

Physico-Chemical and Thermal Characterization of Some Lignocellulosic Fibres: *Ananas comosus* (AC), *Neuropeltis acuminatas* (NA) and *Rhectophyllum camerunense* (RC)

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Abstract

This paper focuses on the study of the physical, biochemical, structural, and thermal properties of plant fibres of *Rhectophyllum camerunense* (RC), *Neuropeltis acuminatas* (NA) and *Ananas comosus* (AC) from the equatorial region of Cameroon. The traditional use of these fibres inspired researchers to investigate their properties. This study aims at improving the state of knowledge with a view to diversifying applications. The fibres are extracted by retting. Then, their apparent density was measured following the ASTM D792 standard and their water moisture absorption and moisture content were also evaluated. Their molecular structure was studied by ATR-FTIR spectroscopy. A quantitative analysis of the biochemical composition was performed according to the analytical technique for the pulp and paper industry (TAPPI). A TGA/DSC analysis was also performed. The results reveal that the AC, NA and RC fibres have densities of 1.26 ± 1.06 , 0.846 ± 0.13 and 0.757 ± 0.08 g·cm⁻³ respectively. They are also hydrophilic with a water absorption rate of $188.64 \pm 11.94\%$, $276.16\% \pm 8.07\%$ and $198.17\% \pm 20\%$. They have a moisture content of 12.21%, 10.36% and 9.37%. The studied fibres exhibit functional groups that are related to the presence of hemicellulose, pectin, lignin and cellulose. The cellulose crystallinity index was found to be 67.99%, 46.5% and 59.72% respectively. The fibres under study have the following chemical composition: an extractive content of 3.07%, 14.77% and 8.74%; a pectin content of 4.15%, 7.69% and 3.45%; a hemicellulose content of 4.90%, 15.33% and 7.42%; a cellulose content of 68.11%, 36.08% and

65.15%; a lignin content of 12.01%, 25.15% and 16.2%; and an ash content of 0.27%, 1.53% and 0.47% respectively. The thermal transitions observed on the thermograms correlate with the TAPPI chemical composition. It is observed that these fibres are thermally stable up to temperatures of 200°C, 220°C and 285°C. These results make it possible to envisage uses similar to those of sisal, hemp and flax fibres.

Keywords

Lignocellulosic Fibre, *Rhectophyllum camerunense*, *Neuropeltis acuminatas*, *Ananas comosus*, Biochemical Composition, Cellulose Crystallinity Index, Thermal Behaviour

1. Introduction

The use of plant fibres (cotton, flax, jute, coconut, wood fibres, etc.) as biobased fibrous reinforcements for polymer and ceramic materials is a current industrial option in the perspective of promoting the development of renewable resources. These fibres are used to obtain insulation panels or are added to polymer or plaster matrices for the design of composites. Many studies aim at identifying fibres with properties that are favourable for industrial applications. It is therefore necessary to evaluate their density, porosity, chemical composition, structural characteristics (slenderness, cross-section shape and area, microfibril angle), mechanical strength, thermal stability and sensitivity to the absorption of moist liquids and vapours [1] [2] [3]. In this perspective of finding new fibrous plant resources, research can draw inspiration from the traditional uses of the ecosystem resources of our immediate environment: ropes, pots, fishing nets, toilet gloves, handicraft furniture and mats. Through these uses, these materials seem to resist severe mechanical and thermophysical stresses. This inspired researchers to investigate the properties of AC, NA and RC fibres. Their work has identified some of their physical, chemical, hygroscopic and mechanical characteristics.

A study on the RC fibre (elementary fibres or in the form of bundles) [1] revealed a density of 0.947 g·cm⁻³, a cellulose content of 68.5%, a microfibril angle of 40.11°, a tensile Young's modulus ranging between 2.3 and 17 GPa, an elongation ranging between 10.9% to 53% and a tensile strength comprised between 150 and 1738 MPa. When used as reinforcement in plaster [4], these fibres improve the mechanical, thermal and hygroscopic properties of these materials. They confer to plaster a more ductile fracture behaviour and reduce the kinetics of heat propagation. RC plasters are also more moisture absorbent than unreinforced plaster, which qualifies them for comfort and building application applications.

Previous studies [5] [6] that the NA fibres (elementary fibres or in the form of bundles) have a rectangular cross-section, Young's modulus of 6 GPa, tensile stress of 62 MPa and an elongation at break of 0.01%, cellulose crystallinity in-

dex of 42%. Their microfibril angle is 1.4° , their cellulose content 39.2% and their lignin content 20.4%. The latter is high compared to fibres such as flax, bamboo, kenaf and sisal.

AC fibre has generally been used in the manufacture of yarns for textile fabrics for several decades [7]. AC fibre has been used in the for example in the Piñatex fabric is an alternative to leather in the production of sporting goods and bags. It is reported [7] that AC fibres extracted from Malaysian pineapple leaf species are one of the natural fibres with the highest cellulose content (80%) compared to kenaf (57%), flax (71%), jute (71.5%), sisal (78%), hemp (77%). Moreover, its density of $1.6 \text{ g}\cdot\text{cm}^{-3}$ is similar to other natural fibres e.g., cotton, jute, flax, hemp, sisal, ramie. While it's Young's modulus (83 GPa) is higher or of the same order of magnitude as that of than that of cotton (12.6 GPa), jute (80 GPa), flax (70 GPa), sisal (6 GPa), and its tensile strength (1627 MPa) is the highest compared to cotton (597 MPa), jute (800 MPa), flax (1500 MPa).

