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Adama Kabé

kabeadama@gmail.com

University of Maroua

Frédéric Djoda Pagore

University of Maroua

valéry Hambaté Gomdjé

University of Maroua

moussa sali

University of Maroua

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Influence of extraction methods on the mechanical properties, statistical homogeneity, and moisture regain of Hyphaene thebaica petiole fibers for composite reinforcement

Kabé Adama¹, Frédéric Djoda Pagore^{3*}, Hambaté Gomdjé Valéry¹, and Moussa Sali³

¹ Department of Textile and Leather Engineering, National Advanced School of Engineering, University of Maroua, P.O. Box 46, Maroua, Cameroon.

² Department of Civil Engineering and Architecture, National Advanced School of Engineering, University of Maroua, P.O. Box 46, Maroua, Cameroon.

³ Department of Chemistry, School of Physical and Chemical Sciences, Faculty of Natural and Agricultural Sciences, North-West University (Mafikeng Campus), Private Bag X2046, Mmabatho, 2735, South Africa

⁴ Material Science Innovation and Modelling (MaSIM) Research Focus Area, Faculty of Natural and Agricultural Sciences, North-West University (Mafikeng Campus), Private Bag X2046, Mmabatho, 2735, South Africa

Email: kabadama@gmail.com,

Abstract

This study examines the influence of three extraction methods on the mechanical and hygroscopic properties of *Hyphaene thebaica* petiole fibers. Two biological processes water retting and underground retting were compared to an alkaline treatment with NaOH in order to identify the most suitable method for use as a reinforcing material in polymer matrix composites. The extracted fibers were characterized using tensile tests, Weibull statistical analysis, and moisture regain tests. The results show that the alkaline treatment produces the strongest fibers, with a maximum stress of 47 MPa, as well as the lowest moisture regain, at 13.898%. Underground retting yields the most homogeneous fibers, with the highest Weibull moduli for stress and Young's modulus, indicating greater mechanical reliability. Water retting results in fibers that are more ductile but more sensitive to moisture. These results demonstrate that the extraction process strongly influences fiber performance and that *Hyphaene thebaica* fibers represent a promising lignocellulosic material for future use as a composite reinforcement.

Keywords: *Hyphaene thebaica*; natural fibers; extraction methods; retting; alkali treatment; Weibull analysis; mechanical properties; moisture absorption; bio-based composites

1. Introduction

Against a backdrop marked by the climate emergency and the need to reduce the environmental footprint of industrial materials, the development of sustainable solutions is a major challenge for materials science research. Natural-fiber composites are thus attracting growing interest due to their low density, their renewable nature, and their potential to replace synthetic reinforcements derived from fossil resources. Recent reviews confirm that these materials are gradually establishing themselves as credible alternatives for structural and semi-structural applications, provided that their compatibility with the matrix and their sensitivity to moisture are better understood [1] [2], [3], [4], [5].

Among the most studied natural fibers are flax, hemp, jute, sisal, kenaf, and bamboo, whose performance depends heavily on their chemical composition, morphology, and the treatments applied during extraction. Several recent reviews show that improving fiber-matrix adhesion and reducing water absorption often require surface treatments, particularly alkaline treatments, silane treatments, or other chemical modifications [6].

However, the use of natural fibers remains limited by their hydrophilic nature, their intrinsic variability, and the dispersion of their mechanical properties [7], [8], [9], [10]. Recent studies on the durability of natural-fiber composites show that moisture absorption can cause a significant decrease in tensile strength and Young's modulus, as well as a gradual degradation of the fiber-matrix interface. These effects are of particular concern in applications where dimensional stability and mechanical reliability are essential [11].

In this context, *Hyphaene thebaica*, or the doum palm, is an abundant local lignocellulosic resource in the Sahelian and Sudano-Sahelian regions of Africa. Although this species is traditionally harvested for its fruit, leaves, and wood, its petioles remain largely underutilized, even though they contain fibers that could be used as reinforcements in bio-based composites. Recent literature on natural fibers highlights the growing interest in underutilized local resources, particularly when they are regionally available and have potential for value-added applications [12], [14].

