Research article

Influence of sampling area and extraction method on the thermal, physical and mechanical properties of Cameroonian Ananas comosus leaf fibers

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A R T I C L E   I N F O

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A B S T R A C T

The wide dispersion of the properties of plant fiber bundles further limits their use. In this study, manual and retting methods were used to extract fiber bundles from the available and underutilized Ananas comosus (AC) leaf waste in Cameroon. These fibers were sampled in three zones: base, middle and head. The fibers extracted by retting in the different zones were found to have better thermal stability. A 2.5% solution of sodium hypochlorite was used for bleaching the various pineapple fibers. Density, fineness, functional groups, mechanical properties, water absorption and morphology of different fibers were characterized. Density and fineness decreased from the head to the base of the leaf and were lower for manually obtained fiber bundles. The peaks of the infrared spectra associated with the functional groups of the pineapple fibers change very little along with the leaf but are clearly more pronounced for the fibers extracted manually. Fibers in the middle of the leaf have been found to have slightly better mechanical and water absorption properties. All fiber bundles can rapidly absorb water following simple exponential kinetics. Bleaching partially removes non-cellulosic materials from the fibers with a transverse shrinkage effect, which improves their fineness, density and hydrophilic function. Unfortunately, it reduces their tensile strength and fracture toughness. These results show that the whole leaf can be used without restriction to extract manually or by retting the fiber bundles intended for the manufacture of textiles and composites. Furthermore, bleaching with sodium hypochlorite seems ineffective due to fiber degradation.

1. Introduction

Plant fibers are an interesting alternative to synthetic fibers which are frequently used as textile fibers and reinforcing fibers for polymer or ceramic matrix composites (Baley et al., 2020; Rajesh et al., 2020). Examples of bio-based fibers with textile and reinforcement potential are cotton (Doraiswamy et al., 1993), flax (Baley et al., 2020), jute (Fidelis et al., 2013), hemp (Yu and Frank, 2005), kenaf (Shaeifah and Martin, 2004) and sisal (Belaadi et al., 2013). These low-density biobased materials are inexpensive and have specific properties that are acceptable for the development of new high-performance biodegradable and environmentally friendly materials (Bledzki and Gassan, 1999; Singha and Rana, 2012). Many studies have however shown that plant fibers are generally limited by a wide dispersion of their physical, thermal and especially mechanical properties due to their natural character, experimental analysis conditions (Dumont et al., 2017), plant growth (soil and climate) (Mediavilla et al., 2001), degree of maturity of the plant (Pickering et al., 2007), position of fibers in the plant (Charlet et al., 2007), fiber extraction and processing methods (Kabir et al., 2012; Reddy et al., 2013). In addition, defects such as stiffness, yellowing, roughness and hard feeling limit the use of lignocellulosic fibers in the manufacture of value-added textile fabrics or fabrics (Hassan and Saifullah, 2019).

The fibers that reinforce the structure of pineapple leaves are used for the manufacture of high quality Pinatex ropes and leather (Asim et al., 2015). Many studies (Reshmy et al., 2020; Prado et al., 2020) have demonstrated the textile potential and the composite reinforcement potential of these fibers. Pineapple fiber is a secondary lignocellulosic fiber obtained from agricultural by-products or pineapple leaves and contains mainly cellulose (55–70%) and non-cellulose materials, namely hemicelluloses (15–20%), lignin (8–12%), pectin (2–4%), and low proportions of extractables (1–3%) (waxes and proteins) (Pandit et al., 2020; Betene et al., 2020). Its crystalline structure is essentially

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composed of type I cellulose with crystallinity indices between 61-79\% (Neto et al., 2015). The angle of the microfibers that make up its structure is between 8° and 15° (Jawaid et al., 2020). Its density (1.26–1.5 g.cm\(^{-3}\)) is equivalent to other natural fibers (cotton, jute, linen, kenaf and banana). It can be extracted in the form of fiber bundles manually, mechanically or by retting (Rafiqah et al., 2020). Due to its high moisture content and hydrophilicity, the dye absorption tendency of pineapple fiber is higher than that of cotton (Doraiswamy and

Figure 1. Image showing the main steps of the pineapple fiber extraction process: a) by manual scraping and b) by retting in stagnant water. SEM micrographs of the raw fiber bundles of c) ACR and d) ACG.
Chellamani, 1993; Yu and Frank, 2005). However, there are very few studies on pineapple fibers originating from regions of Cameroon. In a recent study (Betene et al., 2020), it was shown that pineapple fibers of Cameroonian origin can withstand service temperatures of around 200 °C. In all these studies, the fibers were randomly selected along the leaf without considering their location, which could partly explain the large dispersion of their properties and consequently limit their use. In addition, the pineapple leaf is long and narrow, so it is necessary to determine the influence of the sampling area in a leaf on the fiber properties of pineapple.

Bleaching is necessary to remove the yellowish color from vegetable fibers and other non-cellulosic materials such as hemicelluloses, lignin and layers of waxes, which will improve the fineness of the fiber and enhance the fixation of reactive dyes (Wang et al., 2008). Pandit et al. (2020) reported that bleaching pineapple fibers with hydrogen peroxide (H₂O₂) reduce the mass content of non-cellulose materials and improves fineness by 5–6%. Unfortunately, the authors report that bleaching with H₂O₂ reduces the tensile strength of 40–50% of raw pineapple fibers. In addition, H₂O₂ bleaching consumes a lot of energy, since it is usually applied at a high alkaline condition (pH between 10.5 and 12) and at a high temperature close to boiling (Fang et al., 2000; Wang et al., 2020). For vegetable fibers, chlorine and its derivatives are the most effective bleaching agents due to their superior delignification capacity (Girijappa et al., 2019; Wang et al., 2020). Sodium hypochlorite (NaClO) is a common and inexpensive chemical solution with many uses such as disinfection and bleaching. To our knowledge, there are no studies that focus on NaClO bleaching of plant fibers. However, its use in the care of clothing made from plant fibers (for example, cotton, and jute) is very common in households. In order to promote the use of fibers in clothing textiles, it is crucial to understand the effect of NaClO on the physical and mechanical properties of natural textile fibers.