Plaster setting [4] and cross-linking of thermosetting matrices [8] can lead to temperature rises that can degrade the lignocellulosic the fibres. As far as we know, no studies on AC, NA and RC fibres have been carried out to characterize their thermal properties such as thermal stability and thermal transitions. However, it is important to consider these properties to select application for composite materials that will not degrade these reinforcement fibres. This work is intended to complete the knowledge of these fibres by studying their thermal behaviour. For validation or comparison purposes, the evaluation of their properties has also been performed.

2. Experimental

2.1. Fibres Extraction

Figures 1-3 show the main steps in the extraction of harvested plant material and the fibres extracted.

2.1.1. AC Fibres

Pineapple leaves were harvested from adult plants in a field in the locality of

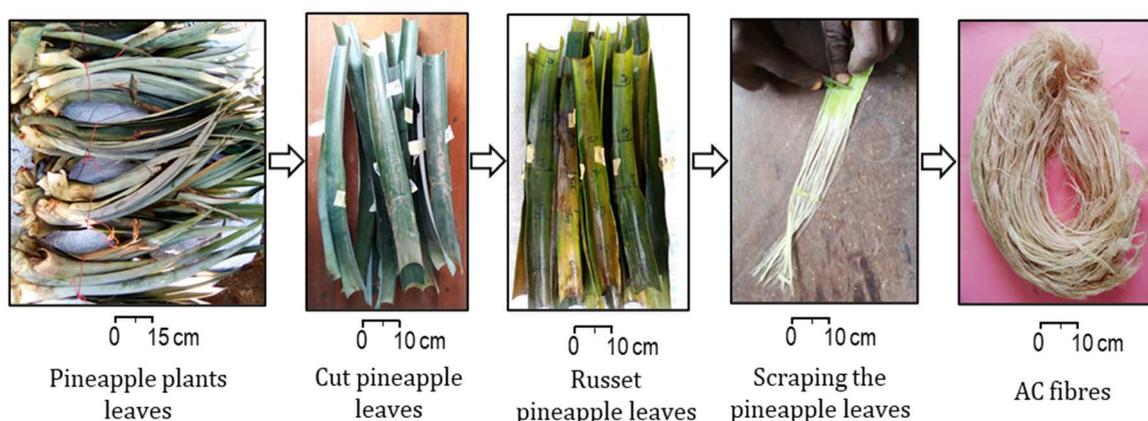


Figure 1. Image showing the main steps of the AC fibre extraction process.

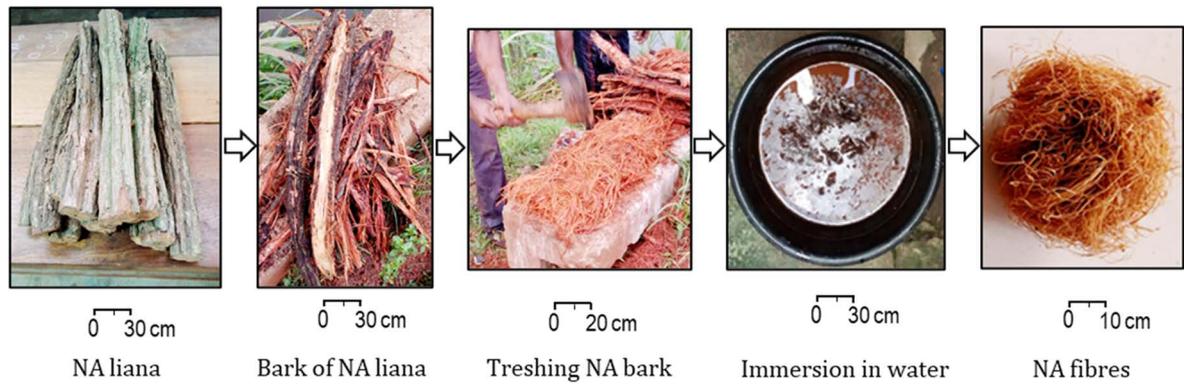


Figure 2. Image showing the main steps of the NA fibre extraction process.

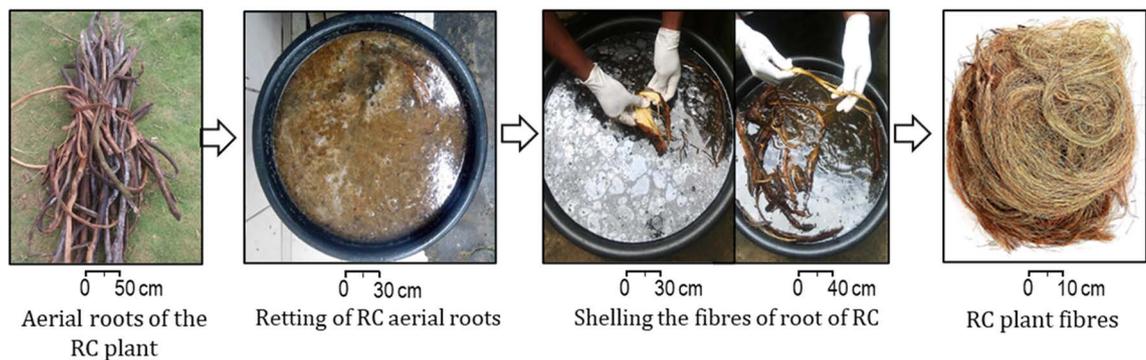


Figure 3. Image showing the main steps RC fibre extraction process.

