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Research article

Impact of extraction methods on the properties of Carica papaya pseudostem fibers from Cameroon used as reinforcement in biocomposites

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ABSTRACT

Carica papaya pseudostems are widely available as biomass waste in Cameroon. These agricultural wastes can be effectively used as natural fibers in the manufacture of biocomposites. In this study, Carica papaya fibers were extracted from papaya pseudostems by retting with water and an alkaline sodium hydroxide (NaOH) solution at different concentrations (2.5 %, 5 %, and 7.5 %). An experimental campaign is being conducted on the physical, chemical, thermal, mechanical, and morphological characteristics of Carica papaya fibers. Fourier transform infrared spectroscopy (FTIR) of Carica papaya fibers extracted by water retting and those extracted with NaOH indicates that the cellulose, hemicellulose, and lignin functional groups are present in the fibers and are dissolved considerably as the percentage of NaOH increases. Scanning electron microscopy (SEM) in the longitudinal plane gives a visual representation of the rough and irregular surfaces without the presence of impurities on the chemically extracted fibers compared to that extracted with water. In contrast to the decrease in diameter, the measured density of Carica papaya pseudostem fibers increased with NaOH concentration (0.633 – 1.522 g cm^{-3}), all of which remained light fibers. Water absorption decreased from 159.36 % to 141.28 % with increasing NaOH, and relative humidity dropped to 6.41 %. The thermal stability of Carica papaya fibers extracted at concentrations of 2.5 % NaOH (215 °C), 5 % NaOH (200 °C), and 7.5 % NaOH (175 °C) showed a clear decrease compared to those extracted by water retting (220 °C). Fibers extracted at 2.5 % NaOH achieved a tensile strength of 287.55 ± 56 MPa, a tensile modulus of 8.271a± 1.62, and 3.505 ± 1.01 % elongation. The tensile properties of these fibers showed great variability, and an influence of diameter was observed, indicating the need to study the influence of technique and NaOH mass concentration. The results show that Carica papaya fiber extracted at a concentration of 2.5 % will be the most suitable for biocomposite applications.

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1. Introduction

Today, natural plant fibers are increasingly studied throughout the world to reduce domestic consumption and the carbon impact produced by synthetic materials [1,2]. They are widely used in many fields, including textiles, automobiles, construction, insulation, and furniture [1,3–5]. Cameroon's biomass is rich in a variety of natural resources, some of which are unexploited and others little exploited [1,6]. It is worth continuing to study these available natural resources, most of which are wild or invasive plants that are particularly rich in fibers with interesting properties [6–8].

Papaya (*Carica papaya*) mainly belongs to the *Caricaceae* family, which comprises around 31 species [9]. It is a fast-growing herbaceous plant that can reach 10 m in height. The *Carica papaya* plant is mainly cultivated for its fruits, for their high nutritional and medicinal value [9]. Cameroon ranks 64th in the world in papaya production, with an annual output of 44 tons, which generates a large mass of *Carica papaya* (CP) trunk at the end of production. Once this has reached the end of its life cycle, it is cut down and disposed of in the wild, increasing the volume of agricultural waste in the environment.

With the current evolution of our society towards a more sustainable and eco-responsible economy, which favors the development of locally available renewable resources, Pawpaw pseudo-trunks, which are agricultural by-products rich in natural plant fibers, can be valorized and used on an industrial scale, both nationally and internationally. At the same time, these fibers could also diversify the world's production of natural fibers, which is dominated by three fibers (flax, hemp, and coir) that alone account for over 80 % of production, excluding cotton, used in textiles and many other applications [10].

Recent studies [11–13] have demonstrated the effectiveness of *Carica papaya* fibers in composite reinforcement. However, it has been previously shown that fiber from the pseudo-trunk of the Indian *Carica papaya* has a cellulose content of 38 %, 11.8 % hemicellulose, a crystallinity index of 56.34 %, and a density of 943 kg m^{-3} [9]. With a thermal stabilization temperature of between 120 °C and 220 °C and a fiber constituent degradation temperature of 322 °C. Its microstructure does not have a good surface finish. According to the authors, it will be important to improve the surface finish by fiber treatments for use in composites [9]. Similar observations have been reported by Refs. [14,15]. The results obtained on Brazilian papaya trunk fibers give a density of 1.12 g cm^{-3}, a cellulose content of 62 %, a lignin content of 15 %, a thermal stability temperature of 254 °C, and a degradation temperature of 346 °C, with a less rigorous surface finish than that obtained on Indian papaya fibers [16]. In view of the above, it is clear that the properties of natural fibers, such as morphology, biochemical composition, and internal structure of the natural fiber, such as microfibril angle and crystallinity index (CrI), vary with the maturity of climatic conditions, geographical location, and extraction conditions, as well as the fiber preparation stages, in particular the extraction and drying processes [17–19]. This justifies further studies on fibers derived from *Carica papaya* pseudo-trunks, and in particular extraction techniques, to further enrich the data available in the literature for use in composites or as textile fibers.

Lignocellulose fibers are a natural composite in which cellulose fibrils are embedded in a lignin matrix. In addition to cellulose, lignin, hemicellulose, pectin, and wax are other important components of lignocellulose fibers [20]. Traditionally, lignocellulosic and bast fibers were extracted by the dew retting process. Dew retting is also an alternative and sustainable method of extracting lignocellulosic fibers. However, due to the longer lead times and water pollution problems associated with water retting [1,21] researchers have developed other fiber extraction methods, including decortication, post-decortication cleaning, enzymatic retting, and chemical retting [11]. Chemical extraction using alkaline NaOH treatment is a widely used technique [9,22]. It can be effective when the elements (cellulose, hemicellulose, lignin, and pectin) contained in the fiber dissolve without risk of fiber damage [20,23]. Hemicellulose is highly hydrophilic and dissolves easily under alkaline conditions. Lignin, being entirely amorphous and hydrophobic, is soluble under warm alkaline conditions [18,22]. Several factors influence the extraction process (extraction time and NaOH concentration percentage) of the fibers. Some authors [19,22,24,25] point out that beyond 5 % NaOH during the extraction process, the mechanical characteristics in traction (tenacity) decrease by degrading the crystalline structure of the fiber [1,19].

In this study, we investigate the extraction of fibers from its pseudo-trunk of Cameroonian origin. Physical tests (density, yield, and absorption), chemical and microstructural tests (FTIR, ATG, and SEM), and mechanical tensile tests will be carried out. Two extraction techniques will be used in this study (water and chemical techniques).

2. Materials and methods

2.1. Raw materials

Carica papaya pseudostem stems were collected at the ENSET site of the University of Douala (Wouri Department, Littoral Region, Cameroon). Sections 50 ± 5 mm long were cut using a hand saw and then washed with water. Sodium hydroxide (NaOH) concentrated to 99 % and acetic acid were supplied by Laboratoire PYCNOLAB (Douala, Cameroon). Fibers are extracted by water retting (Water. Ext), water supplied by CAMWATER, and chemically with NaOH at concentrations of 2.5 % NaOH, 5 % NaOH, and 7.5 % NaOH chosen according to the work of Soppie et al. [23] and Obame et al. [19].