The present study aims to determine the influence of two extraction techniques and the leaf sampling area on the physical, thermal and mechanical properties of Ananas comosus fibers. Thermogravimetric analysis were performed on the different fibers to identify their thermal degradation properties. In addition, the effect of sodium hypochlorite bleaching on fineness, density, water absorption, and tensile properties was studied. SEM and ATR-FTIR analysis were performed to better confirm and compare the effect of bleaching on the removal of non-cellulose materials.

2. Materials and methods

2.1. Raw materials

Ananas comosus (AC) leaf waste was collected from a private plantation in the town of Penja on the Cameroonian coast. The average temperature of this locality is 31 °C, with a relative humidity of 73%. Leaves were randomly selected from mature plants directly after harvesting pineapple fruits.

2.2. Fiber extractions

Pineapple leaf fibers were extracted by two methods: manual and retting.

(1) Manual scraping extraction was performed on freshly collected leaves using a table as a support and a kitchen knife as a scraping tool. The process involved scraping the pineapple leaf, gently peeling and scraping the mat of fiber bundles, then washing the fiber bundles, as shown in Figure 1a. The fiber bundles obtained were dried at room temperature for 24 h and then stored in polyethylene bags.

(2) For retting extraction (Figure 1b), the collected pineapple leaves were immersed in a pool of water from the national distribution network for 10 days at room temperature. The fiber bundles were thus extracted from each retted leaf as described for the previous method.

The two types of fiber bundles obtained (Figure 1c and d) have a length of the same order as the base leaf (between 50 cm and 65 cm). The SEM micrographs (Figure 1c and d) show that these fiber bundles are made up of several dozen elementary fibers, mucilage from the leaves and impurities. Elemental fibers (20–115 mm long) are more visible on the fiber bundles obtained by retting (ACR) than on the bundles obtained by the manual method (ACG), indicating that the retting technique is effective in reducing mucilage and surface impurities fibers.

2.3. Fiber bundles sampling

As the pineapple leaf has a long and narrow shape, three sampling areas were defined on the extracted fiber bundles (Figure 2): the bottom (15 cm above the base of the fiber bundle), the middle (between the bottom and the middle) and the top (15 cm below the base of the top of the fiber bundle).

2.4. Thermogravimetric analysis (TGA) of raw fibers

The thermal properties of the pineapple fiber samples were determined using a TGA Q50-0836 Instruments Thermal analyzer. For this analysis, samples of 4 mg of pineapple fibers ground to a size of 110 μm were heated from room temperature to 650 °C at a heating rate of 10 °C.min⁻¹ under a nitrogen atmosphere (flow rate = 10 ml.min⁻¹).

2.5. Sodium hypochlorite treatment of fiber bundles

A bleach solution of 250 ml of sodium hypochlorite (NaClO) of 2.5% concentration was prepared in a glass container. The pineapple fibers were immersed in this solution for 15 min. The mixture was stirred throughout the process under ambient atmosphere (25 ± 1 °C, 48 ± 2 HR). The bleached fibers were rinsed several times with distilled water until a neutral pH was obtained, dried in an oven at 105 °C for 24 h and then cooled in a desiccator.
stored in polyethylene bags. In the following, the bleached pineapple fiber bundles initially obtained by manual retting and scraping will be referred to as ACRW and ACGW respectively.

2.6. Characterisation of raw and processed fiber bundles

2.6.1. Fourier transform infrared (FTIR) spectroscopy analysis

FTIR analysis was performed using a Bruker Alpha-P spectrometer equipped with an ATR module and controlled by Opus/Mentor software. Samples of a few milligrams of powder (size = 315 μm) of pineapple fibers were scanned over a spectral region of 4000 to 400 cm\(^{-1}\) with 32 scans, giving a resolution of 4 cm\(^{-1}\). FTIR spectra were recorded in absorbance mode.

2.6.2. SEM analysis and diameter determination of fiber bundles

The surface states of the fiber samples were observed using a Hitachi S-3500N scanning electron microscope. Before the observations, the samples were covered with a layer of metal alloy (palladium/gold) to make them conductive.

The average diameter was obtained from the diameters measured with the SEM at three different points (the two ends and the middle) along the fiber. This technique is commonly used by researchers (Silva et al., 2008; Placet et al., 2012) to assess the average diameter of plant fibers. Moreover, to simplify the analysis (Rosa et al., 2010; Hanana et al., 2013), the fiber bundles were considered perfectly cylindrical. Sixty x80 magnification SEM micrographs of the central area were used to determine the diameter distributions.

2.6.3. Measurement of the fineness of fiber bundles

The fineness index \(f_f\) (in tex) of the raw and bleached fibers was evaluated by gravimetry according to the ISO 1889 standard (Vasugi et al., 2019).

