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Original Article

Physico-Chemical And Thermal Characterisation of Canarium Schweinfurthii Engl (Cs) Shells

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Abstract *- The present work focuses on studying the Physico-chemical and thermal properties of CanariumSchweinfurthiiEngl (CS) core shells. These shells, abandoned after consumption, pose an environmental problem, and their hardness inspires the study of their properties. This study aims to improve the state of knowledge to use them for the reinforcement of composites. Their density and moisture content are investigated. Their molecular structure is studied by ATR-FTIR spectroscopy. Quantitative analysis of the biochemical composition was carried out using the TAPPI method and TGA/DSC analysis. The results revealed that the CS shells had a water content of 6.26%, a holocellulose content of 53.07%, and lignin content of 35.79%. The crystallinity index of the CS shells is 82%. The chemical composition of the studied shells is as follows: an extractives content of 6.887%; a water content of 17.669%; a lignin content of 36.96%, a holocellulose content of 54.48%, a cellulose content of 37.31%, and a hemicellulose content of 19.14%. The thermal transitions observed in the thermograms correlate with the chemical composition of the TAPPI.*

Keywords - *ATR-FTIR, Canarium Schweinfurthii Engl, crystallinity index, density, TAPPI method.*

I. INTRODUCTION

Composite materials are nowadays of great interest to researchers to substitute traditional materials, reduce mass while increasing mechanical performance, and respond to a concern for the preservation of the environment. A distinction is made between fiber-reinforced and particle-reinforced composites [1]. While composites with fibrous reinforcements benefit from high-quality reinforcement, those with particulate reinforcements are also applied in fields such as transport, construction, sport, and leisure [2]. For this purpose, a thorough knowledge of the particle material to be used as reinforcement is essential. Knowledge of CS shells indicates that the Ayele tree (CS producing tree) belongs to the Burseraceae family and grows in a solitary state in humid savannahs. It is widespread in sub-Saharan Africa and Cameroon throughout the humid dense forest, humid and sub-humid savannah zones [3,4].

CanariumSchweinfurthiiEngl is an oleaginous fruit species, in the family Burseraceae, with an ellipsoidal drupe that turns green as it grows and turns purplish at maturity. This species consists of pulp, a hard shell with a kernel inside [5]. The population consumes this product, and the kernels are dumped in nature, causing a pollution problem as they are slow to degrade[40].

Knowledge about the shells of CanariumSchweifurthiiEngl is limited. To date, some authors have produced activated carbons based on CS cores [6,7,8,9] as an absorbent in filters for copper II and other heavy ions. Others have shown that all flow properties have increased except for the mobility coefficient [10,11] and have determined the physical and aerodynamic characteristics of Canariumschweinfurthiiengl cores [12]. Studies have shown high water diffusion stability on a macroscopic scale [13] and that these materials can be used as reinforcement for polyester matrix composites [14].

To our knowledge, many intrinsic characteristics of CanariumschweinfurthiiEngl core shells have not yet been evaluated. This study proposes to complete the knowledge on the intrinsic characteristics of this material by determining the density, moisture content of CS shells, chemical composition, and ATR-FTIR and TGA-DSC analysis.

II. MATERIALS AND METHODS

A. Process of obtaining CS powder

The fruits (figure 1-a) are collected in the West Cameroon region, washed with water, the pits are extracted (figure 1-b) according to a method described in the literature [15]. The pits are crushed, the kernel is separated from the shell (Figure 1-c), and the shells are crushed and sieved (Figure 2 d, e, f).

B. Density and moisture content

The samples were first placed in a Heraeus VT5042 EK oven at 50°C for 24 hours and then weighed individually on a precision balance (0.0001g). The measurements were repeated ten times on each sample, and the result reported was the average.