2.2. Extraction in stagnant water

Extraction took place in two phases, the first of which involved immersing (Fig. 1a) the papaya sections in a tank containing water at room temperature. After fourteen (14) days [9], decomposition of the sections due to microbial degradation [15] was observed (Fig. 1b). The resulting mattress was rinsed with ambient water (Fig. 1c). The second phase involved immersing the resulting mat

(Fig. 1c), after cutting it finely to facilitate extraction, in ambient water (Fig. 1d) for a further two weeks (Fig. 1d). After two weeks, the fibers could be extracted before being rinsed and sun-dried for 24 h to remove moisture [21].

2.3. Chemical extraction in sodium hydroxide (NaOH)

Sections of papaya pseudostem were cut into small pieces to facilitate extraction (Fig. 2a). As shown in Fig. 2b, the cut pseudostems were immersed in 2.5 %, 5 %, and 7.5 % NaOH solutions by weight and boiled in a 1:20 (w/v) solid/liquid bath for 2 h (Fig. 2b). The fibers were successively washed several times with demineralized water before being washed in a solution containing 1 % acetic acid to neutralize the effects of NaOH, followed by a thorough rinse with running water to remove any additional alkaline chemicals adhering to the surface until a pH of 7 was obtained [19,23]. The fibers extracted by the different techniques were dried at 28 °C in the sun for 72 h to reduce the water content. These were fibers extracted by water retting and fibers extracted chemically at concentrations of 2.5 %, 5 %, and 7.5 % NaOH by weight, respectively, as shown in Fig. 3a for water extraction, Fig. 3b–. c, and Fig. 3d for 2.5 % NaOH, 5 % NaOH, and 7.5 % NaOH, respectively. The water-extracted fibers are whitish in color, unlike the chemically extracted fibers, which are yellowish. It is also observed that this coloration increases with the percentage of NaOH.

To determine the extraction yield of the fibers, the masses before extraction (Me) and the dried masses of the fibers after drying for 72 h (Ms) were recorded. These masses were measured using a digital balance. Fig. 3a shows fibers extracted with water; Fig. 3b with 2.5 % NaOH; Fig. 3c with 5 % NaOH; and Fig. 3d with 7.5 % NaOH. The extraction yield (Rdt) was calculated using equation (1) [7].

$$\text{Rdt}\ (\%) = \frac{M_e}{M_s} * 100 \tag{1}$$

2.4. Physical characterization of Carica papaya fibers

2.4.1. Determination of fiber diameter

Apparent diameter was measured according to the method reported in the literature [21] at three points spaced 5 mm apart on a 40 mm-long fiber using a micrometer. Twenty-five (25) samples were tested for each extraction technique, making a total of one hundred (100) samples.

2.4.2. Determination of water absorption degree

Five constant mass packages per extraction process are tested, for a total of 20 samples. Samples are dried at 105 °C for 3 h to render them anhydrous. The anhydrous mass (Mi) of the samples was measured on an electronic balance before being immersed in distilled water (30 °C) for 24 h [8]. Once removed from the water, a dry cotton cloth is used to remove surface water before being reweighed and the final mass (M_f) recorded. The method used was gravimetric [26–28]. The absorption degree (W) is calculated by relation 2 [8].

$$W\ (\%) = \left(\frac{M_f}{M_i} - 1\right) * 100 \tag{2}$$

With W (%): water absorption degree; M_f: mass of water-saturated fibers (g); M_i: mass of fibers in anhydrous state (g).

2.4.3. Determination of relative humidity recovery degree

The relative humidity recovery degree was determined using a gravimetric technique [6]. Five fiber bundles of constant mass per extraction process were used, for a total of twenty bundles, and dried at 105 °C in an oven to make them anhydrous. Once anhydrous, the dry mass is recorded (Mi), then the bundles were conditioned in an adiabatic chamber saturated with sodium chloride at a relative humidity of 75 % [29]. The fiber bundles remained in the chamber for 48 h. After this time, the samples were removed from the chamber and weighed to record the wet mass (M_f), and the relative humidity à was calculated using relation 2.

Fig. 1. Stagnant water extraction; (a): Submerged papaya trunk; (b): Decomposing trunks; (c): Fiber mat; (d): Mat submerged in water.

Fig. 2. Extraction with NaOH; (a): Pieces of *carica* papaya pseudostem; (b): Pieces of pseudostem immersed in NaOH; (c): Extraction of fibers; (d): Rinsing of extracted fibers.

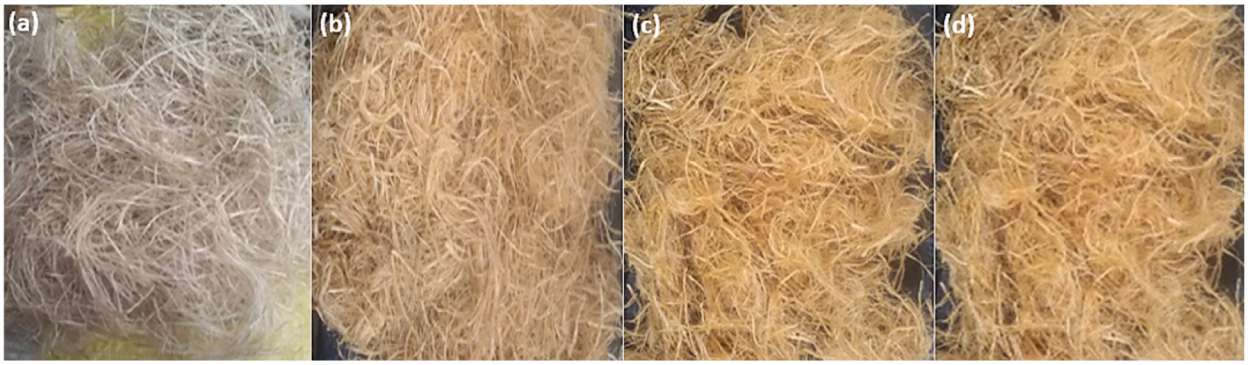

Fig. 3. Presentation of extracted fibers; (a): extraction with retting; (b): 2.5%NaOH; (c): 5%NaOH; (d): 7.5%NaOH.

2.4.4. Determination of fiber density

The test was carried out in the PYCNOLAB Laboratory, with samples made anhydrous in a ventilated oven in accordance with ISO 3344 [30]. The mass of the anhydrous sample was measured (m_0) on a digital scale. The pycnometer is then filled with demineralized and degassed water up to meniscus zero, and stabilized at reference temperature before weighing (m_1). The sample is introduced into the pycnometer, and the water level is adjusted using a propipette after the sample has been immersed for 24 h. The sample is stabilized at the reference temperature before being weighed (m_2). Equation (3) [2] is used to calculate the absolute density. Five samples were used per extraction process for a total of twenty samples.

$$\rho_{abs} = \frac{m_0 * \rho_{eau}}{m_0 + m_1 - m_2} \tag{3}$$

m_0: the mass of the sample in the anhydrous state; m_1: the mass of the Gay-Lussac pycnometer filled with water; m_2: the mass of the Gay-Lussac pycnometer containing the sample and water after 24 h immersion in the water bath.