However, lignocellulosic fibers exhibit significant variability in their mechanical properties, which is related to the age of the plant, growing conditions, and extraction methods. This variation in performance can reduce their reliability in structural applications. Furthermore, their affinity for water can lead to significant moisture absorption, which affects their long-term mechanical performance [4]. Recent studies show that moisture absorption is a key design criterion for natural-fiber composites, on par with initial mechanical strength and interface quality [16].

This study compares three methods for extracting fibers from the petioles of *Hyphaene thebaica*: water retting, underground retting, and alkali treatment with soda ash. The objective is to identify the method offering the best balance between mechanical strength, statistical homogeneity, and moisture stability for use as a reinforcing material in polymer matrix composites. To this end, the fibers were characterized through tensile tests, statistical analysis using the Weibull model, and moisture absorption measurements. This approach is consistent with recent trends in the literature, which emphasize the need to simultaneously evaluate mechanical performance, property dispersion, and durability in a humid environment.

2. Material and method

2.1 Raw materials

The fibers used in this study were extracted from the petioles of a mature doum palm (*Hyphaene thebaica*), harvested in the village of Birio (Balaza), located in the Maroua III district of Cameroon's Far North Region (10.6754821° N; 14.4445153° E). This species was selected

because of its abundance in Sahelian areas, its local availability, and the potential for utilizing its lignocellulosic fibers as a reinforcing material in composites.

After harvesting, the petioles were transported to the laboratory and prepared for fiber extraction. Three extraction processes were studied: two biological methods—namely, water retting and underground retting—as well as a chemical method involving alkaline treatment with sodium hydroxide (NaOH). This comparative study aims to evaluate the influence of each extraction process on the mechanical properties of the fibers with a view to their use as reinforcing fibers in composites.

2.2 Fiber Extraction Methods

2.2.1 Water retting

Water retting is a traditional biological method for extracting plant fibers that is widely used to separate fibrous bundles from non-fibrous tissue; it relies on the action of microorganisms naturally present in the aqueous medium [15].

In this study, the previously cleaned petioles of the doum palm (*Hyphaene thebaica*) were completely submerged in a container of distilled water for 30 days. The progress of the retting process was monitored every seven days to track the degradation of the plant tissue and prevent excessive fermentation. At the end of the treatment, the fibers were extracted by hand, rinsed with distilled water to remove organic residues, then dried in an oven before being stored in a dry environment until characterization tests were conducted.

2.2.2 Retting underground

Unlike retting on the ground, which under the combined action of the sun and rain promotes the development of microorganisms that make it possible to dissociate the non-cellulosic elements from the fibrous part of the plant by eliminating the bonds that connect them together [16, p. 25], Underground retting, on the other hand, is based on the activity of bacteria and fungi naturally present in the soil [17].

For our study, the petioles of doum palm (*Hyphaene thebaica*) were buried in naturally moist soil for 35 days. After retting, they were dug up and the fibres were manually separated, rinsed with water to remove organic residues, and then air-dried before being kept in a dry environment until characterization testing.

2.2.3 Alkaline treatment

Chemical maceration, or surfactant retting, refers to a set of retting processes in which a heated chemical solution is used to promote the breakdown of pectins and thus release the fibres from the plant tissue [17], [18].

In the framework of our study, the petioles, once prepared, will be immersed in a 1 N NaOH solution, prepared by dissolving 40 g of NaOH in 1 L of distilled water and held in an oven at 100 °C for 1 h. At the end of the treatment, the fibres were neutralised in an acetic acid solution, washed with detergent and then rinsed thoroughly with warm distilled water until a neutral pH was obtained. They were then dried in an oven at 65 °C for 24 hours before being stored in an environment until the next step.