2.6.4. Density measurement

The standard pycnometer technique for solids with toluene \((\rho = 0.866 \text{ g cm}^{-3}\) at room temperature) was applied to perform fiber density measurements according to ASTM D 2320-98. Before the experiment, the fibers were cut into 5 mm lengths and then dehumidified at 105 °C for 24 h. The measurements were carried out on a balance with a sensitivity of 0.1 mg. The density \(\rho\) was determined by Eq. (1):

\[
\rho = \rho_t \frac{m_3 - m_0}{(m_1 - m_0) - (m_3 - m_2)}
\]

where \(m_0\) is the mass of the empty pycnometer, \(m_1\) is the mass of the pycnometer filled with toluene at room temperature, \(m_2\) is the mass of the pycnometer filled with fibers and \(m_3\) is the mass of the pycnometer filled with fibers and toluene at room temperature.

\[
W_{abs} = 100 \times \frac{(m_f - m_0)}{m_0}
\]

where \(m_f\) is the mass of the sample at time \(t\) and \(m_0 = 100 \pm 5\) mg is the mass of the dried sample at initial time \(t_0\).

2.6.6. Fiber bundles tensile tests

The tensile tests were carried out on the bundles of pineapple fibers using an LDW-5 universal testing machine according to standard NF T25 501-2. The fiber bundles specimens were fabricated with a gauge length of 10 mm (Betene et al., 2021) and conditioned in a humidifier for 1 h at 23 °C and 50% relative humidity. The test was performed in an ambient environment \((23 \pm 1 ^\circ \text{C and 50 HR})\) with a constant speed of 2 mm min\(^{-1}\). Due to the variability in fiber properties, at least 25 fiber specimens from each sampling area were tested.

The tensile strength was calculated considering the apparent cross section of the circular fiber bundle. The area of this cross-section was calculated from the average bundle diameter of the fiber, while taking into account an average porosity rate of 30% reported by Betene (2021). Young’s modulus was measured as the slope of the curve. The fracture toughness was obtained by dividing the breaking strength by the fineness.

3. Results and discussions

3.1. Thermal degradation properties

Figure 3a and b present the TG and DTG curves of raw ACG and ACR respectively for the three sampling areas. The thermograms of fiber bundles from different parts are not too different, and are similar to those of Indonesian pineapple fibers (Jawaid et al., 2020). When ACG and ACR fiber bundles are exposed to high temperatures, they degrade similarly to other natural fibers (Sango et al., 2018). In Figure 3a), the ACG fiber bundles showed an initial mass loss between 35 °C and 114 °C related to
Ananas C.*
Banane* 40
Jute* 40
Sisal 50

ACGW(Top)* 68.6
ACGW(Bottom)* 96.1
to the degradation of hemicelluloses, pectin, cellulose, lignin and wax. In
ACRW(Middle)* 75.2
Agave
ACR(Top)* 106.4
Banane* 40-140 13-17
Ananas C. 70-315 2.5-5.5
Ananas C.* 100-280 3.4-5.7
Ananas C. 37-96
Hemp 150-263 –
Hemp* 257 ± 62 –
Agave* 171-403 12-24
Sisal 50-300 -
Urena lobata* – –
Kenaf* – 9-15.5
Alfa* 150-350 6.5-7.7
Typha* – 5.8-8.7

ACR(Top)* 106.4 ± 18.0
ACR(Middle)* 129.7 ± 40.2
ACR(Bottom)* 142.6 ± 47.6
AGC(Top)* 83.2 ± 13.7
AGC(Middle)* 90.9 ± 44.8
AGC(Bottom)* 118.4 ± 51.2
ACRW(Top)* 56.1 ± 8.0
ACRW(Middle)* 75.2 ± 22.2
ACRW(Bottom)* 82.9 ± 27.8
AGGW(Top)* 68.6 ± 32.0
AGGW(Middle)* 74.8 ± 11.1
AGGW(Bottom)* 96.1 ± 45.0

Table 2. Physical and mechanical properties of raw and bleached pineapple fibers extracted by two methods from three sampling areas and comparison with some other plant fibers.

| Fibers      | Diameter (μm) | Fineness (tex) | Density (g.cm−2) | Tensile strength (MPa) | Young modulus (GPa) | Tenacity (gN теку) | Elongation break (%) | References                  |
|-------------|---------------|----------------|------------------|------------------------|---------------------|-------------------|---------------------|--------------------------|
| Cotton      | 317-1739      | –              | 1.56-1.6         | 287-597                | 5.5-12.6            | 28-48             | 3-12.6              | Domiswamy et al. (1992)   |
| Flax        | 270-900       | 1.07-1.5       | 1.56-1.6         | 287-597                | 5.5-12.6            | 28-48             | 3-12.6              | Domiswamy et al. (1992)   |
| Jute*       | 40-134        | 1.1-1.5        | 393-773          | 265                    | 50-70               | 25-26             | 2.7-3.3             | Bezakou et al. (2008)     |
| Banane*     | 347-1035      | 1.4-1.5        | 345-1035         | 26-50                  | 70-90               | 25-26             | 1.2-1.9             | Bezakou et al. (2008)     |
| Ananas C.   | 140-676       | 1.35           | 126-1627         | 42-83                  | 11-34               | 3-2-6             | 1.4-3.4             | Sangi et al. (2013)       |
| Ananas C.*  | 154-675       | 1.52-1.56      | 350-600          | 40-80                  | 9-39                | 34-65             | 2.6-4.8             | Javahid et al. (2021)     |
| Hemp        | 140-360       | 1.3-1.5        | 80-360           | 9-24                   | 21-41               | 2-4               | 1.7-3.2             | Nkemajia et al. (2020)    |
| Hemp*       | 147-900       | 1.3-1.5        | 23.5-90          | 13-24                  | 23-70               | 47-67             | 1.6-2.4             | Bezakou et al. (2008)     |
| Agave*      | 141-321       | 1.1-3          | 63-211           | 1.1-3                  | 21-41               | 2-4               | 1.4-3.4             | Mshali et al. (2006)      |
| Sisal       | 80-360        | 9-39           | 21-41            | 1.1-3                  | 21-41               | 2-4               | 1.4-3.4             | Mshali et al. (2006)      |
| Urena lobata* | 34-65        | 2.6-4.8        | 21-41            | 1.7-3.2                | 23-70               | 47-67             | 1.6-2.4             | Grassey et al. (2020)     |
| Kenaf*      | 350-600       | 19-103         | 14-22            | 2.8-3.9                 | 23-70               | 47-67             | 1.6-2.4             | Bezakou et al. (2008)     |
| Alfa*       | 134-220       | 16-77          | 8.5-124          | 16-77                  | 23-70               | 47-67             | 1.6-2.4             | Dalhel (2012)             |
| Typha*      | 9-14          | 3.5-5.5        | 1.4-1.45         | 1.4-1.45                | 23-70               | 47-67             | 1.6-2.4             | Sana (2016)               |