2.5. Chemical and microstructural characterization of Carica papaya fibers

2.5.1. Fourier transform infrared spectrometry (FTIR)

The functional chemical groups were identified using a Shimadzu IRAffinity^{-1} CE instrument equipped with an ATR module and controlled by Opus/Mentor software. Samples of a few milligrams of powder (0.3 μm) were scanned over a spectral region from 4000 to 400 cm^{-1} with an accumulation of 24 scans and a resolution of 4 cm^{-1}. The FTIR spectra were recorded in absorbance mode.

2.5.2. Thermogravimetric analysis (TGA)

Thermal degradation of *Carica* papaya pseudostem fibers was carried out in a TGA Q50-0836 thermal analyzer with an open platinum crucible. Approximately 20 mg of extracted fiber powder, ground to a size of 0.3 μm, was 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}).

2.5.3. Determination of microstructure

The longitudinal surface of the extracted fibers was observed using a Hitachi s-3500N scanning electron microscope (SEM). Prior to

observation, the fibers were sputtered with a thin layer of gold/palladium.

2.5.4. Mechanical characterization of Carica papaya fibers by tensile testing

Tensile tests were carried out in accordance with the protocol of standard NF T25-501-2 [1,12,21]. Fiber samples were produced with a length L_0 of twenty (20) mm and subjected to a load of 5 kN at a loading speed of 5 mm min^{-1} at room temperature. Twenty-five (25) samples were tested on each extraction process for a total of one hundred samples for all processes. Stress at break (σ_r) was calculated from equation (4) and strain at break ε_r: by equation (5) [6]. Young's modulus is the linear part of the stress-strain curve.

$$\sigma = \frac{F}{S} \tag{4}$$

$$\varepsilon = \frac{\Delta l}{l} \tag{5}$$

With F: Force (N); S: Cross-sectional area (mm^2); σ: Breaking stress (MPa); ε: Strain; Δl: Comparator displacement; L_0: Working length.

2.6. Statistical analysis

A multivariate study of variance (MANOVA) was carried out to determine the influence of the extraction method on the physical and mechanical responses of *Carica* papaya fibers. A confidence level of 95 % was set to test the hypothesis that the extraction method did not influence the observed properties. IBM SPSS 27 software was used.

3. Results and discussions

3.1. Physical results of Carica papaya fibers

3.1.1. Extraction yield

Fig. 3 shows that fiber yield (Fig. 4) and texture are influenced by the parameters used to obtain the fibers. Yield is lower for fibers extracted by retting with stagnant water (17.98 %) and whitish in color (Fig. 3a); it changes with increasing NaOH (19.92 %, 24.39 %, and 28.63 %), respectively, for fibers extracted with 2.5 %, 5 %, and 7.5 % NaOH with a change in color (Fig. 3b, c, and 3d). The change in color is attributed to the effectiveness of NaOH in eliminating non-cellulosic elements and thus favoring fiber recovery [31]. Similar results were obtained for banana skirt, kenaf, and flax fibers [7,31,32]. The low amount of fiber obtained with water extraction may be due to the additional extraction process, the first phase of which initially produces a honeycomb. Unlike chemical extraction, which is carried out directly. This was reported by Hamidon et al. [33] on kenaf fibers.

3.1.2. Fiber diameter

The curve in Fig. 5 shows the results for fiber diameter. A decrease of 302.02 ± 4 μm for fibers extracted by water retting and 196.33 ± 4 μm - 221.94 ± 6 μm - 219.31 ± 4 μm respectively for fibers extracted with 2.5 %, 5 %, and 7.5 % NaOH. This indicates that the NaOH extraction process creates a narrowing of diameters due to the removal of non-cellulosic material. This was reported by Cesarino et al. [34] and corroborated by Betené et al. [30], Mono et al. [6]. This narrowing of fiber diameters is likely to make them denser, less hydrophilic due to their porous nature resulting from the dissolution of the hemicelluloses contained in the fiber, and is in agreement with the results of the degree of moisture absorption and recovery, less resistant, and less tenacious [6,19,30,34]. The

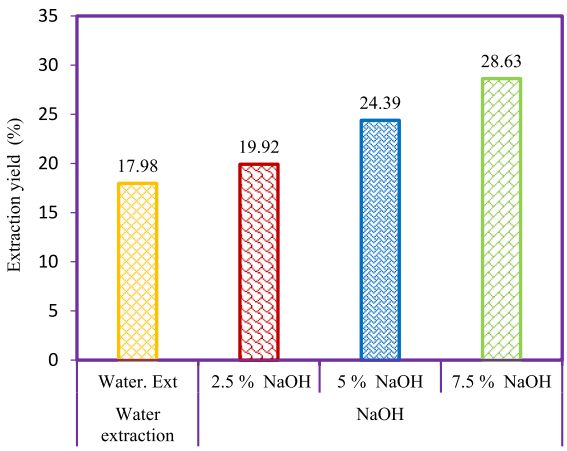

Fig. 4. Extraction yield distribution curve.

diameters are smaller than those of plantain pseudostem fibers (117–247 μm) [21] for water-extracted fibers and are of the same order for NaOH-extracted fibers. They are smaller than those of cylindrical loofah fibers from Cameroon (233–419 μm) [6]. Dispersions around diameters due to their natural character and the various defects obtained during their extraction process are generally observed in the literature on plant fibers [6,19,23]. These dispersions of fiber diameter values could lead to heterogeneous properties in the production of composites with polymers or in the textile yarns that can be manufactured.

3.1.3. Water absorption degree

The evolution of the distribution (Fig. 6) presents the results of the water absorption degree. Like other plant fibers, Carica papaya pseudostem fiber has a hydrophilic character, which can be explained by its porous structure [35]. This may explain why it absorbs 159.36 % water in dry mass gain for fibers extracted by water retting and [141.28–153.64 %] for chemically extracted fibers. An overall decrease in the water absorption degree was observed with increasing NaOH concentration. Fibers extracted by water retting are more hydrophilic than those extracted chemically, which can be justified by their higher hemicellulose content [36,37]. Similar observations have been made of pineapple, jute, hemp, sisal, and loofah fibers [6,30,36,38]. Capacity to absorb water most often leads to a reduction in the mechanical strength and rigidity of the fibers, and can lead to the appearance of cracks in the production of biocomposites [30,39,40]. The results obtained are of the same order of magnitude as those for palm nut mesocarp fiber (OPMF) [41], plantain pseudostem fiber [2,21], and *Triumfetta pentendra* fibers [42]. Increasing the percentage of NaOH considerably reduces the rate of water uptake. This shows that NaOH is effective in dissolving the amorphous hemicellulose contained in papaya fibers [19], and that the use of treated fibers could limit shrinkage, swelling, and cracking phenomena in the production of fiber biocomposites with possible applications in wetlands.

3.1.4. Determination of relative humidity recovery degree

The moisture absorption degree (Fig. 7) follows the same profile as that of water absorption. It decreases with increasing NaOH percentage, in particular from 9.14 % (fibers extracted by water retting) and from 9.09 % to 6.41 % (fibers extracted with NaOH) in gain of their dry masses over a period of 48 h. This water content confirms the hydrophilic behavior of these fibers and is in agreement with the results obtained on water absorption, confirming the effectiveness of NaOH extraction in dissolving the amorphous hemicellulose contained in these fibers. The results obtained are similar to those for *Triumfetta cordifolia* (TC) fibers (8.1–12 %) [23], and lower than those for sisal (13.6 %), jute (12 %), and flax (12 %) fibers used in the production of textile yarns [23,40,43], and *luffa cylindrica* fibers from Cameroon (9–15 %) [6], *sida rhombifolia fibers* (11.73–13.28 %) [1], plantain pseudostem fibers (11.6–13 %) [21], and plantain leaf fibers (11.73–13.28 %) [2]. The emphasis is therefore on strengthening biocomposites for use in dry and humid environments.