2.3 Fiber Characterization Method

2.3.1 Tensile test

Thirty-five fibre samples extracted from the three methods were selected. The mechanical property of the fibres was determined by tensile test, carried out on a universal reference testing machine COYON CJ-0820 at a speed of 2 mm/min. the fibres were previously dried in an oven at 105°C for 24 hours (ISO 3344), then cut to a length of 50 mm and then placed on a glass slide and measured with an electron microscope Their diameter. The fibers are then mounted on cardboard supports, and numbered according to the order of diameter measurements. The Test Master software plots stress-strain curves and calculates mechanical parameters. Before mounting the fiber samples on the tensile device, they are packaged in a desiccant formulated at 65% relative humidity using a saturated saline solution. After 24 hours, the fibers are successively installed on the jaws of the universal testing machine, where a laying tension is applied to them before the start of the test.

2.3.2 Weibull Statistical Analysis

The doum palm (*Hyphaene thebaica*) petioles used for fiber extraction were harvested from trees of various ages, resulting in significant variability in their diameter, mechanical properties, and the presence of intrinsic defects. Hence the need to use statistical analysis to determine the extraction method that yields fibers with the best properties [19], [20].

In this study, the two-parameter Weibull distribution was used to evaluate the statistical dispersion of the mechanical properties of doum palm (*Hyphaene thebaica*) petiole fibers obtained using different extraction processes. This approach was applied to the values of tensile strength (σ), elongation at break (ϵ), and Young's modulus (E) [21], [22].

The probability of failure is described by the following equation:

$$P(X) = 1 - \exp \left[- \left(\frac{X}{X_0} \right)^m \right]$$

Where X represents the mechanical property under consideration, X_0 is the characteristic value corresponding to a 63.2% probability of failure, and m is the Weibull modulus, which characterizes the statistical dispersion of the data.

After linearization, the equation becomes:

$$\ln \left[\ln \left(\frac{1}{1-P} \right) \right] = m \ln(X) - m \ln(X_0)$$

The probabilities of rupture were estimated from the increasing ranking of the experimental results according to Bernard's estimator:

$$P_i = \frac{i - 0.5}{n}$$

Where i is the rank of each experimental value and n is the total number of samples tested.

The Weibull parameters were determined by linear regression of $\ln \left[\ln \left(\frac{1}{1-P} \right) \right]$ as a function of $\ln(X)$. The Weibull modulus (m) corresponds to the slope of the regression line, while the characteristic value (X_0) is calculated from the y-intercept. The coefficient of determination (R^2) was used to verify the quality of the fit of the Weibull model to the experimental data $\ln \left[\ln \left(\frac{1}{1-P} \right) \right] = m \ln(X) - m \ln(X_0)$ [21], [23].

2.3.3 Moisture recovery

The hygroscopic behaviour of natural fibres is a decisive parameter for their use as reinforcement in composite materials. Due to their lignocellulosic nature, these fibers possess numerous hydroxyl (-OH) groups capable of binding water molecules, which can lead to a change in their mechanical properties and compatibility with polymer matrices [20], [24], [25]. In this study, the moisture recovery of the *Hyphaene thebaica* extracted according to the different processes was determined by the gravimetric method.

The Moisture Recovery Rate is defined as the amount of water taken up per 100g of dry matter, in an atmosphere at 65% RH [26]. The following equipment is used for this experiment: Oven, 1 mg precision scale, Dryer, Saturated saline solution NH_4NO_3 , Weighing cups.

The samples are cut and then dried in a 105° ventilated oven for 24 hours. The anhydrous samples are then weighed on a 0.001g precision balance and packaged for 24 hours in a desiccant containing a saline solution producing an atmosphere of 65% relative humidity. After 24 hours, the samples are taken and weighed a second time, and the moisture recovery is obtained using the formula below:

$$RH = \frac{m_f - m_0}{m_0} \times 100$$

With m_0 (g) the mass of the dried test piece, m_f (g) the mass of the specimen coming out of the dryer and RH (%) the Moisture Recovery Rate after 24 hours of conditioning [27].