The evaporation of water and volatile matter (between 3.5% and 6.5% by weight), followed by thermal stability in the 210–230 °C range, and significant mass loss (between 70% and 84% of the initial fiber mass) due to the degradation of hemicelluloses, pectin, cellulose, lignin and wax. In general, the thermal depolymerization of the glycosidic bonds of cellulose occurs between 220 and 310 °C and that of α-cellulose takes place between 310 and 390 °C, while that of lignin occurs slowly over the entire decomposition temperature range (210–450 °C) (Rosa et al., 2010).

Table 1 compares the thermal degradation properties of different fiber bundles. It is noted that the location of the fiber in the leaf and the method of extraction play an important role on the thermal stability, the temperature at the peak of degradation of hemicellulose and cellulose, the content of volatile matter and the rate of residues. The highest thermal stabilities were obtained for fiber bundles produced by retting. This result may be associated with the partial removal of non-cellulose material and other impurities that coat the fibers during retting (Figure 1). In general, the elimination of non-cellulosic materials is accompanied by an improvement in the thermal degradation properties of natural fibers (Thirumurugan et al., 2019). It is also noted that these properties decrease progressively from the lower part to the upper part. This could be associated with the growth of the plant and its different varieties (Neto et al., 2015).

The residue content is between 4.8% and 20.9% by weight. ACR(Middle) samples showed the highest residue content values (20.9%), indicating that the middle pineapple fiber bundles obtained by retting have the highest amounts of inorganic substances. These residue levels are higher than the pineapple fiber varieties of Neto et al. (2015). The

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DTG curve (Figure 3b), shows that no samples of ACR and ACG show significant weight loss above 550 °C.

The thermal stability is considered as the threshold temperature of degradation. When natural fibers are used in the implementation of polymer matrix composites or in surface treatments in hot environments, thermal stability must be taken into account (Neto et al., 2015; Betene et al., 2020). On the criterion of thermal stability, two classes of fibers can be distinguished: the first class associated with thermal stability values ranging from 210 °C to 217 °C includes all ACG and ACR(Top), and the second class includes ACR(Middle) and ACR(Bottom) with higher values of 227 °C–230 °C. Class 1 thermal stability values are higher than those of pineapple fibers from a recent study (Betene et al., 2020), as well as those of other natural fibers (Tiwari and Sarangi, 2022). Class 2 has much higher values than fiber from Neopeltis A. (220 °C), Rhecktophyllum C. (220 °C), okra (222 °C), sisal (222 °C) (Betene et al., 2020), banana (219 °C) (Sango et al., 2018), but not higher than those of Aloe vera (238 °C), Sida rhombipohila (250 °C) and Acacia L. (280 °C) (Reported by Tiwari and Sarangi, 2022), and pineapple fibers (240–272 °C) reported by Neto et al. (2015). The thermal stabilities thus identified provide details on the maximum service temperatures of the fibers studied for forming applications of thermoplastic composites.

3.2. Physical and morphological properties of raw and bleached fibers

3.2.1. Density

The values of the densities of the different fibers under study are presented in Table 2. The densities of the fibers of the different positions vary between 1.14 g.cm⁻³ and 1.45 g.cm⁻³. The fiber values of the upper parts are the lowest, while those of the lower parts are the highest. These values are within the range of values reported for Pineapple comosus, jute and hemp (Asim et al., 2015; Betene et al., 2020). In addition, the use of Cameroonian pineapple fibers could significantly contribute to reducing the mass of textiles and composites compared to cotton fibers (1.5–1.6 g.cm⁻³) (Charlet et al., 2007) and of glass (2.5 g.cm⁻³) (Bledzki and Gassan, 1999).

The low values of the standard deviations of the different parts indicate that taking into account the location of the fibers in the leaf makes it possible to reduce the dispersion of the density, which makes it possible to facilitate their choice in the manufacture of objects. Nevertheless, different parameters can explain these slight deviations such as the climatic conditions and the maturity of the plant, as well as the morphology of the fiber (Baley, 2002).

The retting extraction method appears to be more effective in improving the density of pineapple fibers compared to manual
extraction, which is in good agreement with the observations of the micrographs in Figure 1. The lower density values (compared to raw fibers ACR and ACG) of bleached pineapple fibers (ACRW and ACGW) indicate that removal of non-cellulosic substances (hemicellulose and lignin) is more important than fiber shrinkage during processing NaClO bleaching process.

