark skin residue and crimp on the fibres of these 6 batches. This study also aimed to understand the effect of alkaline extraction conditions on the biochemical composition and morphological, physical, thermal and mechanical properties of the fibres. The results revealed that the modification of the evaluated properties depended on the severity of the extraction conditions, except for the thermal stabilities which showed little difference (231.7 $\pm$ 5.2 °C).

Under mild conditions (3.5 wt%/25 °C/120 min), the SEM surfaces of the fibres showed large residues of the middle lamella, which made the lignin content (10 wt%) and hydrophilic function higher. Under medium conditions (2.5 wt%/25 °C/150min; 3.5/25 °C/180 min; 1.0 wt%/80 °C/120 min), the fibre surfaces were clean and slightly wrinkled (at 80 °C; 120min) with a cellulose content between 41 and 49 wt%, a density that varied from 1.31 to 1.36 g.cm-3 and a water absorption of 204–222 wt%. Under severe conditions (1.0 wt%/100 °C/240 min; 1.5 wt%/100 °C/180 min), heterogeneous transverse shrinkage and wrinkling were observed and accompanied by cellulose degradation (39 wt%) with a significant reduction in tenacity to 16cN/tex and a density of 1.32 g cm^{-3}. The tensile properties of the TC fibres showed great variability and a large influence of diameter was observed, indicating the need to study the influence of fibre location in the stem. However, the average extraction conditions resulted in higher tensile strength (113 MPa), tenacity (40 cN/tex) and elongation at break (8.4%). In addition, these fibres can be used alone or mixed with other fibres such as cotton to produce light, soft, tough and stretchy yarns, as can Typha latifolia, jute and sisal.

Author contribution statement

A.G. Soppie: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper. A.D.O. Betené: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper. Pierre Marcel Anicet Noah, Ateba Atangana: Conceived and designed the experiments; Analyzed and interpreted the data. A.E. Njom: Performed the experiments, Contributed reagents, materials, analysis tools or data. F. Betené Ebanda: Conceived and designed the experiments; Analyzed and interpreted the data, Contributed reagents, materials, analysis tools or data. A. Mewoli: Contributed reagents, materials, analysis tools or data. D. Nkemaja Efeze: Conceived and designed the experiments. R. Moukéné: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