3.1.5. Density

The density (Fig. 8) is 0.633 ± 0.06 g cm^{-3} for fibers extracted by still water retting, 1.14 ± 0.03 g cm^{-3}, 1.396 ± 0.06 g cm^{-3}, and 1.522 ± 0.08 g cm^{-3} for fibers extracted with 2.5 %, 5 %, and 7.5 % NaOH, respectively. The values increase with the percentage of NaOH. The increase in density is due to the reduction in non-cellulosic elements, the narrowing of diameters, and the porous nature of the fibers [19,23]. Several studies have shown fiber densification with increasing NaOH percentage [23,23,42,44]. Although the density of the water-extracted fiber in this study is close to that obtained in the work of Kempe et al. [45], the results show that the location where the Carica papaya fiber is planted influences these characteristics. The low density of Carica papaya fiber from Cameroon makes it possible to produce composite materials of lower weight than those made from synthetic fibers (the density of synthetic fibers varies between 1.8 and 2.54 g cm^{-3}). The results obtained are lower than those for cotton (1.5–1.6 g cm^{-3}), sisal

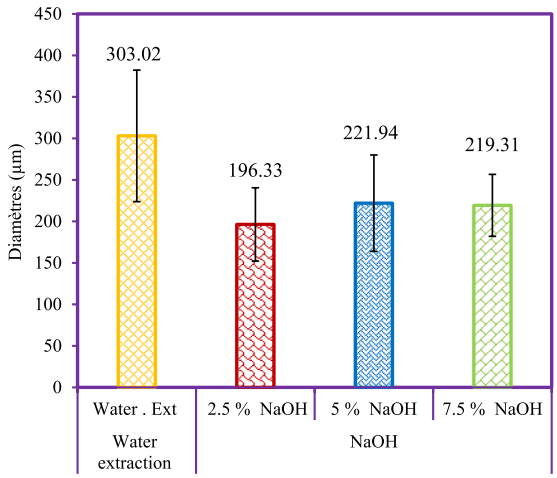

Fig. 5. Diameter distribution curve.

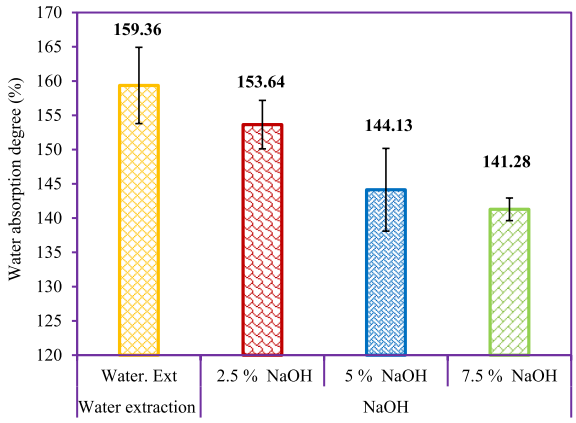

Fig. 6. Water absorption degree distribution curve.

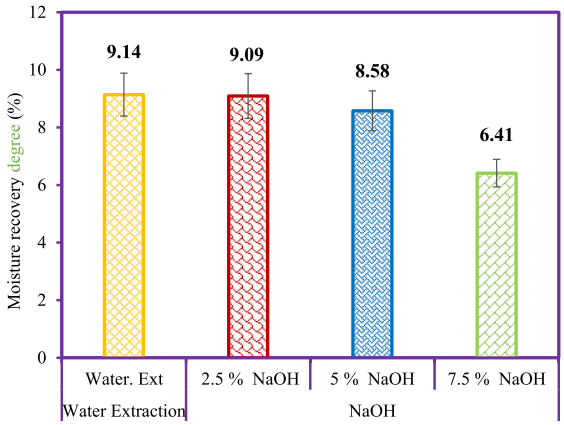

Fig. 7. Moisture recovery distribution curve.

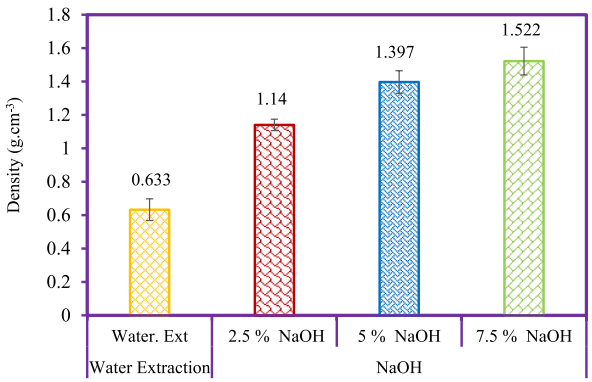

Fig. 8. Density distribution curve.

(1.33–1.5 g cm^{-3}), and flax (1.52 g cm^{-3}) fibers [6,23,46] for fibers extracted with water, 2.5 %, and 5 % NaOH, and of the same order of magnitude for fibers extracted with 7.5 % NaOH.

Table 1 compares the physical characteristics of a number of plant fibers. The diameter of the extracted fibers is comparable to that of coir, jute and sisal fibers, which are marketed and exploited for biocomposite reinforcement. These extracted fibers are denser than coir, jute and papaya fibers when extracted with NaOH, and in the same order of magnitude when extracted by water retting. Carica papaya pseudostem fibers are less hydrophilic than jute, sisal, TC and Comus pineapple fibers [19,47], so applications have been

directed towards biocomposite reinforcement and yarn production. Carica papaya fibers can be oriented in this direction, making them an alternative material for composite reinforcement.

3.2. Chemical and microstructural results for Carica papaya fibers

3.2.1. Fourier transform infrared spectrometry (FTIR)

Fig. 9 shows the FTIR spectrum of papaya fibers extracted with water and NaOH. The different group identification parameters are shown in Table 2. A similarity is observed in the spectra of Fig. 8, with a broad band at 3342 cm^{-1}, 3339 cm^{-1}, 3324 cm^{-1}, and 3334 cm^{-1} for fibers extracted with water, 2.5 % NaOH, 5 % NaOH, and 7.5 % NaOH, respectively, attributed to the hydroxyl group -OH and the O-H hydrogen bonds of hemicellulose and cellulose [44]. A decrease in these peaks is observed as the percentage of NaOH increases. This leads to a decrease in the hydrophilicity of these fibers and corroborates the results obtained on water absorption [23].