3.2.2. Fiber bundle fineness

The fineness indices of the raw and bleached fiber bundles extracted by the two methods from three different positions in the pineapple leaf, namely top, middle and bottom, were determined and the results are shown in Table 2. Fineness of raw and bleached pineapple fibers decreases progressively from the head towards the base of the leaf for the two extraction methods, which corresponds to the results obtained for the density.

For raw fibers, the fineness indices of the fiber bundles of the different zones are not very different. The fineness indices of ACR fiber bundles are slightly lower than those of ACG. These slight deviations can be attributed to the irregularities and differences observed in Figure 4c and d. For bleached fibers, the fineness indices of ACR fibers become lower than those of ACG.

In comparison with raw fibers, the fineness indices of bleached fibers are lower. This improvement is linked to the partial elimination of non-cellulose materials on the surface of the fibers (Wang et al., 2020). The greatest yields of fineness improvement are 29% and 23% for ACRW(Top) and ACGW(Top) fibers, respectively. Therefore, NaClO bleaching is more effective in improving the fineness of retted fibers of the upper part of the leaf. However, the yields of the central and lower zones are not very different, they are in the 11–12% range. The values obtained in this study are larger compared to the range of values (2.5–5.5 tex) reported

Figure 5. SEM micrographs of a) ACG(Middle), b) ACGW(Middle), c) ACR(Middle) and d) ACRW(Middle).

Figure 6. Effect of bleaching on the diameter distributions of fiber bundles extracted manually and by retting from the middle part of the pineapple leaf.
by Pandit et al. (2020). This variation is probably due to a difference in the pineapple varieties studied (Neto et al., 2015) and to a lesser extent at the scale of the fiber tested (fiber bundles or elementary fibers) and to the climatic growing conditions. However, these values compare very well with those of kenaf and esparto fibers already used in the textile field (Dallel, 2012).

In order to validate the effects of sampling area and extraction method, as well as NaClO bleaching on fineness, the experimental data obtained were analyzed with the continuous Weibull probability distribution and reported in Figure 4. The figure shows the non-linear evolution of the double logarithm of the probability of failure $P_f$ as a function of the logarithm of the fineness $\mu$ of the fibers. This dispersion may be associated with the natural character of pineapple fibers (Dumont et al., 2017). However, the layouts of the distributions on the figure confirm earlier discussions that the fineness indices of the fiber bundles in the upper zone of the leaf are lower, while those of the fiber bundles in the lower zone are higher. Furthermore, the distributions of bundles bleached by NaClO are weaker than those of raw fibers.

### 3.3. Morphology and diameter distribution

Scanning electron microscopy observations revealed that the NaClO bleaching process removed non-cellulosic materials (hemicelluloses, lignin, pectin, wax and fats).

SEM images (Figure 5b and d) confirmed the shrinkage in the transverse direction of the fibers during the bleaching process. Compared to ACGW bleached fibers (Figure 5b), ACRW bleached fibers (Figure 5d) exhibit a cleaner and smoother surface with more microcracks and wrinkles. The observations are in good agreement with the properties results reported in Table 2. These results are also in agreement with those of Pandit et al. (2020) who reported that the removal of non-cellulosic materials by bleaching products (hydrogen peroxide) slightly damages the fibers. Figure 5b and d also show that the elementary fibers are still linked together, which confirms that the elimination of non-cellulosic materials has been partial.

Figure 6 shows the asymmetric distributions of the diameters of the raw and bleached fiber bundles of the middle zone. The diameters vary in the range 60.5–237.1 μm, 45.0–192.7 μm, 26.7–133.6 μm and 38.8–105.7 μm for ACR(Middle), ACG(Middle), ACRW(Middle) and ACGW(Middle) respectively. In addition, Table 2 reports the average values of the diameters of the different fiber bundles in this study. The values of the average diameters are in the range 56.8–142.4 μm. The highest value was obtained for the ACR(Bottom) sample, which is in the range 100–280 μm (Jawaid et al., 2020) and 70–315 μm (Asim et al., 2015) for pineapple fiber bundles, but higher than that reported in the work of Neto et al. (2015) for pineapple fibers.

Table 2 also makes it possible to compare the average diameters of the fiber bundles according to the extraction method and their location in the leaf. Depending on the type of extraction, it is observed that the fiber bundles produced by retting have higher average diameters, indicating that the retting process has caused the fiber bundle to swell.

When pineapple fiber bundles undergo NaClO treatment, the diameters decrease. The reduction rate of ACR is higher (38–45%) than that of ACG (16–21%), which shows that the removal of non-cellulosic materials is more successful for ACR. The decrease in diameter is obviously greater for the middle and lower fiber bundles. Furthermore, the
values of the mean diameters and their relatively large standard deviations can be considered to be statically large. This shrinkage in the transverse direction is likely to make the fiber harder, less strong and less tenacious (Pandit et al., 2020).

3.4. Mechanical properties of raw and bleached fiber bundles

Figure 7a shows the average stress-strain and tangent-strain modulus curves obtained from tensile tests performed on 25 specimens of raw ACR and bleached ACRW fiber bundles from the central part, respectively. These curves show a linear behavior for the raw fiber bundles and a bilinear behavior for the bleached fiber bundles. Regarding the three types of tensile behavior of plant fibers described in Duval et al. (2011), the tensile curve of ACR raw fibers bundle is similar to type I (elastic behavior characterized by a straight line), already observed by Budsaraporn et al. (2019). The curves of ACRW bleached fiber bundles could approach the type II obtained by Neto et al. (2015) for pineapple fibers, and Duval et al. (2011) for certain hemp fibers and Ndoumou et al. (2022) for fibers from Cameroonian Cola lepidota. Interestingly, all raw fiber bundles (ACR and ACG) and bleached fiber bundles (ACRW and ACGW) exhibited behaviors similar to those associated with ACR(Middle) and ACRW(Middle), respectively.