M_0(g) is obtained and after drying, weighed again, a mass M_f(g) is obtained at 105°C (after 24h). The moisture content is defined by equation (3) :

$$\%H_u = \frac{M_0 - M_f}{M_f} \times 100 \qquad (3)$$

a) Fruit of *CS* b) Fruit kernel of *CS* c) CS kernel shells

Fig. 1-a: Process for obtaining CS shells

d) Grinding of CS shells e) CS powder sieving f) CS shell powder

Fig. 1-b: Process for obtaining CS shell powder

a) Density

In accordance with the French standard NFT 51-033, the density is determined with the Gay-Lussacs pycnometer. The sample is dried and then weighed, and the mass $M_0$is recorded. In a pycnometer, distilled water is poured until it is full and the massM_1 is weighed. This water is removed from the pycnometer, and the sample is introduced into the pycnometer; and the pycnometer is refilled with distilled water, weighed, and the mass M_2 is recorded. The density of the CS shells is determined by equation (2):

$$d = \frac{M_0 \times d_e}{(M_0 + M_1) - M_2} \qquad (2)$$

Where M_0mass of sample (g), M_1mass of water distilled in the pycnometer (g), M_2mass of sample and water distilled in the pycnometer (g); d_e The density of distilled water (g/cm^{-3}).

b) Moisture content

The moisture content is determined according to the French standard NF EN ISO 665. In a temperature-controlled chamber of 25°C ± 2 with 63% relative humidity, the samples are exposed for 24 hours, then weighed, a mass M_0 (g) initial wet mass; M_f(g) the mass obtained after 24 hours in the oven

C. Evaluation of the chemical composition of CS shells

The chemical analysis of the CS shells is carried out in accordance with the TAPPI method. The CS particles are sieved to an average particle size of about 160 µm, then dried for 24 hours in an oven at a temperature of 50°C.

a) Evaluation of the extractable content

A cartridge containing 8 g of CS shell powder is introduced into a Soxhlet mounted between a flask filled with ethanol (30%) and ketone (70%), which is then introduced into a paddle and a refrigerator, the whole fixed on a support. The separation of the extracts was carried out by regulated leaching so that a siphon was available every 10 minutes. Dissolution was carried out for 7 hours. The cartridges were removed from the Soxhlet, lyophilized at 50°C for 1 hour, and then cooled in a desiccator. The extractable content (%Es) is determined using equation (4) [16], [17], [18]:

$$\%Es = \frac{M_{Es}}{M_i} \times 100 \qquad (4)$$

M_{es} the mass obtained after extraction with ethanol-ketone; M_i Mass of previously dried hulls.

b) Evaluation of the water content (%W)

The dried cartridge (residue 1) of ethanol-ketone is introduced into the Soxhlet. The previous mixture is reconstituted by replacing the solvent with 100 ml of distilled water. The mixture is then heated at reflux for 7 hours. The residue 2 obtained is filtered on a sintered glass crucible, washed with distilled water, dried in an oven at 60°C for 12 hours, and weighed after cooling in a desiccator. The hot water extract content (%W) is determined by equation (5) [16], [17], [18]:

$$\%W = \frac{M_{es} - M_{res1}}{M_i} \times 100 \quad (5)$$

M_{es}mass of ethanol-ketone extraction; $M_{res\,1}$ dried mass of residue 1; M_i Mass of previously dried husks.

c) Evaluation of lignin content (%L)

The lignin content is determined by the Klason method: 500 mg of the dried residue 2 is hydrolyzed in a 72% sulphuric acid solution for 2 hours at room temperature and under stirring. The mixture is then diluted to 3% by adding a volume of distilled water and refluxed for 2 hours, filtered with a No. 4 filter crucible, washed with water to neutral pH, and placed in an oven at 103°C for 4 hours. The lignin content is determined using equation (6) [16],[17] :

$$\%L = \frac{M_{res1} - M_{res2}}{M_i} \times 100 \quad (6)$$

$M_{res\,2}$ dried mass of residue 2;$M_{res\,1}$ dried mass of residue 1; M_iMass of previously dried husks.

d) Evaluation of holocellulose content (%H)

A volume of 25 ml of a 2% soda solution is taken and introduced into the flask containing residue 2, and 2% oxygenated water is added to the mixture. The resulting residue 3 was washed, filtered, and air-dried for 24 hours. The holocellulose content is determined using equation (7) [16],[17],[18].