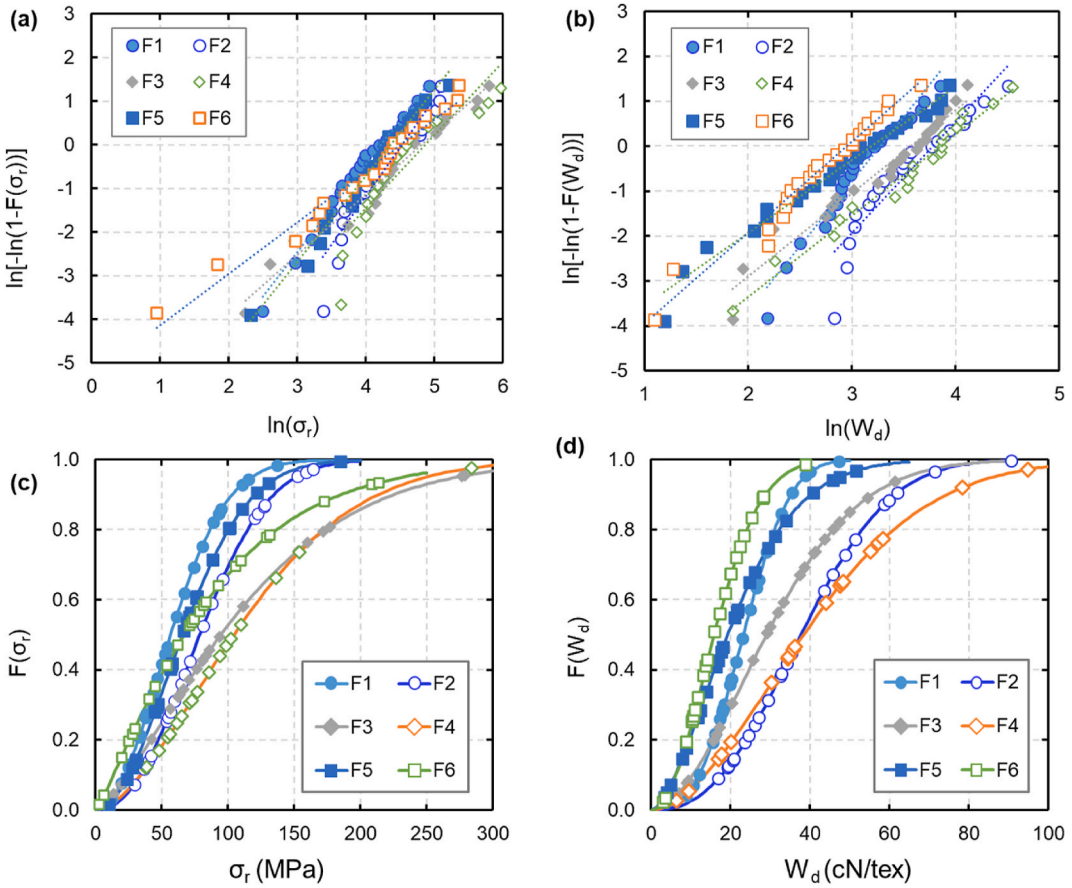


Fig. 12. Graphs showing Weibull distributions of (a,c) tensile strengths σ_r and (b,d) tenacities W_d of CT fibres. In plots (a,b), the marks correspond to experimentally obtained data and the dotted lines are regression lines for the graphical determination of the Weibull moduli.

Table 6
Summary of the Weibull parameters of TC fibres corresponds to the Weibull moduli m_{σ_r}, m_{W_d}; the characteristic values of tensile strength σ_{r0} and toughness W_{d0} and the correlation coefficients R^2 linked to σ_r and W_d.

		F1	F2	F3	F4	F5	F6
Resistance	m_{σ_r}	2.0	2.2	1.4	1.7	2.0	1.2
	σ_{r0} (MPa)	68.1	92.8	123.5	130.1	78.9	91.4
	R^2	0.98	0.91	0.96	0.87	0.98	0.97
Tenacité	m_{W_d}	2.9	2.5	1.9	1.8	1.6	2.0
	W_{d0} (cN/tex)	26.4	43.8	35.5	47.0	24.1	19.1
	R^2	0.96	0.90	0.96	0.97	0.98	0.97

Data availability statement

Data will be made available on request.

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.

Acknowledgements

The authors thank the *Centre de Caractérisation des Matériaux* (CeCaM) and the *Laboratoire* Pycnolab in Douala (Cameroon), and research site of INSA Lyon at Oyonnax for technical support. We would also like to thank Issa Vondou Monglo and Elie Julien Donald

Banyongen for their technical assistance in setting up the fibre extraction system.

References

[1] W. Wang, M. Xu, J. Lou, A. Dong, Changes in physicomechanical properties and structures of jute fibers after tetraacetylethylenediamine activated hydrogen peroxide treatment, J. Mater. Res. Technol. 9 (2020) 15412–15420.

[2] B.Y. Patel, H.K. Patel, Retting of banana pseudostem fibre using Bacillus strains to get excellent mechanical properties as biomaterial in textile & fiber industry, Heliyon 8 (2022).

[3] L. Libog, F. Biyeme, A.D.O. Betené, A.B. Biwolé, B. Ndiwe, J.P.E. Mbang, T. Claude, J.A. Mbey, J.R.L. Mevaa, Influence of the extraction location on the physical and mechanical properties of the pseudo-trunk banana fibers, J. Nat. Fibers 20 (2023), 2204451.

[4] A.D.O. Betené, C.S. Batoum, R.L. Ndoumou Belinga, F. Betene Ebanda, J.G. Tamba, A. Atangana, R. Moukené, G.K. Tcheudjo, A. Nyembè, Extraction and characterization of a novel tropical fibre Megaphrynium macrostachyum as a biosourced reinforcement for gypsum-based biocomposites, J. Compos. Mater. 57 (2023) 2543–2562.

[5] A. Khan, K. Iftikhar, M. Mohsin, M. Ubaidullah, M. Ali, A. Mueen, Banana agro-waste as an alternative to cotton fibre in textile applications. Yarn to fabric: an ecofriendly approach, Ind. Crop. Prod. 189 (2022), 115687.

[6] K. Shaker, R.M. Waseem Ullah Khan, M. Jabbar, M. Umair, A. Tariq, M. Kashif, Y. Nawab, Extraction and characterization of novel fibers from Vernonia elaeagnifolia as a potential textile fiber, Ind. Crop. Prod. 152 (2020), 112518.

[7] L.A. Elseify, M. Midani, A.H. Hassanin, T. Hamouda, R. Khiari, Long textile fibres from the midrib of date palm: physiochemical, morphological, and mechanical properties, Ind. Crop. Prod. 151 (2020), 112466.

[8] M. Hasan, M. Rahman, Y. Chen, N. Cicek, Optimization of Typha fibre extraction and properties for bio-composite applications using desirability function analysis, Polymers 14 (2022) 1685.