The peak near 3342-3334 cm^{-1} that was observed in the specimens is impacted by the presence of water in the fiber and is characteristic of the elongation of -OH from hemicellulose and O-H from cellulose [19,50]. The bands between 2920 and 2900 cm^{-1} show stretching of C-H and CH_2 [20]. However, the characteristic peak at 1731 cm^{-1}, which corresponds to the symmetrical extension of the C=O of hemicelluloses and pectins [20], is highlighted by the decrease in signal. These results confirm that hemicellulose is also eliminated from papaya trunk fiber surfaces only after alkaline pre-treatment with 2.5 % NaOH. A peak was also observed at 1625 - 1593 cm^{-1} corresponding to the shearing of the -OH bond in free water [19,44]. The position of this peak remained unchanged for both water-extracted and NaOH-extracted fibers. However, the intensity of the peaks decreased after the fibers were treated. As an additional indication of the presence of lignins in our fibers, we observed a peak at 1507-1506 cm^{-1} which corresponds to a symmetrical C=C stretching of the lignin [18,20]. We also observed a peak at 1317 cm^{-1} corresponding to the stretching vibrations of CH and C-O in the ester (cellulose and hemicellulose) [20]. The band between 1029 and 1027 cm^{-1} corresponds to the vibrations of the OH and C=O bonds in cellulose [19]. We also observed stretching of the CH and OH bonds in cellulose at the peak located at 898- 897 cm^{-1} [1, 18]. Similar observations have been made on the extraction of fibers using alkaline treatments [19,23].

3.2.2. Thermogravimetric analysis (TGA)

The thermal behavior of treated and untreated papaya fibers analyzed by TGA and DTG is presented in Fig. 10. The thermograms are similar to those of other plant fibers observed in the literature [20,23]. A mass loss of 7.76 %, 9.07 %, 8.69 %, and 13.27 % respectively for fibers extracted by water retting, 2.5 % NaOH, 5 % NaOH, and 7.5 % NaOH is observed around 30 °C and 150 °C. This is attributed to moisture loss and decomposition of the low molar masses of the fibers [22,30]. After this dehydration phase, thermal stability is observed around 150 °C and 225 °C, indicating that these fibers are suitable for use in the production of biocomposites with polymers. The onset of thermal degradation between 250 °C and 352 °C is attributed to depolymerization of hemicellulose and cleavage of glycosidic bonds in amorphous cellulose, cellulose, pectin, and lignin. In general, the thermal depolymerization of glycosidic bonds in cellulose occurs between 220 °C and 310 °C and that of α-cellulose between 310 °C and 390 °C, while that of lignin occurs slowly over the entire decomposition temperature range (210 °C–450 °C) [22,30,51]. From 352 °C to 600 °C corresponds to the thermal decomposition of the different elements to form volatile products [19], which are 13.8 %, 18.24 %, 24.59, and 16.22 %, respectively, for fibers extracted with water, 2.5 % NaOH, 5 % NaOH, 7.5 % NaOH.

The analysis in Table 3 compares the extracted fibers with other fibers in the literature. The extraction technique and the percentage of NaOH have an influence on thermal stability. The thermal stabilization temperature is higher for fibers extracted with 5 % NaOH (200 °C–332.66 °C) than for fibers extracted with 2.5 % NaOH (215 °C–351.72 °C), 7.5 % NaOH (175 °C–322.9 °C), and close to that extracted with water (220 °C–352.95 °C). This result may be associated with the partial dissolution of non-cellulosic matter and other impurities contained in the fibers during the retting extraction. In general, the removal of non-cellulosic matter is accompanied by an improvement in the thermal degradation properties of natural fibers [23,52].

The residue content of the extracted fibers ranged from 13.8 % to 24.59 % by mass. Chemical extraction with 5 % NaOH showed a higher residue content of 24.59 %, indicating that it contains higher quantities of inorganic substances. Thermal stability is considered to be the degradation threshold for the production of composite materials with thermoset and thermoplastic polymers or during

Table 1
Comparison of some plant fiber characteristics.

fibers	Diameters (μm)	Density ($g.cm^{-3}$)	Water absorption degree (%)	References
Coconut fiber	110–530	0.67 à 1	100 ± 19,5	[1]
Jute	40–350	1.1–1.5	281	[48]
Sisal	80–300	0.75–1.07	230 ± 16	[49]
Flax	39.70–620	1.5	136 ± 25	[19,47]
TC	72.49	1.47	342.3	[44]
Ananas comosus	56.1–142.6	1.14–1.45	186.9–267.7	
Agave	171–403	1.25–1.5	–	[1]
Papaya carica	309.1–356.9	0.84–0.97	–	[9,45]
Coconut	100–450	1.18	94–180	[50]
Water. Ext	**303.02**	**0.63**	**159.36**	**Present study**
2.5%NaOH	**196.33**	**1.140**	**153.64**	
5%NaOH	**221.94**	**1.397**	**144.13**	
7.5%NaOH	**219.31**	**1.522**	**141.28**	

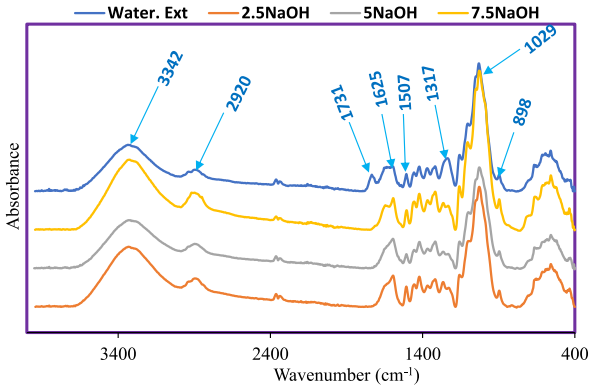

Fig. 9. FTIR spectrum of papaya fibers.

Table 2
Identification of papaya fiber parameters.

Wavenumber (cm^{-1})				Designations
Water. Ext	2.5%NaOH	5%NaOH	7.5%NaOH	
3342	3339	3324	3334	Elongation of -OH from hemicelluloses and O-H from cellulose
2920	2915	2913	2900	C-H and CH2 stretching of cellulose
1731	1731	1731	1731	Symmetrical C=O extension of hemicelluloses and pectins
1625	1593	1593	1593	Shearing of the -OH bond in free water
1507	1507	1506	1506	Symmetrical C=C stretching of lignin
1317	1262	1264	1317	CH and C-O stretching vibration in the ester (cellulose and hemicellulose)
1029	1026	1025	1027	Elongation vibration of OH and C=O bonds in cellulose.
898	898	897	897	Symmetrical stretching of the CH and O-H bonds in cellulose.

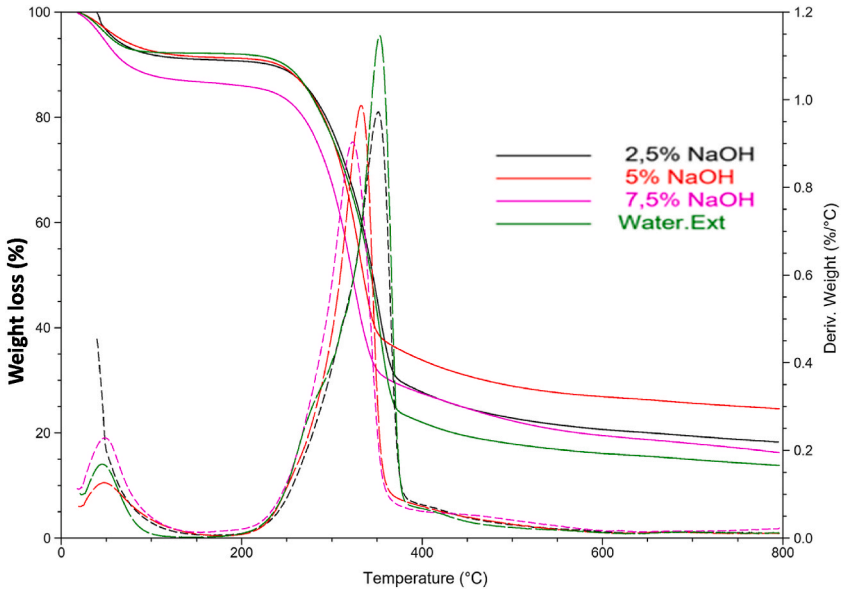

Fig. 10. TGA/DTGA curves for extracted fibers.