$$\%H = \frac{M_{res2} - M_{res3}}{M_i} \times 100 \quad (7)$$

M_{res3} dried mass of residue 3;$M_{res\,2}$ dried mass of residue 2; M_i Previously dried mass of husks.

e) Evaluation of the cellulose content (%C)

For the extraction of cellulose, a soda solution (17.5%) is prepared, 50 ml of this solution is introduced into the quantity of residue 3 obtained previously, the mixture is stirred for 30 minutes, then 50 ml of distilled water is added to bring the concentration back to 8%, then stirring again for 30 minutes. This residue is then washed with distilled water, filtered under vacuum, air-dried for 24 hours, and weighed. The cellulose content is obtained by equation (8):

$$\%C = \frac{M_{rés3} - M_{rés4}}{M_i} \times 100 \quad (8)$$

M_{res4} dried mass of residue 4; M_{res3} dried mass of residue 3;M_iPreviously dried mass of husks.

II.3.4 Evaluation of the hemicellulose content (%HC)

Holocellulose is composed mainly of cellulose and hemicellulose [16],[17],[18]. The hemicellulose content is determined using equation (9):

$$\%HC = \%H - \%C \quad (9)$$

D. FTIR analysis

The Fourier Transform Infrared Spectroscopy (FTIR) technique aims to determine the chemical composition of CS shells by extracting the functional groups and chemical bonds present in CS shells. The infrared spectrum is recorded using a BRUKER ALPHA spectrometer equipped with an attenuated total reflectance (ATR) module with a diamond crystal and controlled by Opus Lab v 7.0 122 software. A few milligrams of CS powder were deposited on the diamond crystal of the ATR module. Acquisitions are obtained by performing 20 scans in the frequency range 4000 and 400 cm^{-1}, at a spectral resolution of 4 cm^{-1}.

E. TGA-DSC analysis

This analysis allows the prediction of thermal stability and the quantification of mass losses due to decomposition, oxidation, or desolvation. It is performed on a NETZSCH STA 449F3 synchronous thermogravimetric analyzer with a resolution of 0.5 µg capable of combining TG (thermogravimetric) and DSC (differential scanning calorimetry) information using the same sample. The instrument is equipped with a high balance with a resolution of 0.1mg, an oven with a maximum calcination temperature of 1000°C, a very fast cooling system, and controlled by Opus Lab v 1.0 182 software.

A 100mg mass of CS powder (~ 315 µm) is introduced into an aluminum crucible, then introduced into the apparatus at a temperature rise from 20 to 900°C, with a rate of rising of 5°C/min, and an isotherm at 900°C for 30minutes under air.

F. Crystallinity index evaluation by FTIR-ATR

The crystallinity index (CrI) is evaluated by calculating the lateral order index (LOI), also called the O'Connor crystallinity index from the FTIR-ATR spectrum of CS shells [16], [17], [18]. After extracting the cellulose, and ATR-FTIR analysis is performed, which allows determining the crystallinity of CS shells with the maximum intensities of the FTIR spectra at 1453 cm-1 and 832 cm-1. It is defined by :

$$X = \frac{I_{1453}}{I_{832}} \quad (10)$$

III. RESULTS AND DISCUSSION

A. Density and moisture content

Table 1 shows the physical properties (density and moisture content) compared to the values of other fillers in the literature.

Table 1: **Physical characteristics of some fruit shell**

Shell	Density ($g.cm^{-3}$)	Moisture content (%)	References
CS shell	1,33	5,10	**Currentstudy**
Palmistshell	0,74	6,11	[19]
Argan tree shell	1,3	/	[2]
***Coco Nucifera*shell**	/	10,10±0,01	[20]
Hazelnutshell	1,02	/	[21]
Pistachioshell	/	6,99	[22]
Peanutshell	/	3,5	[23]
PKS Dura	1,502	/	[24]
PKS Tenera	1,381	/	[24]

Biomass-based reinforcements are characterized by their low density compared to mineral and organic fillers. Density is one of the physical characteristics that make the use of bio-fillers useful as reinforcement for bio-composites in the transport, construction, sports, and leisure industries [2]. The method used shows that the density of CS shells is 1.33 g/cm^{-3}. It is comparable to Arganier shells and other shells (PKS Dura and Tenera) due to its lightness which is conducive to reinforcing biocomposites. It is denser than palm kernel and hazelnut shells.