[9] P. Lyu, L. Xia, X. Jiang, X. Liu, W. Xu, C. Hurren, X. Wang, Efficient extraction of technical fibers from hemp in an ethanol-water mixture, Ind. Crop. Prod. 178 (2022), 114620.

[10] L.A. Elseify, M. Midani, L.A. Shihata, H. El-Mously, Review on cellulosic fibers extracted from date palms (Phoenix Dactylifera L.) and their applications, Cellulose 26 (2019) 2209–2232.

[11] M. Ramesh, 9 - hemp, jute, banana, kenaf, ramie, sisal fibers, in: A.R. Bunsell (Ed.), Handbook of Properties of Textile and Technical Fibres, second ed., Woodhead Publishing, 2018, pp. 301–325.

[12] G. Bar, K. Chaudhary, Characterization of textile grade novel Bauhinia vahlii fiber, J. Nat. Fibers 20 (2023), 2143464.

[13] A. Vinod, M.R. Sanjay, S. Siengchin, Recently explored natural cellulosic plant fibers 2018–2022: a potential raw material resource for lightweight composites, Ind. Crop. Prod. 192 (2023), 116099.

[14] J. Zhu, F. Guo, C. Ma, H. Wang, J. Wen, Y. Yu, The alkaline extraction efficiency of bamboo cell walls is related to their structural differences on both anatomical and molecular level, Ind. Crop. Prod. 178 (2022), 114628.

[15] S. Amroune, A. Bezazi, A. Belaadi, C. Zhu, F. Scarpa, S. Rahatekar, A. Imad, Tensile mechanical properties and surface chemical sensitivity of technical fibres from date palm fruit branches (Phoenix dactylifera L.), Compos. Appl. Sci. Manuf. 71 (2015) 95–106.

[16] A. Vinod, S.M. Rangappa, R. Srisuk, J. Tengsuthiwat, R. Arun Ramnath, S. Siengchin, Agro-waste Capsicum Annum stem: an alternative raw material for lightweight composites, Ind. Crop. Prod. 193 (2023), 116141.

[17] M. Brink, E.G. Achigan-Dako, Plant Resources of Tropical Africa 16 Fibres, PROTA Foundation/CTA, 2012. https://research.wur.nl/en/publications/plant-resources-of-tropical-africa-16-fibres. accessed May 29, 2023.

[18] P. Grosser, C. Siegel, C. Neinhuis, T. Lautenschlage, Triumfetta cordifolia: a valuable (African) source for biocomposites, Bio 13 (2018) 7671–7682.

[19] C. Senwitz, A. Kempe, C. Neinhuis, J.L. Mandombe, M.F. Branquima, T. Lautenschlager, Almost forgotten resources – biomechanical properties of traditionally used bast fibers from Northern Angola, Bioresources 11 (2016) 7595–7607.

[20] M.A. Uddin, M.M. Rahman, A.N.M.A. Haque, S.A. Smriti, E. Datta, N. Farzana, S. Chowdhury, J. Haider, A.S. Muhammad Sayem, Textile colouration with natural colourants: a review, J. Clean. Prod. 349 (2022), 131489.

[21] A.D.O. Betené, F.E. Betené, F.E. Ngali, P.M.A. Noah, B. Ndiwé, A.G. Soppie, A. Atangana, R. Moukené, Influence of sampling area and extraction method on the thermal, physical and mechanical properties of Cameroonian Ananas comosus leaf fibers, Heliyon 8 (2022), e10127.

[22] A.E. Mewoli, C. Segovia, F.B. Ebanda, A. Ateba, P.M.A. Noah, B. Ndiwe, A.E. Njom, Physical-chemical and mechanical characterization of the bast fibers of triumfetta cordifolia A. Rich. from the equatorial region of Cameroon, J. Miner. Mater. Char. Eng. 8 (2020) 163–176.

[23] M. Dallel, Evaluation du Potentiel Textile des Fibres d'Alfa (Stipa Tenacissima L.): Caractérisation Physico-chimique de la Fibre au Fil, These de doctorat, Mulhouse, 2012. https://www.theses.fr/2012MULH6853. accessed May 29, 2023.

[24] A.D.O. Betene, F.E. Betene, F. Martoia, P.J. Dumont, A. Atangana, P.M.A. Noah, Physico-chemical and thermal characterization of some lignocellulosic fibres: Ananas comosus (AC), Neuropeltis acuminatas (NA) and Rhecktophyllum camerunense (RC), J. Miner. Mater. Char. Eng. 8 (2020) 205–222.