surface treatments in hot environments [2,55]. Based on Table 3 and taking into account the thermal stability criterion, two categories of fibers can be distinguished. The first category is associated with thermal stability values between 175 °C and 200 °C, which includes extraction at 5 % NaOH and 7.5 % NaOH. The second category is associated with higher thermal stability values and lies between 215 °C and 220 °C, in which we find extraction with water and 2.5 % NaOH. The thermal stability of the first category is lower than that of *Carica* papaya (220 °C) [9] on the same plant, which is due to the variation in the place of harvest, the conditions for obtaining the fibers and testing, and the maturity of the plant, and is also lower than that of other natural fibers [2,30]. The second category has

Table 3
Comparison of the thermal behavior of extracted fibers with other plant fibers.

Fibers	Dehydration (%)	Degradation temperature		Degradation rate (%)	Residues (%)	References
		start (°C)	end (°C)			
Water. Ext	**7.795**	**220**	**352.95**	**78.41**	**13.8**	**Present study**
2.5%NaOH	**9.073**	**215**	**351.72**	**72.74**	**18.24**	
5%NaOH	**8.695**	**200**	**332.66**	**66.72**	**24.59**	
7.5%NaOH	**13.27**	**175**	**322.9**	**70.47**	**16.22**	
Carica Papaya	–	220	322.1	46.13	36.18	[9]
Kenaf	–	219	284	82	10.5	[22]
Banana	–	263	374	51.5	24.3	[53]
Sisal	–	220	415	72	5.0	[19]
Triumfetta Cordifolia	–	235	420	72	12.69	[44]
Okra	8.4	220	359	60.6	7.6	[54]
Jute	–	230	442	78.9	11.8	[53]

values of the same order of magnitude as Carica papaya [9], kenaf (219 °C) [22], sisal and okra (220 °C) [19,30,54]. But lower than that of *triumfetta cordifolia* (235 °C) [44], Banana (263 °C) [19], jute (230 °C) [53], aloe vera (238 °C), *sida rhombifolia* (250 °C), *Acacia* (280 °C) [56], and pineapple fibers (240–272 °C) [30,55]. The thermal stabilities thus identified provide details on the maximum service temperatures of some of the fibers studied in the literature and in this study for the application of composites with polymers.

3.2.3. SEM analysis of the microstructure

Fig. 11 shows the appearance of the longitudinal surfaces of plant fibers extracted by different methods. Fig. 11a shows that water-extracted papaya fiber has deposits of impurities (non-cellulosic material) that were not removed during the water retting process [44] on the surface of the fibers. By observing the enlargement of a fiber with fewer impurities (Fig. 11b), it is possible to see that the presence of micropores could promote premature fiber breakage [23]. In addition, the presence of micropores may explain the high absorption rate highlighted above. With chemical extraction, the surfaces are cleaner but reveal rough and irregular textures that may be favorable to the fiber/matrix interface depending on the type of polymer matrix. The fiber extracted with a concentration of 2.5 % NaOH is shown in Fig. 11c and its enlargement (Fig. 11d and e) highlights hollow and grooved textures on observation. The severity of the NaOH concentration above 2.5 % reveals the beginnings of micro-cracks that will be detrimental to the mechanical properties (Fig. 11g), the surface finish is improved but the cracks are accentuated. Gradually, rough textures are observed (Fig. 11(f–k). Extracting Carica papaya fibers with NaOH considerably reduces impurities and fiber porosity, as observed by SEM. Similar observations have been made for NaOH treatments [1,19,20].

3.3. Mechanical properties of Carica papaya fibers

The stress-strain curves for each extraction technique are shown in Fig. 12a. The results obtained show that the mechanical characteristics are greatly influenced by the extraction methods used for Carica papaya fibers, as has been observed with other plant fibers [6,19,23]. Firstly, identical behavior was observed for each fiber, associated with linear elastic (brittle) behavior followed by sudden rupture, probably due to the pores observed in Fig. 12b. This behavior has been observed in other fibers studied in the literature [1,6,23,24]. The Young's modulus (Fig. 12b) of water-extracted fibers is 2.142 ± 0.83 GPa with a variation rate of 39.07 % and lower than that of fibers extracted with 2.5 % NaOH is 8.271 ± 1.62 GPa with a variation rate of 19.69 %. That of fibers extracted at 5 % NaOH is 3.543 ± 0.89 GPa with a variation rate of 25.32 %. The strength of fibers extracted with 7.5 % NaOH is 2.595 ± 0.74 GPa with a variation rate of 28.86 %. The mechanical strength at break (Fig. 12c) of the fibers extracted with water (42.14 ± 10.71 MPa) and the fibers chemically extracted with a concentration of NaOH at 2.5 % NaOH (287.55 ± 56.67 MPa), 5 % NaOH (114.94 ± 31.7 MPa) and at 7.5 % NaOH (58.33 ± 16.52 MPa) and the coefficients of variation of 25.41 %, 19.7 %, 27.58 %, and 28.32 %, respectively, are given. Young's modulus and tensile strength decrease with increasing NaOH percentage. This has been observed with NA fibers [19]. The Young's modulus and mechanical strength curves for chemical extraction show linear behavior. The elongation at break (Fig. 12d) of fibers extracted by water retting (1.16 ± 0.69 %), fibers chemically extracted with a NaOH concentration of 2.5 % NaOH (3.505 ± 1.01 %), 5 % NaOH (1.905 ± 0.73 %) and 7.5 % NaOH (2.352 ± 0.69 %) for coefficients of variation of 16.02 %, 29.07 %, 38.52 %, and 29.34 %, respectively, are also given. Non-linear dispersions are observed for chemical extraction. Contrary to the fiber extracted with a concentration of 2.5 %, which has better mechanical properties overall (Fig. 12b–d), the water-retted fibers have lower values. Prolonged retting is detrimental to the fiber because, in addition to destroying non-cellulosic compounds (waxes, pectins, and fats), it also destroys the cellulosic and woody compounds that are responsible for the fiber's greater strength [6,19]. The mechanical properties of the Carica papaya fibers highlighted are close to those of other fibers (Table 4) and make them serious candidates for reinforcement of composite materials (thermoplastic and thermosetting matrices) and make it possible to envisage textile structures such as non-wovens or yarns compared with other natural plant fibers (Table 3). In the end, this study confirmed that fiber properties vary according to the extraction method used and the origin of the plant, as shown in the literature [9].