The average moisture content of CS shells is 5.10%. This relatively low-value content would predict good filler-resin interaction in the manufacture of composites reinforced with the new lignocellulosic resource [25],[42]. CS shells absorb more moisture than peanut shells and less than palm kernel, Coco Nucifera, and pistachio shells. High moisture content compromises the stability of the composite in terms of dimensions, tensile strength, swelling behavior, and porosity formation [26],[27]. Therefore, the lower moisture content is desirable, as it would make the composite hydrophobic.

B. Chemical composition

Table 2 presents the chemical components of the CS shells evaluated in the study. For comparison purposes, information on other fillers available in the literature is presented.

Table 2: Chemical composition of CS shells and some fruit shells.

Fruit shell	Hemicellulose (%)	Cellulose (%)	Lignins (%)	Ash (%)	References
CS	19,14	37,31	36,96	6 ,59	Currentstudy
Almond	27,74	21,72	36,12	6,85	[28]
Nuts	22	25,6	52,3	1	[21]
Olive	35	25	35	5	[29]
Hazelnut	28,7	25,9	44,4	1.3	[30]
Apricot	17	29,57	47,97	0,95	[31]
Pistachio	20,1	53,98	25,25	0,1	[32]
Wood	23,6	45,15	27,65	0,3	[33]
Argan treenuts	34,3	25,7	34,5	5,4	[2]
Coconut	29	35	28	3,38	[34]
PKS Dura	18,79/13,82	35,20	44,20	1,81/6,79	[24]
PKS Tenera	20,77/15,41	34,04	43,76	1,43/6,78	[24]

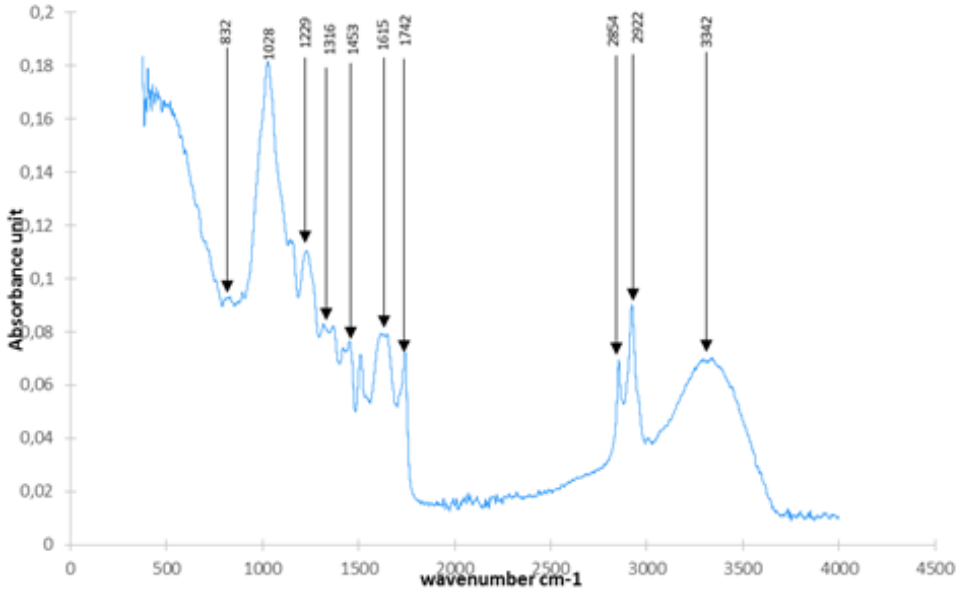

Fig. 3: Spectrum of CS shells

The results confirm that CS shells comprise three main components (more than 80%): cellulose, hemicellulose, and lignin. The rest of the composition includes extractives and ash. This corresponds to the chemical composition of some fillers in the literature [2],[28],[29],[34]. Cellulose is the main constituent of CS hulls which is 37.31% of the dry matter. Hemicelluloses constitute the second major constituent, i.e., 19.14%, while the percentage of lignin is around 36.96%, which is comparable to almond, argan, olive, and coconut shells [2],[28],[29],[34].