[25] N.B.R. Legrand, O. Pierre, B.E. Fabien, N.P. Marcel, A.A. Jean, Physico-chemical and thermal characterization of a lignocellulosic fiber, extracted from the bast of cola lepidota stem, J. Miner. Mater. Char. Eng. 8 (2020) 377–392.

[26] S.V. Obame, A.D.O. Betené, P.M.A. Noah, F.E. Betené, A. Atangana, Characterization of the Neuropeltis acuminatas liana fiber treated as composite reinforcement, Results Mater. 16 (2022), 100327.

[27] P. Manimaran, V. Vignesh, A. Khan, G.P. Pillai, K.J. Nagarajan, M. Prithiviraj, A.N. Al-Romaizan, M.A. Hussein, M. Puttegowda, A.M. Asiri, Extraction and characterization of natural lignocellulosic fibres from Typha angustata grass, Int. J. Biol. Macromol. 222 (2022) 1840–1851.

[28] Z. El Hachem, A. Celino, G. Challita, M.-J. Moya, S. Freour, Hygroscopic multi-scale behavior of polypropylene matrix reinforced with flax fibers, Ind. Crop. Prod. 140 (2019), 111634.

[29] C. Baley, M. Gomina, J. Breard, A. Bourmaud, P. Davies, Variability of mechanical properties of flax fibres for composite reinforcement. A review, Ind. Crop. Prod. 145 (2020), 111984.

[30] A.D.O. Betené, F. Martoia, P.J.J. Dumont, F. Ebanda Betené, A. Ateba, Gypsum plaster composites reinforced with tropical fibre bundles extracted from Rhecktophyllum camerunense and Ananas comosus plants: microstructure and mechanical performance, Construct. Build. Mater. 392 (2023), 131815.

[31] A.D. Nkapleweh, J.F. Tendo, F.B. Ebanda, P.M.A. Noah, A.E. Mewoli, T.T. Stanislas, Physico-chemical and mechanical characterization of triumfetta Pentandra bast fiber from the equatorial region of Cameroon as a potential reinforcement of polymer composites, J. Nat. Fibers 19 (2022) 13106–13119.

[32] N. Kambli, S. Basak, K.K. Samanta, R.R. Deshmukh, Extraction of natural cellulosic fibers from cornhusk and its physico-chemical properties, Fibers Polym. 17 (2016) 687–694.

[33] A.D. Gudayu, L. Steuernagel, D. Meiners, A.M. Woubou, Sisal fiber reinforced polyethylene terephthalate composites; Fabrication, characterization and possible application, Polym. Polym. Compos. 30 (2022), 09673911221103317.

[34] A. Guo, Z. Sun, J. Satyavolu, Impact of chemical treatment on the physiochemical and mechanical properties of kenaf fibers, Ind. Crop. Prod. 141 (2019), 111726.

[35] R. Vijay, D.L. Singaravelu, A. Vinod, M.R. Sanjay, S. Siengchin, Characterization of alkali-treated and untreated natural fibers from the stem of parthenium hysterophorus, J. Nat. Fibers 18 (2019) 80–90.

[36] N.P. Kumar, M. Chellapandian, N. Arunachelam, P. Vincent, Effect of mercerization on the chemical characteristics of plant-based natural fibers, Mater. Today Proc. 68 (2022) 1201–1207.

[37] S. Sayeb, I. Marzoug, M.B. Hassen, F. Sakli, S. Rodesli, Study of water sorption properties for esparto grass ultimate fibre (ALFA fibre), J. Text. Inst. 101 (2010) 19–27.