Njombé-Penja on the Cameroonian coast. The average temperature of this locality is 31°C, with a relative humidity of 73%. The harvested leaves were cut off at their ends and then immersed in stagnant water from the national distribution network for four days. When the leaves reddened, they were subjected to a kind of decortication that left only the fibres remaining. The fibres were rinsed with distilled water and then dried in the shade for 24 hours.

2.1.2. NA Fibres

NA lianas were harvested in the equatorial forest of Mvila-Yévol in South Cameroon. The bark was peeled off on pieces previously cut into 50 cm long pieces, then they were beaten with a 2 kg sledgehammer. The fibres were extracted by retting with stagnant water from the national distribution network.

2.1.3. RC Fibres

The aerial roots of RC were harvested in the undergrowth of the Nkol-Mefembe equatorial forest in Central Cameroon. They were cut into 100 cm long pieces and then laminated. The laminated roots were immersed in water from the national distribution network. After 4 days of immersion, the fibres were extracted, rinsed and dried at room temperature away from the sun for 24 hours.

2.2. Physical Properties

The individual weighing operations were carried out using a 0.1 mg precision bal-

ance. Each test was carried out on 10 samples and the reported result is the average.

2.2.1. Apparent Density

The apparent density ρ_f of AC, NA and RC fibres was calculated by dividing the mass by the volume as shown in Equation (1) based on ASTM D3800-99 (2005) and ASTM D792 (2013). These standards allow the bulk density to be evaluated by the gravimetric method based on Archimedes principle. First, the mass of the fibres m_f previously dried at 50°C for 24 hours was measured. The samples are made up by covering these fibres with paraffin of volume v_p . The volume of the sample v_i was evaluated by immersion in a test tube containing a volume of distilled water v_0 (at 25°C). A metallic volume element v_a ensures the immersion of the sample. The volume v_f of the fibres was determined by Equation (2).

$$\rho_f = \frac{m_f}{v_f} \quad (1)$$

$$v_f = v_i - (v_p + v_a + v_0) \quad (2)$$

2.2.2. Water Absorption Rate (%TA)

The water absorption rate of the studied fibres previously dried at 105°C for 24 hours was evaluated using Equation (3) [9]. Ten samples with a mass m_0 were prepared. These samples were immersed in distilled water for 24 hours at room temperature. The mass m_1 after absorption was measured after cleaning the water from the surface of the samples.

$$\%TA = \frac{m_1 - m_0}{m_0} \times 100 \quad (3)$$

2.2.3. Moisture Content (%H_u)

The moisture content of plant fibres was determined using Equation (4) [9] [10]. Ten samples with the mass m_i were placed at room temperature 25°C ± 2°C with 63% relative humidity for 24 hours. The hydrophilic character favours the absorption of relative humidity from the air of the plant fibres [10]. Finally, the samples were dehumidified in an oven set at 105°C for 24 hours and the final mass m_f of each sample was measured.

$$\%H_u = \frac{m_i - m_f}{m_i} \times 100 \quad (4)$$

2.3. Chemical Composition of Fibres

Quantitative analyses of fibre constituents were carried out using the TAPPI method on dry powdered fibres with a mean particle size of approximately 315 µm. Weighings were carried out on a 0.1 mg precision balance.

2.3.1. Ethanol-Benzene Extraction (%Es)

A cartridge containing an average of 6 g of powdered fibres was introduced into

a Soxhlet. The latter is mounted between a flask charged with ethanol (96%) - benzene (99%); $d = 0.878$) (1:2 v/v) solvent and a refrigerator. This assembly is fixed on a bracket. The separation of the extractives was made by regulated leaching so as to have a siphon every 10 minutes. Dissolution was done for 7 hours. The cartridges were removed from the Soxhlet, freeze-dried at 50°C for 1 hour then cooled in a desiccator. The level of ethanol-benzene extractives (%*Es*) was determined using Equation (5) [11] [12].

$$\%Es = \frac{m_{Es}}{m_d} \times 100 \quad (5)$$

where m_{Es} is the mass of the ethanol-benzene extractives and m_d the mass of previously dried fibre removed.

2.3.2. Water Extraction (%*Ws*)

The dried cartridges (residue 1) of ethanol-benzene were introduced into the Soxhlet. The previous assembly was reconstituted by replacing the solvent with 100 ml of distilled water. The mixture was heated under reflux for 7 hours. The Residue 2 obtained was filtered on a weighed sintered glass crucible No 4, washed with distilled water, incubated at 105°C for 12 hours, then weighed after cooling in a desiccator. The content of hot water extractives (%*Ws*) was determined by Equation (6) [11] [12].

$$\%Ws = 100 \times \frac{m_w}{m_{res1}} (1 - \%Es) \quad (6)$$

with m_w the mass of the water extractables, m_{res1} is the dry mass of residue 1.

The extractive content (%*Ex*) of the fibres is the sum of the hot water and ethanol-benzene extractives.

2.3.3. Pectin Content (%*Ps*)

1.5 g of residue 2 was introduced into an Erlenmeyer flask containing a 2% hydrochloric acid solution. The mixture was heated under reflux with magnetic stirring in a water bath at 80°C for 4 hours. Residue 3 was retained by filtration on a weighed sintered glass crucible No 4, washed with distilled water, dried at 105°C for 12 hours and weighed after cooling in a desiccator. The pectin content (%*Ps*) in the hydrochloric acid extract was determined by Equation (7) [11] [12].

$$\%Ps = 100 \times \frac{m_{Ps}}{m_{res2}} (1 - \%Ex) \quad (7)$$

where m_{Ps} is the mass of pectin extracted, m_{res2} is the dry mass of residue 2.