The tangent modulus curves in Figure 7a show that NaClO treatment plays an important role in the tensile behavior of pineapple fiber bundles. For the raw fiber bundle sample, the Young’s modulus is almost constant up to a strain of 4.2%. This evolution was similar regardless of the type of extraction and location of the fiber. For bleached fibers, a decrease in tangent Young’s modulus is observed around 1% strain, followed by an almost constant evolution that decreases again at 4.1% strain. This variation in the tangent modulus of the treated fibers does not depend on the type of extraction, nor on the location of the fiber bundles in the leaf. Based on these observations, the Young’s modulus was calculated in the deformation range between 0.8% and 1.8% as in Betené (2021).

Mean values and standard deviations of tensile strength, Young’s modulus, breaking tenacity and elongation at break of the fibers of each zone, of the two extraction methods, raw and bleached were calculated and presented in Figure 7b, c, and d and Table 2.

Although the fiber bundles were extracted from leaves selected from the same plantation and tested under the same conditions, there was a dispersion of mechanical properties in each sample area regardless of the
extraction method. It should be kept in mind that pineapple fibers are bio-based materials, so the properties can be influenced by many factors during their growth (Pickering et al., 2007). Other parameters affecting the dispersion of mechanical properties are fiber diameters (Duval et al., 2011), morphology, porosity and chemical composition (Nishiyama and Okano, 1998; Bodros and Baley, 2008; Fidelis et al., 2013). In addition to these parameters, dispersion can also be related to the method used to determine the cross-sectional area of the fibers is questionable due to the porosity and defects (kinks, bands) that exist in a natural fiber.

The tensile strength (for ACR only) and the Young’s modulus of the fibers increase from the bottom to the top. These properties are higher for the fibers extracted by the manual method compared to those of the fibers extracted by retting. On the other hand, the tensile strengths (except ACR), tenacities and the elongations at break of the fibers of the central

Table 3. Identification parameters of FTIR spectra of raw pineapple comosus leaf fibers as a function of sampling area and extraction type.

| Wavenumber (cm \(^{-1}\)) | Bond type | Assignment | References |
|---------------------------|-----------|------------|------------|
| 3100–3600                 | O–H and –OH stretching vibration | Hemicellulose and cellulose | Rosa et al. (2010) |
| 2920                      | C–H and CH\(_2\) stretching | Cellulose | Rosa et al. (2010) |
| 1733                      | C–O stretching of carboxylic or acetyl and uronic ester | Hemicellulose, pectin or wax | Mittal and Chaudhary (2018) |
| 1638                      | H–O–H bending or aromatic C–C stretching | Absorbed water or lignin | Wang et al. (2020) |
| 1592                      | aromatic C–C stretching | Fatty acid and lignin | Wang et al. (2020) |
| 1539                      | aromatic C–C stretching | Lignin | Shahbazi et al. (2017) |
| 1433                      | C–O stretching and C–H or O–H bending | Hemicellulose | Betene et al. (2020) |
| 1405                      | CH\(_2\) bending and –OCH\(_3\) stretching | Lignin | Alawar et al. (2009) |
| 1369                      | C–H bending | Cellulose, hemicellulose, or lignin | Wang et al. (2020) |
| 1316                      | O–H in-plane bending or CH\(_2\) wagging | Cellulose or hemicellulose | Rosa et al. (2010) |
| 1245                      | C–O stretching of acetyl group | Hemicellulose and lignin | Wang et al. (2020) |
| 1170                      | asymmetric bridge C-O-C vibration | Hemicellulose or cellulose | Shahbazi et al. (2017) |
| 1032                      | -OH and C–O stretching vibration | Cellulose and lignin | Mouhoubi et al. (2012) |
| 893                       | C–H and O–H symmetrical stretching | Lignin and cellulose | Betene et al. (2020) |
| 833                       | aromatic C–C symmetrical stretching | Lignin | Parida et al. (2015) |

Figure 9. Curves of the water absorption kinetics of bundles of raw and bleached pineapple fibers from the three sampling areas, extracted a) manually and b) by retting. c) Illustration of the exponential fit of the experimental data.
part are the highest, while those of the upper parts are the lowest. However, the differences observed between the lower zones and the central zones are not very different.

The same remarks are valid for the bleached fibers. However, the bleached fiber samples possess lower tenacity, strength and elongation at break than the raw fiber samples, the smallest values were obtained for ACRW, precisely for ACRW(Top). Consequently, the bleached fibers became stiffer. This increase in stiffness is related to fiber shrinkage during the bleaching process Pandit et al. (2020). This characteristic could be improved by enzymatic treatments or coupling agents Dumont et al., 2017). This could be explained by the changes in fineness (Table 2). In general, cleaning of non-cellulosic materials is accompanied by fiber degradation Alawar et al., 2009, and finer fibers generally have lower breaking strength Wang et al., 2020.

Given the many sources of variability in the properties of natural fibers, comparisons with results reported by other researchers for the same types of fibers are not easy. However, the values obtained in this study are in the middle of the range of values reported by Asim et al. (2015), and some discrepancies were observed between these values and those reported by Budarsaporn et al. (2019). These deviations are associated with the natural character of the pineapple fibers and possibly the dispersions caused by the test methods and statistical analysis. Overall, it is noted that the strengths of the fibers studied are within the range of values reported for banana fibers Sango et al., 2018, cotton Doraiswamy et al., 1993 and hemp Béauk et al., 2008. These resistances are higher than those reported for the fibers of esparto Dallel, 2012 and Agave americana Mshahi et al., 2006.