[38] M. Kathirselvam, A. Kumaravel, V.P. Arthanarieswaran, S.S. Saravanakumar, Characterization of cellulose fibers in Thespesia populnea barks: influence of alkali treatment, Carbohydr. Polym. 217 (2019) 178–189.
[39] K. Zhang, F. Wang, W. Liang, Z. Wang, Z. Duan, B. Yang, Thermal and mechanical properties of bamboo fiber reinforced epoxy composites, Polymers 10 (2018) 608.
[40] M. Maache, A. Bezazi, S. Amroune, F. Scarpa, A. Dufresne, Characterization of a novel natural cellulosic fiber from Juncus effusus L, Carbohydr. Polym. 171 (2017) 163–172.
[41] M. Mittal, R. Chaudhary, Experimental study on the water absorption and surface characteristics of alkali treated pineapple leaf fibre and coconut husk fibre, Int. J. Appl. Eng. Res. 13 (2018) 12237–12243.
[42] S.M. Shahril, M.J.M. Ridzuan, M.S.A. Majid, A.M.N. Bariah, M.T.A. Rahman, P. Narayanasamy, Alkali treatment influence on cellulosic fiber from Furcraea foetida leaves as potential reinforcement of polymeric composites, J. Mater. Res. Technol. 19 (2022) 2567–2583.
[43] O. Romruen, T. Karbowiak, W. Tongdeesoontorn, K.A. Shiekh, S. Rawdkuen, Extraction and characterization of cellulose from agricultural by-products of Chiang rai province, Thailand, Polymers 14 (2022) 1830.
[44] R. Sarala, Characterization of a new natural cellulosic fiber extracted from Derris scandens stem, Int. J. Biol. Macromol. 165 (2020) 2303–2313.
[45] E. Trujillo, M. Moesen, L. Osorio, A.W. Van Vuure, J. Ivens, I. Verpoest, Bamboo fibres for reinforcement in composite materials: strength Weibull analysis, Compos. Appl. Sci. Manuf. 61 (2014) 115–125.
[46] F. Ebanda, A. Ateba, Study of the thermal behavior of the plaster reinforced vegetable fiber, Int. J. Eng. Sci. Invention 5 (2016) 62–67.
[47] P.W. Huisken Mejouyo, E.M. Tiaya, N.R. Sikame Tagne, S.T. Tiwa, E. Njeugna, Experimental study of water-sorption and desorption of several varieties of oil palm mesocarp fibers, Results Mater. 14 (2022), 100284.
[48] Z. Samouh, O. Cherkaoui, D. Soulat, A.R. Labanieh, F. Boussu, R.E. moznine, Identification of the physical and mechanical properties of Moroccan sisal yarns used as reinforcements for composite materials, Fibers 9 (2021) 13.
[49] A. Celino, S. Freour, F. Jacquemin, P. Casari, Characterization and modeling of the moisture diffusion behavior of natural fibers, J. Appl. Polym. Sci. 130 (2013) 297–306.
[50] R.F. Pereira Junio, L. de Mendonça Neuba, A.T. Souza, A.C. Pereira, L.F. Cassiano Nascimento, S.N. Monteiro, Thermochemical and structural characterization of promising carnauba novel leaf fiber (Copernicia prunifera), J. Mater. Res. Technol. 18 (2022) 4714–4723.
[51] N. Lemita, S. Deghboudj, M. Rokbi, F.M.L. Rekbi, R. Halimi, Characterization and analysis of novel natural cellulosic fiber extracted from Strelitzia reginae plant, J. Compos. Mater. 56 (2022) 99–114.
[52] Y. Li, Y.-W. Mai, L. Ye, Sisal fibre and its composites: a review of recent developments, Compos. Sci. Technol. 60 (2000) 2037–2055.
[53] I.M. De Rosa, J.M. Kenny, D. Puglia, C. Santulli, F. Sarasini, Morphological, thermal and mechanical characterization of okra (Abelmoschus esculentus) fibres as potential reinforcement in polymer composites, Compos. Sci. Technol. 70 (2010) 116–122.
[54] S.S. Kusumah, K. Umemura, K. Yoshioka, H. Miyafuji, K. Kanayama, Utilization of sweet sorghum bagasse and citric acid for manufacturing of particleboard I: effects of pre-drying treatment and citric acid content on the board properties, Ind. Crop. Prod. 84 (2016) 34–42.
[55] A. Duval, A. Bourmaud, L. Augier, C. Baley, Influence of the sampling area of the stem on the mechanical properties of hemp fibers, Mater. Lett. 65 (2011) 797–800.
[56] R.L. Ndoumou, D. Soulat, A.R. Labanieh, M. Ferreira, L. Mevaa, J. Atangana Ateba, Characterization of tensile properties of Cola lepidota fibers, Fibers 10 (2022) 6.
[57] A. Bezazi, A. Belaadi, M. Bourchak, F. Scarpa, K. Boba, Novel extraction techniques, chemical and mechanical characterisation of Agave americana L. natural fibres, Compos. B Eng. 66 (2014) 194–203.
[58] A. Maghchiche, Chapter 6 - pulping and papermaking of esparto grass, in: Z.M.A. Ainun, S.M. Sapuan, R.A. Ilyas (Eds.), Pulping and Papermaking of Nonwood Plant Fibers, Academic Press, 2023, pp. 101–120.
[59] V. Placet, F. Trivaudey, O. Cisse, V. Gucheret-Retel, M.L. Boubakar, Diameter dependence of the apparent tensile modulus of hemp fibres: a morphological, structural or ultrastructural effect? Compos. Appl. Sci. Manuf. 43 (2012) 275–287.