3. Results and discussion

3.1 Influence of extraction methods on mechanical properties

The mechanical properties of the fibres extracted from the petioles of doum palm (*Hyphaene thebaica*) obtained by the three extraction methods were evaluated on a sample of 35 fibres per extraction method. The corresponding stress-strain curves are shown in Figure 1.

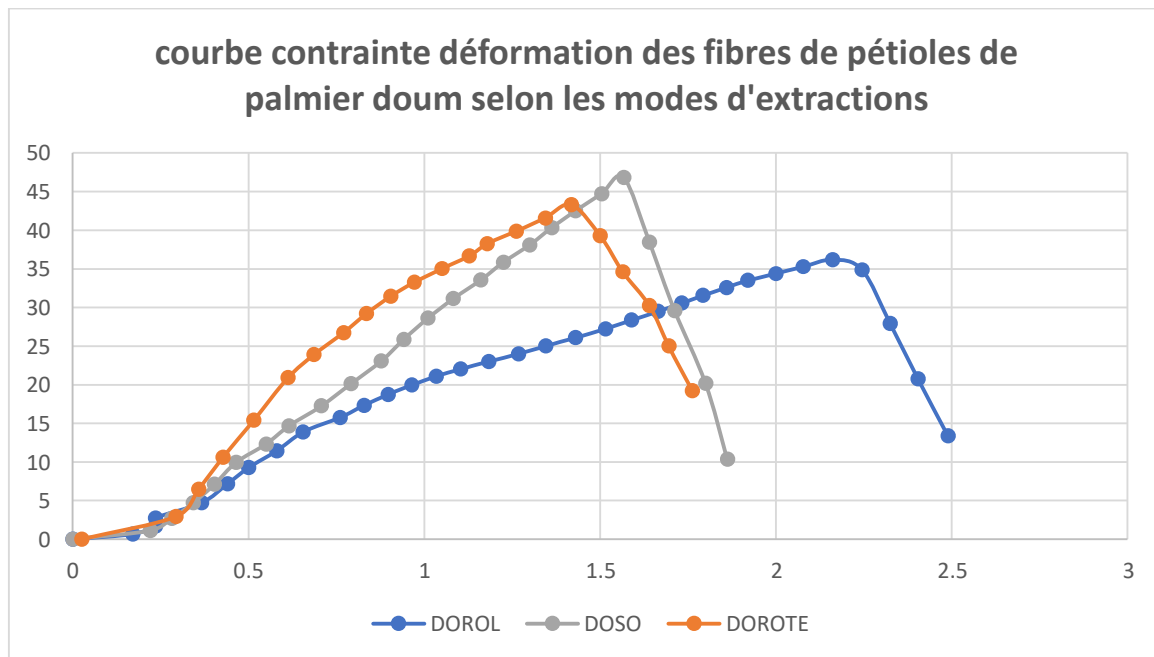


Fig. 1: curve stress deformation of the petiole fibres of the doum palm according to the extraction methods

The stress-strain curves presented in Figure 1 show that: Alkaline fibers (DOSO) have the highest maximum strength (≈ 47 MPa). This improvement is attributed to the removal of some of the hemicelluloses, lignin, pectins and surface waxes by the action of the NaOH solution, which promotes better exposure of cellulose microfibrils and an increase in fibre crystallinity. This interpretation is in agreement with the X-ray diffraction (XRD) results obtained in an earlier study devoted to "the microstructural and thermal characterization of doum palm petiole fibers (*Hyphaene thebaica*)", it was shown that the alkaline treatment led to a higher crystallinity index than that of the fibers obtained by biological processes, reflecting a greater elimination of amorphous constituents, Fourier transform infrared (FTIR) analyses also confirmed this structural modification by the disappearance of

the absorption band located around 1718 cm^{-1} , characteristic of the carbonyl groups of hemicelluloses, reflecting their partial elimination after alkaline treatment [12]. On the other hand, this elimination of amorphous constituents reduces the deformation capacity of the fibers, which explains their low elongation at break ($\approx 1.9\%$) and their more brittle behavior after the stress peak [2], [20], [28].