2.3.4. Lignin Content (%*L*)

The lignin content was carried out according to the Klason method. 500 mg of the dried residue 2 was hydrolysed in 72% sulphuric acid. Lignin being the only insoluble component, it separates from the fibres. Then, the lignin content was determined using Equation (8) [11] [12].

$$\%L = 100 \times \frac{m_L}{m_{res2}} (1 - \%Ex) \quad (8)$$

where m_L is the mass of lignin extracted.

2.3.5. Holocellulose Content (%HC)

1.5 mg of residue 2 was placed in a flask containing 60 ml of distilled water heated to 75°C. Subsequently, 0.2 ml acetic acid and 15% v/v (3 ml) sodium chlorite were added every hour for seven hours. The residue 4 obtained was filtered and then washed with demineralised water and left to stand for two hours before being washed again with ethanol. This residue was dried at 105°C [11]. The holocellulose content was determined using Equation (9) [11] [12].

$$\%HC = 100 \times \frac{m_{HC}}{m_{res2}} (1 - \%Ex) \quad (9)$$

where m_{HC} is the mass of holocellulose extracted.

2.3.6. Cellulose Content (%C)

Cellulose was isolated according to the method of Kurschner and Hoffer [1] [11]. 1 g of holocellulose was introduced into an Erlenmeyer flask containing 50 ml of solvent nitric acid—ethanol (1:4; v/v) prepared from ethanol (96%) and nitric acid (16 N). The assembly was connected to a refrigerant and then heated in a water bath for 1 hour. The solution obtained was filtered using a weighed sintered crucible No. 4, washed with ethanol, then with demineralised water and finally with ether. The cellulose obtained was freeze-dried at 105°C for 12 hours, cooled for 12 hours in a desiccator and weighed. The cellulose content (%C) was determined by Equation (10) [11] [12].

$$\%C = 100 \times \frac{m_C}{m_{HC}} (1 - \%HC) \quad (10)$$

where m_C is the mass of cellulose obtained, m_{HC} is the mass of holocellulose removed.

2.3.7. Hemicellulose Content (%H)

Holocellulose consists mainly of cellulose and hemicellulose [11] [12]. The hemicellulose content was determined using Equation (11).

$$\%H = \%HC - \%C \quad (11)$$

2.3.8. Ash Content (%Cs)

The ground fibres were placed in a porcelain crucible and then calcined at 600°C in a kiln for 6 hours. The ash content was estimated using Equation [11] [12].

$$\%Cs = \frac{m_{Cs}}{m_d} \times 100 \quad (12)$$

where m_{Cs} is the mass of the ashes and m_d is the mass of the sample taken.

2.4. ATR-FTIR Analysis

The main purpose of this analysis was to identify the functional groups of the

studied fibres. The spectra are recorded using a Bruker Alpha-P spectrometer, equipped with an attenuated total reflectance (ATR) module with a diamond crystal and driven by the Opus/Mentor software. The plant fibres were dried at 50 °C for 24 hours and then powdered to the size of 315 µm. A few milligrams of powder from the plant fibres studied were deposited on the diamond crystal of the ATR module. Acquisitions were carried out by scanning over a spectral region ranging from 4000 to 400 cm⁻¹ with a resolution of 4 cm⁻¹.

The crystallinity of cellulose χ in AC, NA and RC fibres was determined by comparing the peak intensities of the FTIR spectra at 1454 cm⁻¹ and 893 cm⁻¹ [13] as shown in Equation (13):

$$\chi = \frac{I_{1454}}{I_{893}} \quad (13)$$

where I_{1454} and I_{893} are the intensities of the FTIR peaks at 1454 cm⁻¹ and 893 cm⁻¹ respectively.

2.5. TGA-DSC Analysis

Thermal analysis was performed on a synchronous STA PT-1000 Linseis thermogravimetric analyzer with 0.5 µg resolution capable of combining TG (Thermogravimetric) and DSC (Differential Scanning Calorimetry) information using the same sample. The device was equipped with a high-resolution 0.1 mg loaded balance, a furnace with a maximum calcination temperature of 1000 °C and a very fast cooling system. The device was connected to a computer and controlled by the Platinum Evaluation v1.0.182 software.

3 to 5 mg of powdered fibres (~315 µm) were introduced into a platinum crucible and then heated in the furnace at a constant heating rate of 10 °C/min in a temperature range from 25 °C to 700 °C under nitrogen flow at a rate of 50 ml/min.

3. Results

3.1. Physical Properties

Several physical property values for fibres in the studies and for some fibres in the literature are given in **Table 1**.

3.1.1. Density of Fibres

The fibre densities (**Table 1**) found for AC, NA et RC fibres are close to those reported in the literature for the same species [1] [4] [14] [15]. The use of AC, NA and RC fibres could significantly contribute to lighten the mass of composites used in the fields of transport, construction, sport and leisure.

3.1.2. Water Absorption Rate of the Fibres

The values of the water absorption rate (**Table 1**) are under study are high (above 100%). These fibres are hydrophilic similarly as sisal, flax, jute and roselle. This hydrophilic character of plant fibres is generally attributed to the presence of hemicelluloses due to their many ramifications [2] [3]. This capacity to absorb water most often leads to a decrease in the mechanical strength of the

Table 1. Density and water absorption rate of AC, NA and RC plant fibres.