C. Results of the ATR-FTIR analysis

The ATR-FTIR analysis gave rise to the graph in Figure 3, which presents the FTIR spectrum of CS shells, and Table 3 the main IR bands corresponding to the characteristic functional groups of CS shells.

Table 3: The main IR bands corresponding to the characteristic functional groups of CS shells

CS	meanings	Component	References
3342	Characteristic of the stretching vibration of -OH bonds	Cellulose and hemicelluloses	[2] [35] [36]
2922	Stretch CH bond	Polysaccharides	[2] [35] [36]
2854	CH2 symmetric elongation of fats	Fats	[2]
1742	C=O ester group elongation of hemicellulose	Xylans (hemicelluloses)	[2] [36]
1615	Shear vibration of the -OH bond	Water	[2] [35]
1453	In-plane deformation of CH group and aromatic vibrations	Pectin, lignin, hemicellulose	[2]
1316	C-O aliphatic ring	Cellulose	[2]
1229	Vibration acetyl group (xylans)	Lignin	[2] [36].
1028	Stretching of C-O-C bonds in cellulose	Cellulose et hemicelluloses	[2] [35]
832	The vibration of the C-O-C glycosidic bond	Polysaccharides	[2] [35] [36].

The strongest peak at 1028 cm^{-1} is found to correspond to the vibration of the C-O-C bonds of cellulose [2],[35]. The characteristic stretching vibration of the -OH bonds of cellulose and hemicellulose is strongly reflected in the band at about 3342 cm^{-1} [2],[35],[36]. The characteristic peak at 2922 cm^{-1} shows the symmetrical stretching of the CH bond of cellulose and hemicellulose [2],[35],[36]. The 2854 cm^{-1} peak of the CS corresponds to the symmetrical elongation of the CH2 bond of fats. The 1742 cm^{-1} peak represents the symmetrical elongation of the carboxyl and acetyl groups (C = O) present in hemicelluloses [2],[35]. The 1615 cm-1 peak represents the -OH bond of free water [2],[35]. The 1453 cm^{-1} peak indicates the in-plane deformation of the C-H group and aromatic vibrations [2]. The 1229 cm^{-1} peak represents the deformation of acetyl groups (xylans) [2]. The 832 cm^{-1} peak indicates the vibration of the C-O-C glycosidic bond of polysaccharides [2],[35],[36]. These results show that the shells consist of lignin, hemicellulose, cellulose. They are similar to the constitution of argan, coconut, almond, and olive shells [2],[28],[29],[34].

Figure 3 is also used to estimate the crystallinity index of CS shells. The transmittances of the CS shell peaks are 1453 and 832 cm^{-1}. The crystallinity index value is 82% for CS shells.

D. Results of TG, DSC, and DTG analysis

Figure 4 shows the TG, DSC, and DTG curves of the CS shell, giving the thermal components' degradation temperatures.