3.5. Fourier transform infrared (FTIR) spectra

Figure 8 shows the raw and bleached fiber spectra from the different sampling areas. The raw ACG (Figure 8a) and ACR (Figure 8b) spectra show peaks at 3338, 2920, 1732, 1630, 1405, 1245, 1030 and 890 cm⁻¹, already observed by Betene et al. (2020) for pineapple fibers. In general, these peaks signal the presence of hemicelluloses, cellulose, pectin and lignin in natural fibers Oujaji and Shanks, 2005, Rosa et al., 2010, Jeong et al., 2016, Tiwari and Sarangi, 2022). The identification parameters of the different fibers are presented in Table 3.

FTIR spectra can help differentiate pineapple fiber bundles from different areas. It is observed that the peak at 3100–3600 cm⁻¹ is wider for the lower fiber bundles and becomes narrower as one approaches the upper part of the pineapple leaf, thus showing a lower amount of –OH groups in the fiber bundles of the upper part of pineapple leaves. In general, the decrease in hydrogen bonds leads to a reduction in the number of active sites for efficient fiber/matrix adhesion Jawaid et al., 2020, Mittal and Chaudhary, 2018). Similarly, the decrease in the peak at 1245 cm⁻¹ from the top to the bottom of the leaf indicates a lower amount of lignin in the lower fiber bundles than in the upper fiber bundles of the pineapple leaf.

A comparison of the area-by-area spectra reveals that the absorption intensities at 3100–3600 cm⁻¹, 1733 cm⁻¹, 1635/1638 cm⁻¹ and 1245 cm⁻¹ and at 890 cm⁻¹ respectively attributed to the hydroxyl groups of α-cellulose Wang et al., 2020) and hemicellulose, the carboxyl acids of the xylan of hemicellulose, the adsorbed water molecules, the carboxyl acids of cellulose and the aromatic groups of lignin are clearly weaker. In general, these functional elements which make up the major elements of plant fibers disappear thermally below 100 °C in pineapple fibers Jawaid et al., 2020. This confirms the results in Table 1, which show a higher volatile content for ACG compared to ACR. Thus, it is relevant to consider that retting is effective in partially removing functional groups associated with volatile materials compared to the scraping method.

FTIR spectra are also a response commonly exploited by researchers Wang et al., 2020) to characterize structural changes induced by chemical treatments in plant fibers. The attenuation of the bands at 3338 cm⁻¹, 1635/1638 cm⁻¹ and 1245 cm⁻¹, indicates that NaClO is effective in partially removing hemicelluloses, pectin and lignin in pineapple fibers. The disappearance of the peak at 1592 cm⁻¹ shows the success of the bleaching process with NaClO in the ACR and ACG which is responsible for the stretching of the C=C double bonds of the fatty acids. The smoothing of the peak at 1733 cm⁻¹ is clearly greater for ACRWs compared to ACGWs, showing that NaClO treatment easily removes carboxylic acids from the hemicellulose xylan in ACRs better than in ACGs. The same effects were observed for caustic soda treatment by Mittal and Chaudhary (2018) for pineapple fibers. According to Jawaid et al. (2020), the elimination of amorphous components e.g. xylan) improves the surface condition of the fibers for a better interface.

3.6. Water absorption kinetics

Figure 9a and b show the representative water uptake rate versus time curves for the pineapple leaf fibers under study. These fibers are hydrophilic and can absorb water up to 268% of their dry mass. This hydrophilic character could be explained by the porous structure of pineapple fibers Budarsaporn et al., 2019 and more generally by the presence of hemicelluloses due to their numerous ramifications Baley, 2004, Betene et al., 2020). Water absorption for these pineapple fibers by immersion for increasing times is considered to be rapid and saturation is reached after 24 h of immersion. Such water absorption kinetics have also been observed for other natural fibers Baley et al., 2012, Hamza et al., 2013). This ability to absorb water most often leads to a decrease in the mechanical strength of the fiber, as well as a decrease in stiffness and the appearance of cracks in the composite Asim et al., 2015, Samouh et al.,

### Table 4. Water absorption rate of raw and bleached pineapple fiber bundles extracted by two methods from three sampling areas.