Fibre	Density (g·cm ⁻³)	Water absorption rate (%)	Moisture content (%)	References
Sisal	1.5	190 - 250	5 - 10	[7] [14]
Hemp	1.15 - 1.45	-	6.2 - 12	[7] [16]
RC	0.63 - 0.947	-	7.5	[1] [4] [17]
NA	0.918	400	-	[14]
AC	1.526	-	12	[7] [15]
Jute	1.3 - 1.41	281	12 - 13.7	[2] [3] [7] [18]
Flax	1.5	136 ± 25	12	[2] [3] [19]
Cotton	1.5 - 1.6	-	8 - 25	[7] [16]
Roselle	1.41	286.5	10.9	[9]
Kenaf	1.45	-	-	[17]
AC	1.256 ± 0.06	188.64 ± 12	12.21	-
NA	0.843 ± 0.13	276.16 ± 8.07	10.36	-
RC	0.757 ± 0.08	198.17 ± 20.23	9.37	-

fibre, as well as a decrease in stiffness and the appearance of cracks in the composite [3] [7]. This characteristic could be improved by surface treatments and coupling agents [8] [10] [20].

3.1.3. Moisture Content

The initial moisture content of the fibres studied are reported in **Table 1**. It is noted that the moisture content of AC fibres is higher than those of NA and RC. This can be attributed to the relative humidity of the harvesting site, *i.e.* 73% for AC, 61% for RC and 65% for NA. This sensitivity to wet vapours is also attributed to the presence of free hydroxyl groups [7] [15].

3.2. Chemical Composition

The results of the biochemical analysis of the fibres studied and other plant fibres are presented in **Table 2**. The studied fibres content cellulose (%*C*), hemicellulose (%*H*), lignin (%*L*), pectins (%*Ps*), extractives (%*Ex*) and ash (%*Cs*). This corresponds to the description of the chemical composition of plant fibres in the literature [1] [5] [11] [12].

The extractive content is higher for NA (12.77%) than for RC (8.74%) and AC (3.07%). The content of NA extractives is close to the value reported in a recent study [5]. According to the literature [11] [21], if the content of hot water extractives is less than 7%, there is good compatibility with inorganic binders. Hence, the values obtained for AC and RC fibres show that these lignocellulosic fibres can be considered as fairly compatible with gypsum, cement, earth, clay. NA fibres contain almost twice as many pectins as AC and NA fibres. These contents are very close to that of banana fibres [12]. The difference between the

Table 2. Chemical composition by the TAPPI method of plant fibres of AC, NA and RC.

Fibre	Chemical composition (%)							Ref.
	%HC		%L	%Ps	%Ex		%Cs	
	%C	%H			%E _{EB}	%W _s		
Banana	37.5	27.6	15	5.15	0.2	12	-	[12]
<i>Eucalyptus saligna</i>	48.6	12.4	38.4	-	2.4	2.6	0.092	[11]
<i>Cupressus lusitanica</i>	48.4	21.6	34.3	-	3.4	3.2	0.57	[11]
NA	39.24	7.22	20.47	2.07	18.90		2.18	[5]
RC	68.2	-	15.6	-	-		-	[1]
AC	67.12 - 82	9.45 - 18.8	4.4 - 15.4	1.2 - 3	-		0.9 - 2.7	[15]
Jute	61 - 75	13.6 - 20.4	12 - 13	0.2	-		-	[1]
Lin	64.1	16.7	2	1.8	3.9		-	[1] [2]
Sisal	65.8	12	9.9	0.8	1.2		-	[1] [2]
AC	68.11	4.90	12.01	4.15	0.99	2.08	0.27	-
NA	36.08	15.33	25.15	7.69	3.20	9.57	1.53	-
RC	65.15	7.42	16.2	3.45	2.17	3.05	0.42	-

values found in this study and those reported in the literature [5] [15] can be attributed to the method used. The lignin content of AC fibres is lower than that of NA and RC fibres. However, these values are close to those reported in the literature [1] [5] [7]. It is also observed that the lignin content of AC and RC fibres is similar to that of jute and banana fibres [1] [12], while the lignin content of NA fibres is similar to that of wood fibres of *Eucalyptus* species [11]. The lignin content of NA fibres could be recovered and valorized in the production of biofuels. The hemicellulose contents of the fibres investigated in this study are close to those in the literature. For NA fibres the higher hemicellulose content obtained correlates with its water absorption rate and confirms its more hydrophilic character compared to AC and RC fibres. The values obtained show that cellulose is the predominant constituent for all fibres studied, which is consistent with the literature [1]. It can be seen that the cellulose content of RC (65.15%) and AC (68.11%) fibres is almost the same. These values are close to those obtained in previous studies on the same species. It is also noted that these values are similar to other industrial plant fibres such as jute (61% - 75%), flax (64.1%) and sisal (65.8%). According to previous studies [1], the cellulose content of plant fibres commonly used in composites has a cellulose content of 50% - 85%. Based on these results, AC and RC fibres can be considered as fairly compatible with gypsum, for instance. The cellulose content of NA fibres (36.08%) is the lowest among the fibres studied, but remains close to that reported in a recent study [5] and to banana fibres [12]. The low ash content obtained for all types of fibres is in accordance with the data available in the literature.

The amounts of the chemical constituents of AC, NA and RC fibres here are similar to those obtained in previous studies. In addition, cellulose, lignin and hemicelluloses represent at least 75% of the dry mass of the fibres studied. Hence, these fibres constitute promising biobased fibrous reinforcements for composite materials. Owing to their chemical composition close to that of many other types of natural fibres already used in composite applications such as flax and jute fibres, RC and AC fibres are expected to provide a better reinforcing effect compared with NA fibres.