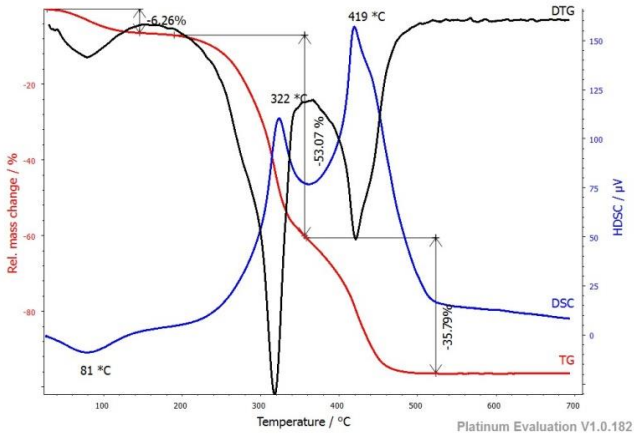

Fig. 4: TG, DSC and DTG of CS shells

a) Thermal degradation temperatures of chemical constituents of CS shells

The TG curve shows that the shells decompose into four phases. This behavior is typical for all fillers or plant fibers [18],[25],[27]. The first phase starts at 25°C and ends at 125°C, which is due to the evaporation of structural moisture and volatile extractives. The mass loss is recorded at 6.26% for CS shells. After dehydration, the mass variation becomes almost constant up to 210°C which marks the temperature of thermal stability of CS shells [37],[41].

In the second range from 210°C to 325°C, a significant mass loss of 53.07% is recorded. This mass loss is attributed to the thermal decomposition of hemicelluloses from 150°C to

210°C [38], and then of cellulose from 210°C to 380°C [39]. The third phase indicates the decomposition of lignin, a mass loss of 35.79% is recorded at 522°C, which marks a slow decomposition until the formation of ash [24]. In the last phase, the mass variation is very small, and this constant is due to the formation of ash. The TGA ash content is 4.88%.
The TG curve also allows determining lignin (%L), holocelluloses (%C+%H), ash, and volatile contents.
The values shown in Table 4 are correlated with the values obtained by the TAPPI method, which shows the accuracy of the chemical and thermal results and thus the behavior of the CS shells.

Table 4: Chemical composition deduced from TGA and TAPPI method.

	Lignins (%)	Holocelluloses (%)	Ash (%)	Volatiles (%)
***CS*shell**	35,79	53,07	4,88	6,26
	36,96	56,45	6,59	/

Table 5 will give the thermal properties of CS shells from TG-DTG.

Table 5: Thermal properties of TG-DTG of CS shells.

CS shell	Degradation temperature (°C)			Thermal stability (°C)	Résidual charge (%)
	T_{ID}	T_{MD}	T_{FD}	T_{FD}	
	210	325	522	[125-210]	4,88

b) Thermal degradation temperature of CS shells
Figure 4 shows the DSC curve, which gives the degradation temperatures of the thermal components.

The DSC curve shows that there are three peaks, one endothermic and two exothermic peaks. The first endothermic peak is reported at 81°C, which corresponds to the dehumidification of the CS shell [35]. The exothermic peaks are observed around 322°C and 419°C. These peaks correspond to the thermal degradation and decomposition of the cellulose and lignin present in the CS shell [36]. Cellulose is a linear macromolecule and is therefore expected to show melting behavior when heated. In addition, the macromolecular chains of cellulose are made up of "O" ether bonds, which means that the ether bonds can be broken with thermal energies lower than the energies needed to melt them [24].

IV. CONCLUSION

In this study, the physical and thermal characteristics of CS shells were evaluated to exploit them as a reinforcement for the shaping of composite materials. The density and moisture content were evaluated according to the recommendations of NFT 51 063 and ASTM D1576-13. The results gave a density of 1.33 g.cm^{-3} and a moisture content of 5.10%. The chemical composition of the CS shells was obtained by the TAPPI method on the shells reduced to 315μm. This shows that they contain 36.96% lignin, 37.31% cellulose, 19.14% hemicellulose, and 6.59% ash. Also, the TG-DTG analysis shows 35.79% lignin, 53.07% holocellulose, 4.88% ash, and 6.26% volatiles. The thermal transition phases observed from the DSC and TG-DTG thermograms show three degradation zones for hemicelluloses/pectins, cellulose, and lignin. This shows that the maximum limit temperature for using, processing, or manufacturing CS-reinforced composites is 360°C[41]. According to the FTIR spectrum, the crystallinity index of CS shells is 82%.

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CONFLICTS OF INTEREST

The authors declare no conflict of interest regarding the publication of this article.

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