The fibres obtained by retting underground (DOROTE) develop an intermediate strength ($\approx 43\text{ MPa}$). Unlike alkaline treatment, underground retting acts through the intermediary of microorganisms naturally present in the soil, which gradually degrade the pectic substances as well as some of the hemicelluloses responsible for the cohesion of plant tissues. The FTIR analyses also showed a reduction in the intensity of certain bands associated with the amorphous constituents, confirming a partial change in the chemical composition without significant alteration of the cellulose. XRD results showed that these fibers retain a satisfactory crystal structure, although slightly inferior to that of alkalized fibers. This preservation of the balance between crystalline and amorphous phases explains their good compromise between mechanical strength and deformation capacity. However, the prolonged action of microorganisms can also cause a slight degradation of certain areas of the cell wall, which could explain their relatively brittle breakdown.

The fibres obtained by water retting (DOROL) have the lowest mechanical strength ($\approx 36\text{ MPa}$), but the highest elongation at break ($\approx 2.5\%$). It can be said that retting with water is the least aggressive biological treatment among the processes studied. XRD analyses showed that these fibers retain a lignocellulosic structure where amorphous constituents remain relatively present, despite good crystallinity. FTIR analyses also reveal the presence of the characteristic bands of lignin and hemicelluloses, reflecting a lower elimination of these compounds. This preservation of amorphous constituents improves the flexibility of the fibers and explains their greater ductility. On the other hand, it could limit the transmission of mechanical stresses to cellulose microfibrils, which would justify the lower tensile strength than that observed for alkalized fibers and underground retted fibers.