3.3. Molecular Structure of Fibres

The spectra from the ATR-FTIR analyses are shown in **Figure 4**. The parameters of their identification are gathered in **Table 3**. These spectra are similar and

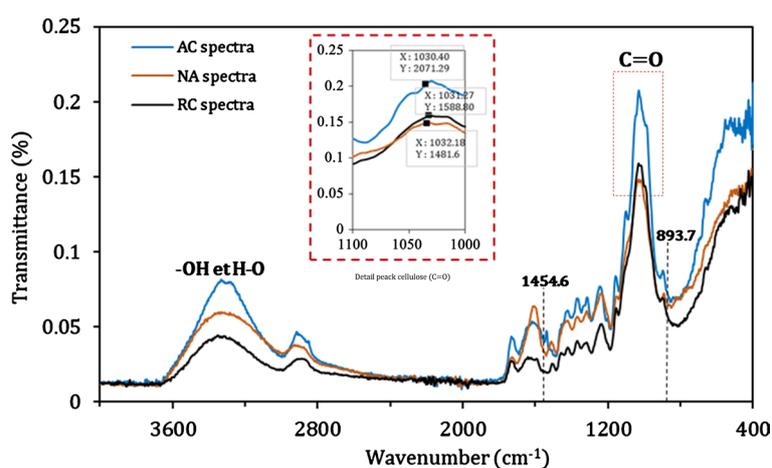


Figure 4. ATR-FTIR spectra of AC, NA and RC fibres.

Table 3. Absorption bands of the functional groups characteristic of AC, NA and RC fibres.

Wavenumber (cm ⁻¹)			Assignments	References
AC	NA	RC		
3333.52	3335.12	3332.40	Elongation of -OH from hemicelluloses and O-H from cellulose	[24] [28] [30]
2916.83	2929.15	2996.52	Stretching of C-H and CH ₂ of cellulose	[13] [22] [23]
1733.03	1729.24	1730.67	Symmetric C=O prolongation of hemicelluloses and pectins	[12] [22] [27] [31]
1632.25	1631.21	1634.50	Shearing of the -OH bond in open water	[22] [23]
1539.25	1508.46	1512.81	Symmetrical stretching of C=C of lignin	[24] [31]
1454.62	1454.62	1454.62	Bending of CH ₂ in cellulose and asymmetric deformation of CH ₃ in cellulose.	[13]
1030.40	1032.18	1031.27	Elongation vibration of the -OH and C=O bonds of cellulose.	[24] [28]
893.74	893.74	893.74	Symmetrical stretching of the C-H and O-H bonds of cellulose.	[13]

show absorption bands similar to lignocellulosic fibres such as hemp and flax [22], alfa [23] [24], rush [24], banana [12] [25]. The IR spectra show many peaks, which are commonly observed in plant fibres [12] [22] [24] [26]. The strongest peak located at 1030.40 cm^{-1} (AC), 1032.18 cm^{-1} (NA) and 1031.27 cm^{-1} (RC) corresponds to the stretching of -OH and C=O bonds present in cellulose.

The polymeric -OH groups of hemicelluloses and the hydrogen bonds (O-H) of the supramolecular structure of cellulose present in the fibres under study are strongly reflected in the band at about 3333.32 , 3352.4 and 3335.12 cm^{-1} for AC, NA and RC, respectively. The characteristic peaks at 2916.83 , 2929.15 and 2896.56 cm^{-1} for AC, NA and RC, respectively, show the stretching of aliphatic and aromatic (C-H, CH₂) groups present in cellulose [13] and lignin [25]. The peaks 1733.03 for AC, 1729.24 for NA and 1730.67 cm^{-1} for RC represent the symmetrical elongation of carboxyl and acetyl (C=O) groups present in hemicelluloses [29], pectin and waxes [22]. The peaks at 1539.25 (AC), 1508.46 (NA) and 1512.81 cm^{-1} (RC) indicate the symmetrical elongation of aromatic groups (C=C) in lignin. The small peak at 1454.63 cm^{-1} indicates not only the asymmetrical deformation of CH₃ in cellulose [13], but also the bending of CH₂ [28] [29]. The mode at 893.73 cm^{-1} fibres indicates the symmetrical stretching of the C-H and O-H bonds in cellulose and lignin [13].

Figure 4 is also used to estimate the crystallinity index of cellulose in plant fibres. The transmittance of peaks at 1454 and 893 cm^{-1} is 0.04908% and 0.07219% for AC, 0.03203% and 0.06888% for NA, and 0.0515% and 0.08624% for RC, respectively. The values obtained are presented in Table 4. The crystallinity in cellulose of AC (67.99%) is higher than that of the other two fibres and is similar to that of *Luffa cylindrica* cellulose (74.71%) [13]. This value of cellulose crystallinity rate could be related to predict high fibre stiffness with high specific Young's modulus compared to RC and NA fibres.

3.4. Thermal Properties

3.4.1. Thermal Characteristics DSC

Figure 5 shows the DSC thermograms of fibre particles ($115\text{ }\mu\text{m}$) of AC, NA and RC.

The DSC curves of the three fibres are similar and show one exothermic and three endothermic peaks. The first convex endothermic peak in each thermogram is attributed to water loss in the fibres [32]. It is observed at 69°C for AC fibres, 72°C for NA fibres and 79°C for RC fibres. This thermal event shows that drying of the fibres prior to analysis did not completely remove water. The three concave exothermic peaks probably correspond to the degradation of hemicelluloses, the

Table 4. Cellulose crystallinity index of AC, NA and RC fibres estimated by FTIR.