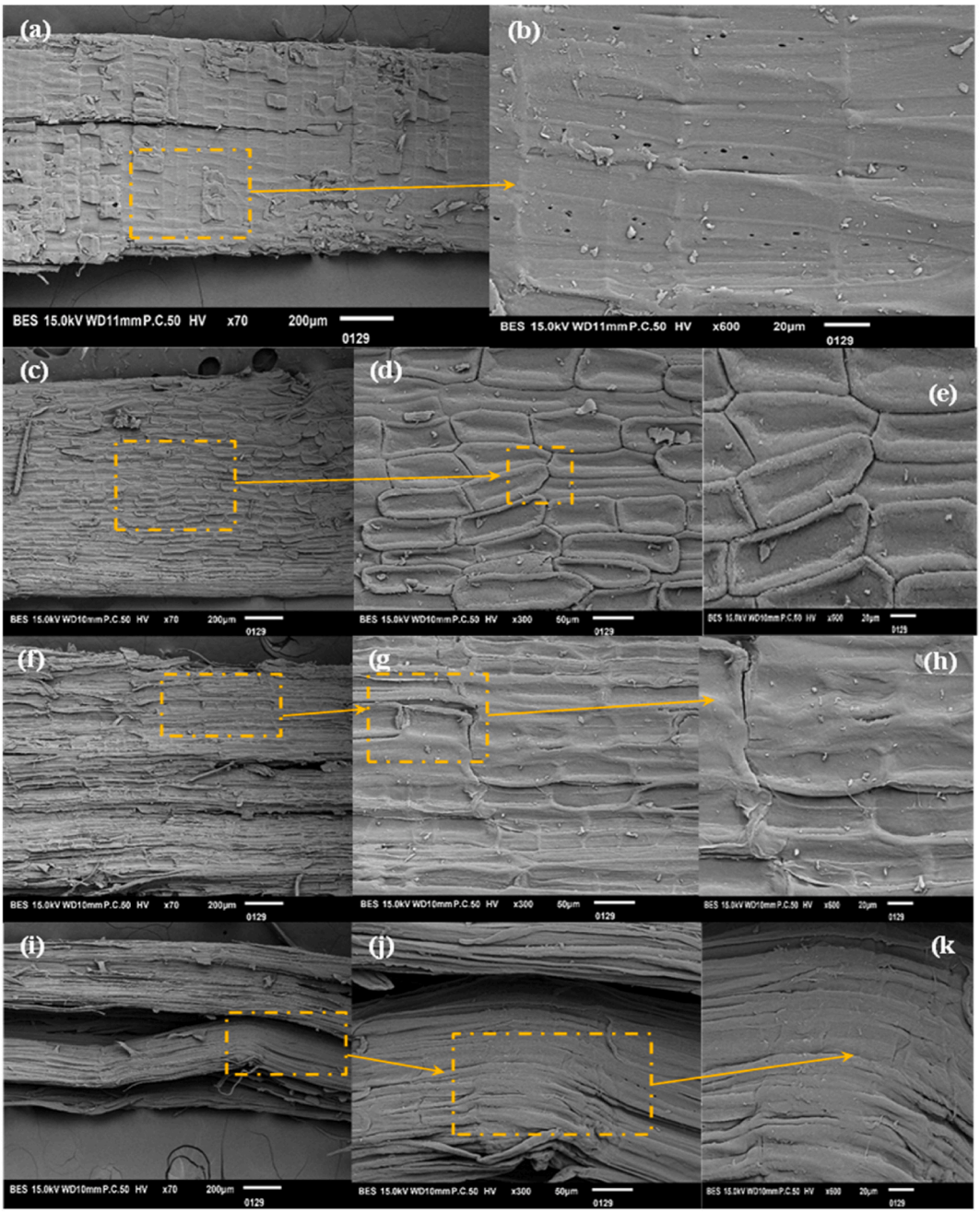

Fig. 11. Microstructure of papaya fiber; (a): Water-extracted fiber; (b): Enlargement at 300 of water-extracted fiber; (c): Extraction at 2.5%NaOH; (d): 2.5 % NaOH enlargement at 300; (e): 2.5%NaOH magnification at 600; (f): Extraction at 5%NaOH; (g): Magnification at 300; (h): Magnification at 600; (i): Extraction at 7.5%NaOH; (j): 7.5%NaOH magnification at 300; (k): 7.5%NaOH magnification at 600.

3.4. Statistical analysis of variance by MANOVA

Table 5 shows the multivariate analysis of variance (MANOVA) for the concentration and extraction methods. Pillai's trace significance level is less than 0.05, rejecting the hypothesis that the extraction method has no effect on the observed properties; there are therefore significant differences due to the extraction method; similar observations have been reported in the literature [7,21,58]. A post hoc test presented in Table 6 was used to analyze the significant differences observed. The Tukey test used for this purpose shows an influence of the NaOH level compared with water extraction on all the physical and mechanical properties. Only the absorption rate showed a non-significant difference between the water method and the chemical extraction with 5 % NaOH. This table also shows that the physical properties (absorption rate) are higher for water extraction than for chemical methods. On the other hand, chemical