3.2 Weibull Analysis

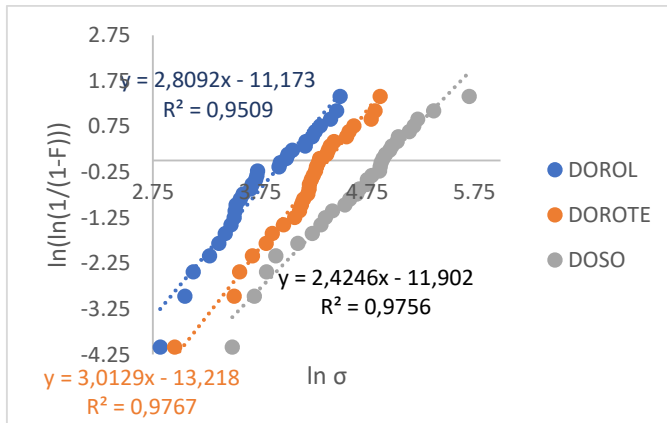


Fig. 2:D Weibull distribution of the tensile stress of doum palm fibers

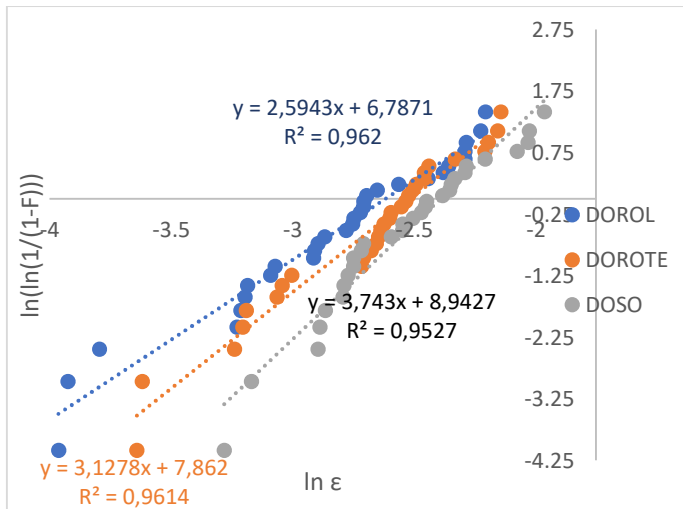


Fig. 3: Weibull distribution of the tensile elongation of doum palm fibers

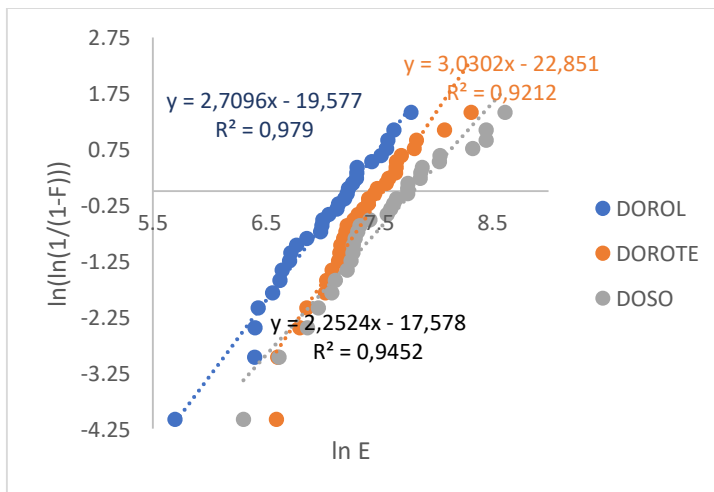


Fig. 4:D Weibull distribution of Young's modulus in tension of doum palm fibers

The Weibull analysis was applied to the three mechanical parameters (σ fracture stress, ϵ fracture deformation and Young E 's modulus) of the extracted fibers according to the three processes studied (DOROL: water retting; DOROTE: retting underground; and DOSO: soda extraction) in order to characterize their statistical homogeneity and mechanical reliability. The

Weibull modulus m , which reflects the degree of dispersion of the properties (a high value indicating homogeneous and predictable fibres), varies from 2.25 to 3.74 depending on the parameter and the process considered. This range is consistent with the values reported for other lignocellulosic fibres (flax, jute, hemp, sisal, date palm), which are generally between 2 and 5 [23], [29]. The coefficients of determination ($R^2 > 0.95$ in all cases) confirm the relevance of the Weibull model to describe the distribution of these three quantities.

The DOROTE process consistently shows the best statistical homogeneity, with the highest Weibull moduli in stress ($m = 3.01$) and stiffness ($m = 3.03$), and a high intermediate strain behaviour ($m = 3.13$). This result confirms that the microbial action of the soil acts uniformly and selectively on the cell wall, limiting the dispersion of structural defects and giving the fibers a regular and predictable mechanical behavior over all the properties studied.

DOSO, on the other hand, shows a contrasting behaviour depending on the parameter considered: the lowest Weibull modulus in stress ($m = 2.42$) and in stiffness ($m = 2.25$), reflecting a strong heterogeneity linked to a non-uniform attack of the soda on the lignocellulosic structure, but also the highest modulus in deformation ($m = 3.74$). This contrast suggests that the alkaline treatment homogenizes the removal of non-cellulosic components, resulting in regular fracture deformation, while introducing areas of local brittleness that vary from one fiber to another.