Fibre	Degree of crystallinity (%)		
	AC	NA	RC
χ	67.99	46.5	59.72

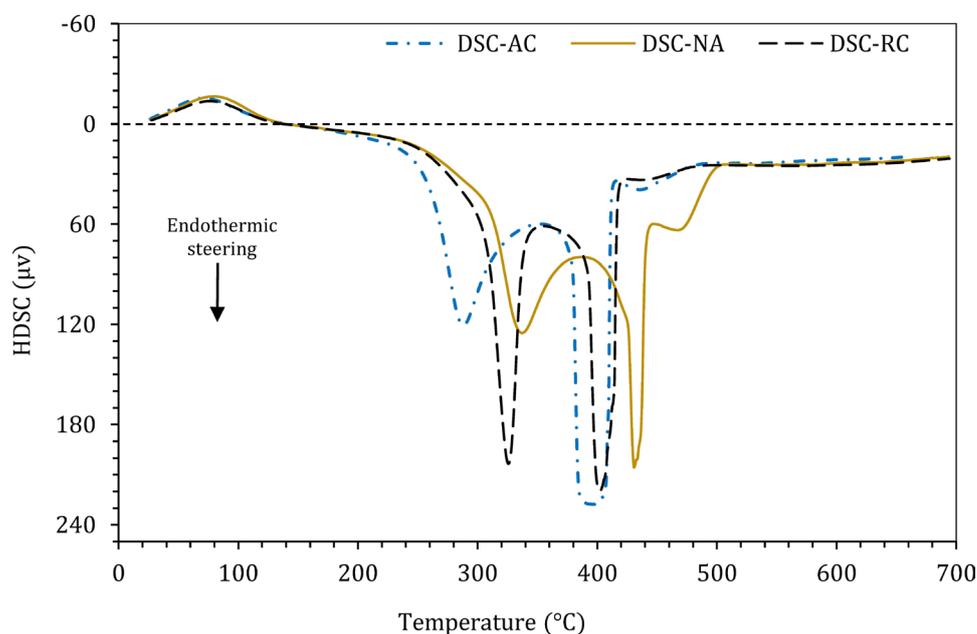


Figure 5. DSC thermogram of AC, NA and RC fibres.

decomposition of α -cellulose and lignin [33] [34].

3.4.2. Thermal Characteristics TGA

The thermal degradation curves (TG) and their derivatives (DTG) of AC, NA and RC plant fibres measured under nitrogen flow are shown in **Figure 6**.

The TGA curves show that the fibres under study break down into four phases. This thermal behaviour is typical of plant fibres to that of plant fibres (flax [9], jute [35], sisal [9], kenaf [35], etc.). The first noticeable change starts at 30 °C and ends at 115 °C, 120 °C and 125 °C for AC, NA and RC fibres respectively, which is due to the evaporation of their structural moisture and volatile extractives. The mass loss recorded in this phase is the same (about 8.2%) for the fibres studied. After dehydration of these fibres, it can be observed that the mass variation is almost constant up to 200 °C for AC, 220 °C for NA and 285 °C for RC. The zone of thermal stability thus identified provides details on the maximum service temperature of the fibres under study [36] [37]. The maximum service temperature of AC fibres is very close to that of jute fibres (205 °C), that of NA fibres is similar to that of roselle (200 °C), okra (220 °C) and close to sisal fibres (222 °C) [9], while that of RC fibres is high compared to kenaf fibres (247 °C) [9]. In the second phase, a greater loss of mass was recorded, *i.e.* 74.41% between 200 °C and 475 °C for AC, 63.53% between 220 °C and 505 °C for NA and 71.42% between 220 °C and 495 °C for RC. This loss of mass is attributed to the thermal decomposition of hemicelluloses and pectin between 150 °C and 250 °C [9] [38], then that of cellulose between 240 °C and 350 °C [33] [38]. The third phase indicates the decomposition of lignin in the studied fibres. A mass loss of 16.27% was observed for AC at 495 °C, 25.04% for NA at 505 °C and 18.11% for RC at 495 °C. In the last phase, the mass variation is very small. This constant is