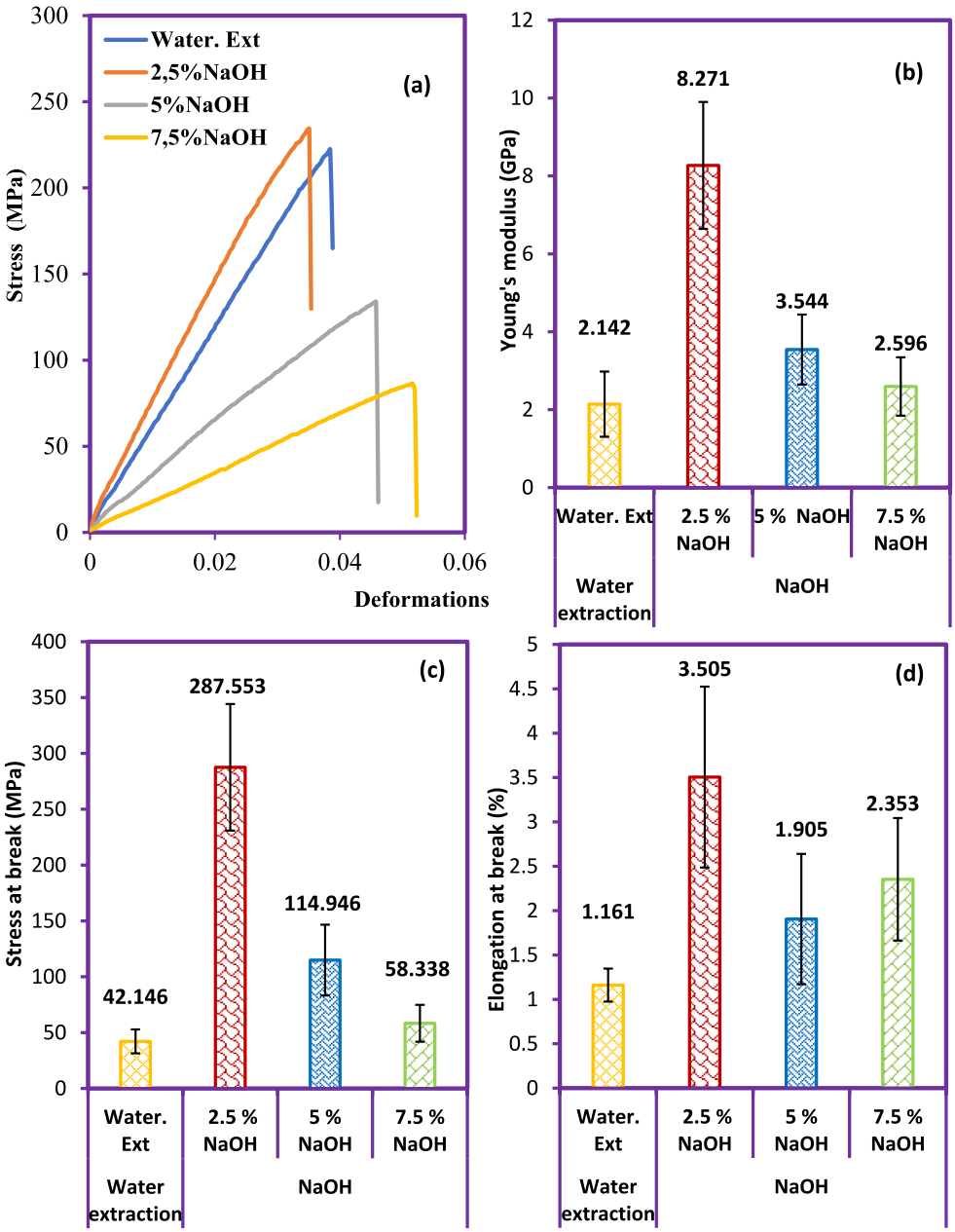

Fig. 12. Tensile test results; (a): Tensile curve; (b): Young's modulus curve; (c): Mechanical strength curve; (d): Elongation curve.

extraction gives higher mechanical properties than water extraction.

4. Conclusions

The exploitation of *Carica* papaya pseudostem fibers remains an alternative solution in the development of biocomposites, and for this reason it requires greater attention given its availability in Cameroon. In this study, the fibers were extracted by retting with water and sodium hydroxide at 2.5 %, 5 %, and 7.5 % NaOH by mass brought to boiling temperature before being characterized. Extraction yield, thermogravimetric analysis, functional grouping, density, diameter, water absorption, relative humidity, and tensile tests were carried out on the fibers extracted by the two techniques. The conclusions drawn in this study give an extraction yield of 17.98 % for extraction by water retting and 28.63 % for extraction with 7.5 % NaOH. Compared with fibers extracted by water retting, fibers produced by sodium hydroxide show fewer impurities on the surface, suggesting that this extraction process removes as many non-cellulosic components as possible. This results in high strength and better adhesion to the polymer matrix, improving their

Table 4
Comparison of the mechanical properties of extracted fibers with other natural fibers.

Fibers	Young's modulus (GPa)	Stress at break (MPa)	Strain (%)	References
Hemp	4.8	270–900	1–3.5	[57]
Flax	50–70	345–1035	2.7–3.3	[57]
Carica papaya	9.89–12.64	101–530	1.2–1.83	[9,13,45]
Jute	15–30	610–780	1 à 1.9	[57]
Triumfetta Cordifolia	12.4–59.8	209.1–916.3	1.56–2.55	[44]
Alfa grass	2.1	63.8	3.1	[1]
Cotton	5.5–12.6	287–597	3–10	[19]
Water. Ext	**2.142 ± 0.83**	**42.14 ± 10**	**1.16 ± 0.69**	**Present study**
2.5%NaOH	**8.271 ± 1.62**	**287.55 ± 56**	**3.505 ± 1.01**	
5%NaOH	**3.543 ± 0.89**	**114.94 ± 31**	**1.905 ± 0.73**	
7.5%NaOH	**2.595 ± 0.74**	**58.33 ± 16**	**2.352 ± 0.69**	

Table 5
Multivariate analysis of variance.

Effect		Value	F	ddl of the hypothesis	Error ddl	Sig.	Partial eta-square
Extraction	Pillai trail	2.491	8.557	12	21	0.001	0.83
	Wilks' Lambda	0.001	24.196	12	13.520	0.001	0.936

Table 6
Post hoc test for extraction methods.

Dependent variable	(I) extraction	(J) extraction	Average difference (I-J)	Standard error	Sig.	95 % Confidence interval	
						Lower terminal	Upper terminal
Water absorption rate	Water extraction	2.5 % NaOH	18.074*	3.712	0.005	6.185	29.963
		5 % NaOH	5.716	3.712	0,460	−6.173	17.605
		7.5 % NaOH	15.224*	3.712	0.015	3.335	27.113
Density	Water extraction	2.5 % NaOH	−0.542*	0.054	0.001	−0.717	−0.368
		5 % NaOH	−0.787*	0,054	0,001	−0.961	−0.612
		7.5 % NaOH	−0.912*	0.054	0.001	−1.087	−0.738
Young's modulus	2.5 % NaOH	Water ext	5.203*	0.786	0.001	2.684	7.722
		5 % NaOH	4.526*	0.786	0.002	2.007	7.045
		7.5 % NaOH	5.056*	0.786	0.001	2.537	7.575
Stress at break	2.5 % NaOH	Water ext	230.353*	35.397	0.001	116.99	343.708
		5 % NaOH	142.2*	35.397	0.016	28.845	255.554
		7.5 % NaOH	213.58*	35.397	0.001	100.228	326.938

densities and reducing their diameters and hydrophilicity. The average diameter, strength, Young's modulus, and strain at break are 196.33 μm, 287.55 ± 56 MPa, 8.27 ± 1.62 GPa, and 3.50 ± 1.01 respectively. The relative humidity is stable (9 %) up to a NaOH concentration of 5 %. The chemical extraction method was found to reduce the hydrophilic character of Carica papaya pseudostem fibers to a water absorption rate of 141.28 %. The tensile properties of *Carica* papaya pseudostem fibers showed a large variability and an influence of the diameter was observed, indicating the need to study the influence of the technique and the mass concentration of NaOH. Thermal analysis concluded that the water-treated fiber and the fiber treated with 2.5 % NaOH exhibited better thermal stability (215–220 °C) and good thermal tolerance (352 °C) for use with polymers. The low density of *Carica* papaya fibers (0.63–1.52 g/cm^3) makes them suitable for lightweight composite materials. In the light of the results presented here, the available pseudo-stem fiber from Carica papaya of Cameroonian origin has the potential to be used as a reinforcing material in the creation of composites, but also in textiles. The authors are currently working on the development of non-woven *Carica* papaya reinforcements obtained by carding for use in the reinforcement of polymer matrices, with a focus on the circular economy.

CRediT authorship contribution statement

Jean Aimé Mono: Supervision, Methodology, Conceptualization. **Sandrine Emvoutou Ndongo:** Writing – review & editing, Writing – original draft, Validation, Conceptualization. **Odette Thérèse Adegono Assiene:** Writing – review & editing, Writing – original draft, Validation, Resources. **Armel Mewoli:** Writing – review & editing, Writing – original draft, Validation. **Rachelle Appolince Nguefack Assona:** Writing – original draft, Validation, Data curation. **Richard Hervé Bitete:** Writing – review & editing, Visualization, Validation, Methodology, Conceptualization. **Giresse Ulrich Defo Tatchum:** Writing – original draft, Methodology, Formal analysis. **Claude Takoumbe:** Methodology, Formal analysis, Data curation, Conceptualization.

Data availability

The data used to support the fundings of this study have not been the subject of any prior study.

Declaration of competing interest

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

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