3.3 Moisture recovery

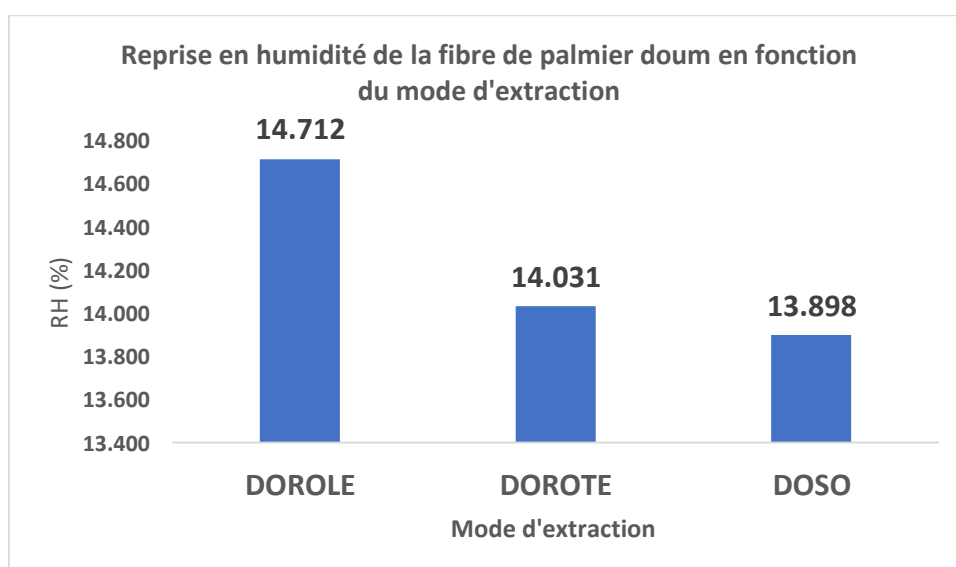


Fig. 5: moisture recovery of doum palm petiole fibres according to their extraction method

The moisture recovery results show that the fibres obtained by water retting (DOROL) have the highest moisture absorption capacity (14.712%), followed by fibres obtained by retting

underground (DOROTE) (14.031%) and the fibres treated by alkaline means (DOSO) (13.898%). These differences highlight the influence of the extraction process on the hygroscopic behavior of lignocellulosic fibers.

The high moisture recovery observed for DOROL fibres is explained by the relatively gentle nature of retting with water, which preserves a greater proportion of the amorphous constituents, in particular hemicelluloses, which are recognized as the most hydrophilic components of the cell wall due to their high density of hydroxyl groups ($-OH$). The FTIR analyses carried out in our study confirm this interpretation, since the characteristic bands of hemicelluloses remain present on the DOROL fibers, reflecting a significant conservation of these compounds.

Conversely, DOSO fibers have the lowest moisture recovery. This reduction is directly linked to the alkaline treatment, which removes some of the hemicelluloses, pectins and surface waxes. The FTIR results showed in particular the disappearance of the band located around 1718 cm^{-1} , characteristic of the carbonyl groups of hemicelluloses, confirming their partial dissolution under the action of soda. In addition, XRD analyses showed an increase in the crystallinity index of DOSO fibers, reflecting a higher proportion of crystalline cellulose, which is less accessible to water molecules than amorphous regions.

DOROTE fibers exhibit intermediate behavior. The action of soil microorganisms leads to a partial elimination of amorphous constituents without reaching the level of modification observed during the alkaline treatment.

These results show that the extraction process directly influences the chemical structure, the crystallinity, which controls their hygroscopic behavior. This property is of particular importance in the field of polymer composites, where low moisture absorption promotes dimensional stability, limits hygrothermal aging and contributes to the maintenance of mechanical properties during service[3], [20], [28].

3.4 Multi-criteria analysis

Table 1: Multi-criteria analysis of Hyphaene thebaica petiole fibers according to the extraction process

Criterion	DOROL (Water retting)	DOROTE (Retting underground)	DOSO (NaOH Treatment)	Best Process

Maximum Strength (MPa)	≈ 36	≈ 43	≈ 47	DOSO
Deformation at break (%)	≈ 2.5	≈ 1.75	≈ 1.85	DOROL
Weibull Modulus (Constraint)	2,81	3,01	2,42	DOROTE
Weibull modulus (Deformation)	2,59	3,13	3,74	DOSO
Weibull modulus (Young's modulus)	2,71	3,03	2,25	DOROTE
FTIR	High presence of hemicelluloses	Partial Elimination	Disappearance of the band C=O (1718 cm^{-1})	DOSO
Moisture recovery (%)	14,712	14,031	13,898	DOSO
Overall Homogeneity	Good	Very good	Medium	DOROTE
Potential for composites	Good	Very good	Excellent	DOSO

This multi-criteria analysis shows that each extraction process gives the fibres specific properties. The alkaline treatment (DOSO) produces the fibers with the highest mechanical strength, the strongest crystallinity, and the lowest moisture recovery, which favors their compatibility with a polymer matrix. On the other hand, this method generates a greater dispersion of certain mechanical properties, reflecting a local heterogeneity induced by the alkaline attack.