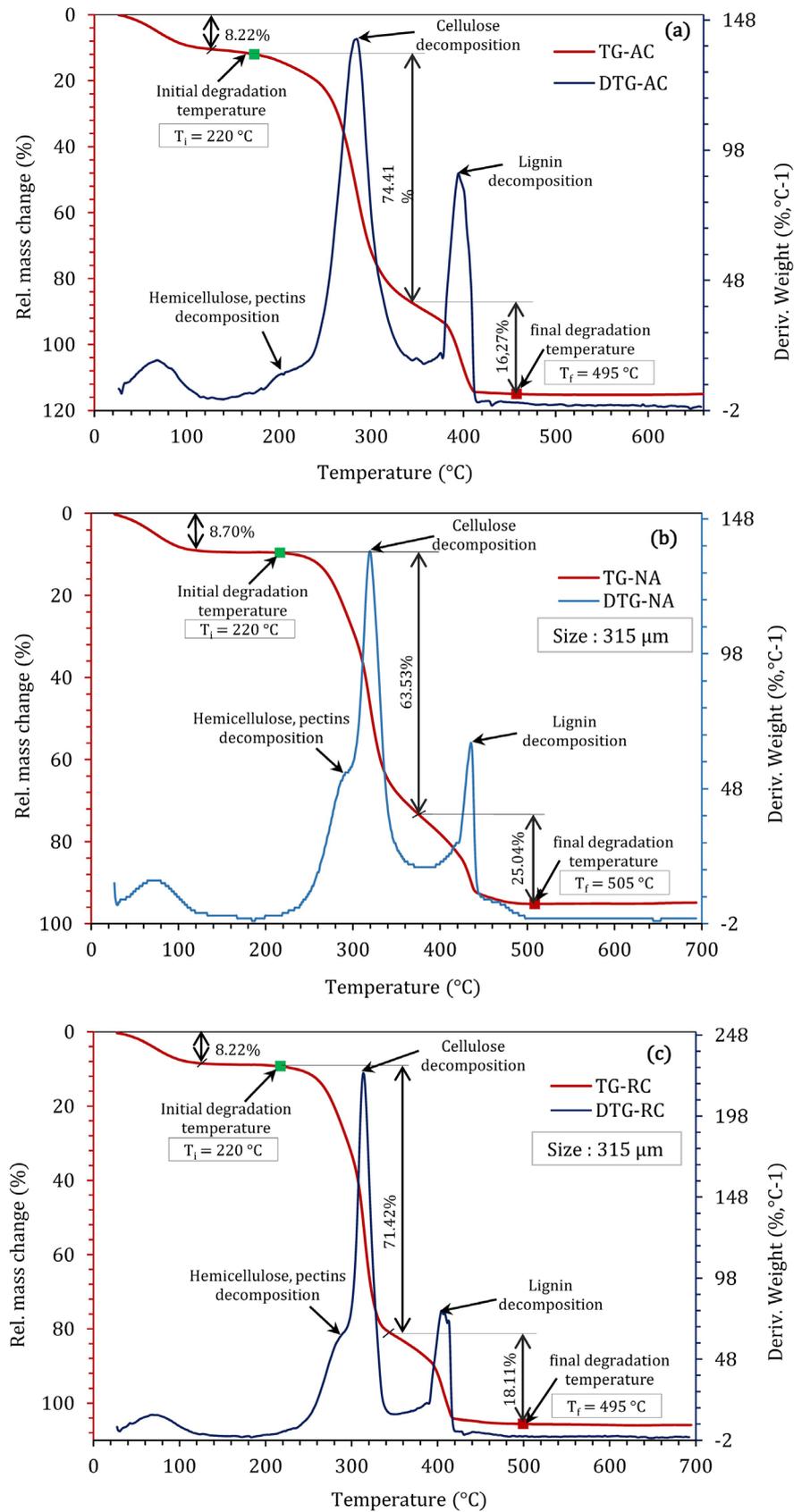


Figure 6. Thermogram TG-DTG of plant fibres: a) AC, b) NA and c) RC.

due to the formation of ashes. The TGA ash content is 1.12%, 2.73% and 2.25% respectively for AC, NA and RC fibres.

The TG curves shown in **Figure 6**, were also used to determine the contents of lignin (%*L*), holocellulose (%*C* + %*H*), ash (%*Cs*) and volatiles of the studied fibres. The values shown in **Table 5** are correlated with the values obtained using the TAPPI method. The content of holocellulose in the fibres of AC and NA is greater than 70%, unlike that of NA.

4. Conclusion

This study examined the potential of certain lignocellulosic fibres as biobased fibrous reinforcements for composite materials used in various fields, including internal building thermal insulation. First, the density, water absorption rate and moisture content of AC, NA and RC fibres were evaluated following the recommendations of ASTM D3800-792 and the method of reported by Nadlène *et al.*, respectively. The results obtained showed that the apparent density of RC fibre is lower than that of AC fibre, which is itself lower than that of NA fibre. The NA fibres were more hydrophilic compared to AC and RC fibres. The water absorption rate of AC fibre was low compared to AC, RC fibres and some fibres widely studied such as sisal, flax, jute and roselle. The chemical composition of each fibre was obtained using TAPPI methods on fibres reduced to a size of 315 μm . The content in polysaccharides (cellulose, hemicellulose) of AC and RC fibres which is about 73% show that these fibres could be interestingly used as reinforcement materials of biosourced materials or as raw materials in the paper industry. On the other hand, the cellulose content (36.08%) of NA fibres was low. This could give the NA fibres a low tensile strength. However, the high lignin content of this fibre could be extracted and valorized in the production of bio-fuel. The spectra recorded by ATR-FTIR analysis indicated the functional. According to the FTIR spectra, the cellulose crystallinity index is 67.99%, 46.5% and 59.72% for AC, NA and RC respectively. Thermal characteristics were studied using DSC and TGA. Thermal degradation of the constituents was observed above 200°C for AC and 220°C for NA and RC fibres. Thermal transition phases observed from DSC and TG/DTG thermograms exhibited three major zones related to the degradation of hemicellulose/pectins, cellulose and lignin. These results clarify the temperature domains for the processing and use of fibres and materials reinforced with them. Further studies could focus on the tensile, XRD,

Table 5. Chemical composition deduced on TGA from plant fibres of AC, NA and RC.

Fibre	Chemical composition (%)			
	% <i>L</i>	% <i>C</i> + % <i>H</i>	% <i>Cs</i>	Volatiles
AC	16.27	74.41	1.12	8.20
NA	25.04	63.53	2.73	8.70
RC	18.11	71.42	2.25	8.22

and SEM characterization of AC, NA, and RC fibres. Investigations could also be done on the characterization of treated fibres, reinforcement of ceramic matrix composites.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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