| Type of fiber | Wₐ (%) | Model parameters C₀ a k R² |
|--------------|--------|---------------------------|
| ACG(Top) | 196.77 ± 9.8 | 207.19 ± 5.9 | 197.34 ± 6.8 | 7.74 ± 0.6 | 0.96 |
| ACG(Middle) | 246.41 ± 18.3 | 250.68 ± 7.6 | 232.40 ± 8.8 | 7.73 ± 0.7 | 0.97 |
| ACG(Bottom) | 219.50 ± 8.7 | 234.58 ± 6.6 | 225.91 ± 7.0 | 8.65 ± 0.7 | 0.97 |
| ACR(Top) | 209.89 ± 10.3 | 208.68 ± 6.3 | 198.39 ± 7.8 | 5.97 ± 0.5 | 0.97 |
| ACR(Middle) | 267.72 ± 22.6 | 269.91 ± 7.3 | 247.30 ± 9.7 | 6.85 ± 0.6 | 0.96 |
| ACR(Bottom) | 228.46 ± 16.7 | 239.07 ± 7.2 | 222.30 ± 7.7 | 8.67 ± 0.8 | 0.96 |
| ACGW(Top) | 186.93 ± 10.7 | 191.43 ± 7.1 | 172.88 ± 8.2 | 7.89 ± 0.9 | 0.95 |
| ACGW(Middle) | 241.03 ± 1.1 | 244.87 ± 5.2 | 230.92 ± 6.9 | 6.54 ± 0.4 | 0.98 |
| ACGW(Bottom) | 232.43 ± 14.8 | 251.83 ± 5.5 | 208.95 ± 7.1 | 6.71 ± 0.5 | 0.97 |
| ACRW(Top) | 202.84 ± 6.9 | 209.16 ± 4.5 | 198.42 ± 5.7 | 6.85 ± 0.4 | 0.98 |
| ACRW(Middle) | 254.60 ± 13.9 | 273.82 ± 6.5 | 272.79 ± 6.6 | 9.04 ± 0.5 | 0.98 |
| ACRW(Bottom) | 224.21 ± 18.5 | 231.78 ± 6.5 | 216.94 ± 7.5 | 7.79 ± 0.7 | 0.97 |
This remark agrees well with the results of the tensile tests shown in Table 2. Figure 9a and b also show that fiber bundles extracted by retting are slightly more absorbent than those extracted by retting. This slight difference can be associated with the diameters of the fibers, it is possible that the retting caused a slight swelling of the fibers.

Figure 9c shows that the experimental curves of water absorption kinetics fit well with the simple exponential function in Eq. (3) well, where $a$, $k$ and $C_0$ are constant parameters. This model is commonly used to describe the evolution of the hydroscopic properties of natural fibers (Ndapeu et al., 2016). The parameters of model (Eq. 3) were determined (Table 4) using a Levenberg-Marquardt iteration algorithm integrated in OriginPro software.

$$W_{ab}(t) = C_0 - a \exp\left(-\sqrt{t} / k\right)$$

As shown in Table 4, the average values of water absorption rate at fiber saturation of the central parts are the highest, while those of the upper parts are the lowest. We can thus distinguish three very interesting classes which provide information on the water absorption capacity of the fibers: the first class includes all the samples of fibers from the central part with high values between 240% and 270%; the second, associated with the intermediate values ranging from 210% to 240%, is made up of all the fiber samples from the lower part; the last class consists of all the samples from the upper part with an absorption rate between 185% and 210%. Relative to the values of water absorption rates of natural fibers reported in the literature, the three classes of this study are comparable to jute, rush and hibiscus fibers (Ramakrishna and Sundararajan, 2005; Toledo et al., 2005) for the second class and sisal fibers (Ramakrishna and Sundararajan, 2005) for the last.

Comparisons of the water absorption kinetics of the two types of fibers according to the extraction method show the great impregnation capacity of ACR compared to ACG. For bleached fibers (ACRW and ACGW), this functionality decreases slightly, demonstrating that hemicelluloses were partially removed from the fiber surface during NaClO bleaching. It is well known that surface treatments reduce the hydrophilic function of natural fibers (Elenga et al., 2009; Baley et al., 2012).

A comparative study with pineapple fibers from the literature (Mittal and Chaudhary, 2018) revealed that all the different types of pineapple fiber bundles studied in this work had water absorption rates (experimental and theoretical) higher. This discrepancy could be explained by the difference in fiber scales, and to a lesser extent by the climatic conditions and the maturity of the plant (Baley, 2002).

4. Conclusions

The exploitation of pineapple fibers in the development of textiles and composites requires a better understanding of their properties. In this study, thermogravimetric, density, fineness, tensile, water absorption and scanning electron microscopy tests were carried out on fiber bundles extracted by retting and manually and divided into three zones. In addition, the effect of bleaching with sodium hypochlorite (NaClO) was examined on the evolution of the properties of the different fiber bundles. The conclusions drawn from this study indicate that the pineapple fiber bundles extracted by retting give thermal stability with an increase of about 2.4% for the upper part, 8% for the central part and 6% for the lower part compared to the bundles obtained manually. Compared to the pineapple fiber bundles extracted by scraping, the fiber bundles produced by retting have fewer impurities on the surface of the fibers, thus improving their density and fineness in each sampling area. Some differences were observed on the mechanical properties in the different areas. The fiber bundles of the central parts show superior mechanical properties than those of the upper and lower parts, and manual extraction seems to produce stronger and tougher fibers than extraction by retting. Plots of water absorption rate as a function of time showed that fiber bundles manually extracted absorb less water than bundles extracted by retting following simple exponential kinetics, and the highest levels of the rate of saturated water absorptions were obtained for the fiber bundles in the middle part. Bleaching is effective in cleaning non-cellulosic materials from the surface of fibers, improving their density, fineness and water absorption kinetics. But it causes the fibers to shrink in the transverse direction and reduce their strength, tenacity and elongation.

The results obtained in this study show that the manual extraction process can be used to extract fibers from pineapple leaves, as it allows to produce fibers that are more tenacious and more resistant compared to retting process. As the specific properties examined in this study indicate minor variations, it therefore seems relevant, for an extraction method, to consider the whole pineapple leaf to extract fibers for the development of bio-based textiles and composites.

Declarations

Author contribution statement

Achille Désiré Omgbeta Betené: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Fabien Ebanda Betené, Ateba Atangana: Conceived and designed the experiments; Analyzed and interpreted the data.

Felitia Enyegue Ngali, Roger Moukene: Performed the experiments; Contributed reagents, materials, analysis tools or data.

Pierre Marcel Anniceet Noah, Benoit Ndiwe: Analyzed and interpreted the data.

Anny Geraldo Soppie: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

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The authors declare no conflict of interest.

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