Underground retting (DOROTE) appears to be the most balanced process. It has the highest Weibull moduli for stress and Young's modulus, reflecting excellent statistical homogeneity and good mechanical reliability. This process is therefore the best compromise between strength, rigidity and regularity of properties.

Water retting (DOROL) retains more of the amorphous constituents of the fibre. This characteristic gives it the best ductility, but also a greater moisture recovery and more modest mechanical performance.

4. Conclusion

The objective of this study was to evaluate the influence of three processes for extracting fibres from doum palm petioles (*Hyphaene thebaica*): water retting (DOROL), underground retting (DOROTE) and alkaline soda treatment (DOSO) on their mechanical and hygroscopic properties, in order to identify the most appropriate method for their use as reinforcement of polymer matrix composites.

The results obtained show that the extraction process significantly influences the behavior of the fibers. Tensile tests showed that fibres extracted by alkaline treatment (DOSO) have the highest mechanical strength (≈ 47 MPa), while fibres obtained by retting underground (DOROTE) have an intermediate strength (≈ 43 MPa). Fibres from water retting (DOROL) have the lowest strength (≈ 36 MPa), but the highest deformation capacity at break, reflecting a more ductile behaviour.

Statistical analysis based on Weibull's law was used to evaluate the mechanical reliability of the fibers. DOROTE fibers have the highest Weibull modulus for stress ($m = 3.01$) and Young's modulus ($m = 3.03$), reflecting excellent homogeneity of mechanical properties. DOSO fibers have the best homogeneity of deformation at break ($m = 3.74$), although their dispersion in strength and stiffness remains greater.

The study of moisture recovery also shows that the extraction process influences the hygroscopic behaviour of the fibres. DOROL fibres have the highest moisture recovery (14.712%), while DOSO fibres have the lowest (13.898%), confirming that the removal of amorphous constituents reduces the number of hydrophilic sites available for the adsorption of water molecules.

The multi-criteria analysis carried out on the basis of all the results shows that each process has specific advantages. Alkaline treatment optimizes mechanical performance, crystallinity, surface quality and moisture stability, while underground retting ensures the best statistical homogeneity of mechanical properties. Water retting, on the other hand, retains more of the ductile character of the fibres.

Overall, this study demonstrates that the petiole fibers of *Hyphaene thebaica* are a promising lignocellulosic reinforcement for the development of bio-based composites. The results obtained show that the choice of extraction process must be made according to the desired performance. For applications requiring high mechanical strength and good hygroscopic

stability, alkaline treatment appears to be the most efficient solution, while underground retting is the best compromise between homogeneity, mechanical reliability and overall performance.

This work provides a scientific basis for the valorization of doum palm petiole fibers (*Hyphaene thebaica*) as reinforcement of composite materials.

The prospects of this research will focus on the development of epoxy matrix composites reinforced by the selected fibers, as well as on the study of their mechanical, thermal, dynamic properties and durability under hygrothermal aging conditions.

Declarations

Funding: This research received no external funding.

Consent for publication: Not applicable.

Availability of data and materials: The datasets generated and/or analyzed during the current study are available from the corresponding author upon reasonable request.

Ethics approval and consent to participate: The plant material used in this study was obtained from wild *Hyphaene thebaica* palms naturally growing in the northern region of Cameroon. The petioles were recovered during the pruning and removal of older palm fronds, in accordance with local practices, to valorize vegetal waste. The collection was carried out in accordance with applicable local guidelines and did not involve endangered or protected plant species.

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