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Research Article

Structural, Anatomy Characteristics and Thermal Properties of *Ampelodesmos mauritanicus* (*Diss*)

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Abstract

The structure, anatomical characteristics and thermal properties of the plant ampledosmos mauriatnicus were analyzed. The analysis of mineral compounds was carried out by X-ray fluorescence (FRX). Physical properties were investigated by Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffraction and Thermogravimetric analysis (TGA). Scanning electron microscopy (SEM) was used to investigate the structure and morphology of sample. The results reveal the % (W/W) cellulose content of Diss is 28.13 %, hemicelluloses content is 26.26% and lignin content is 24.95 %. Analysis of extractives contents in Diss were revealed to be 12.03 %. According to XRD data, Diss showed, a crystallinity index (CI) 52.5 %. High crystallinity of cellulose induces thermal decomposition of fibers at high temperatures. These results show that this plant is rich in cellulosic fibers and that could be used as raw material in industries and paper making.

Keywords: *Ampelodesmos mauritanicus*; Cellulose; Hemicellulose

Introduction

In recent years, more attention has been given to lignocellulosic biomass for sustainable development and environmental preservation [1-3]. Studies focusing on lignocellulosic biomass are increasingly important in view of the various applications [4-7]. The main biomass compounds are cellulose; hemicellulose and lignin. Hemicelluloses and cellulose are polysaccharides. Lignin is made of phenolic polymers that consist of three types of phenylpropaneuntis: p-coumaryl alcohol, coniferyl alcohol, and sinapyl alcohol [8-10]. Cellulose is a polymer of beta-D-glucopyranose moieties linked via beta- glycosidic bonds [11,12]. The cellulosic chains have a variable degree of polymerization between 10000 units of glucopyranose in wood to 15000 units in native cotton [13-15]. The properties of cellulose depend on several parameters such as chain length, crystallinity, hydrogen bonds and the distribution of functional groups [8-16-18]. These important parameters make cellulose a unique material. Therefore, one of the most important sources of cellulose remains wood, however the use of annual plants can be used. Increasingly, annual plants are being used as a potential source for industrial processing. These plants are interesting because of their abundance and low cost and their main advantage is their rapid growth which makes them available in many regions of the world [19-21]. Annual plants, Diss (*Ampelodesmos mauritanicus*), can be also used as an alternative to wood for cellulose-based materials. This plant species belongs to the poaceae, is found in the Mediterranean and resists drought well [22-26]. To appreciate the potential of this plant, a physical, chemical, structural and thermal characterization is necessary. The aim of this work was to characterize the structural fibers of Diss from the north east of Boumerdes in Algeria by using Fourier transform infrared (FTIR) to determine the functional groups present in plant, X-ray fluorescence (XRF) to estimate the quantity of elemental oxides, X-ray diffraction (XRD) to determine the degree of crystallinity in the plant, thermogravimetric analysis (TGA) of the thermal properties of the plant and scanning electron microscopy (SEM) to investigate the structure and morphology of the plant.

Materials and Methods

Materials

The stems of the "Diss" plant were harvested in the Boumerdes region of Algeria, the stems were rinsed with water to remove impurities. The stems were crushed until a homogeneous vegetable powder was obtained and was conserved from light and humidity.

Methods

Extractives

A 2.5g sample of the vegetable powder of Diss previously prepared was put in a cellulose cartridge which will be extracted with Soxhlet with 150ml of solvent composed of a mixture of toluene and ethanol with a ratio of 2:1. The extraction at the Soxhlet lasts 6 hours. After extraction; the sample is dried at 105 °C and the weight difference between the sample before and after extraction represents the extractives content [27].

Lignin

3g of the vegetable powder were put in glass test tubes with 30ml of 1.5% dilute sulfuric acid with stirring at 30mn intervals

for 150mn. After the initial hydrolysis, 10ml of distilled water was added. The tubes are autoclaved at 121 ° C for 1 hour for a second hydrolysis. After autoclaving, the mixture was filtered under vacuum and the residue was dried at 105 ° C to determine the amount of lignin insoluble in acid. Acid soluble lignin was determined by absorbance at 205nm. Total lignin was the sum of soluble and insoluble lignin [28].

Hemicelluloses

1 g of vegetable powder of Diss was mixed with 150 ml of NaOH (0.5 mol/L) in a 250 ml Erlenmeyer flask to boil them with distilled water for 3.5 h. the mixture was vacuum filtered and washed to neutral pH. The powder residue was dried at 105 ° C. The hemicellulose content (% w/w) was calculated by the difference in weight between the sample before and after treatment [28].

Cellulose

The cellulose content was determined using the Kurschner Hoffner approach. 5g of the vegetable powder of the Diss previously extracted from its extractives were mixed with 125ml of the alcoholic nitric acid solution composed of one volume of 65% nitric acid and four volumes of 96% ethanol; the extraction is carried out by reflux for 1 hour (four cycles). Nitric acid is renewed at each cycle. At the end of the experiment, the cellulose was washed and dried [29].

Analysis by X-ray fluorescence spectrometry

X-ray fluorescence spectrometry is an overall elemental analysis technique that identifies and determines most of the chemical elements that make up a sample. The analyzes of the plant powders were carried out by X-ray fluorescence (XRF) on the PHILIPS PW 1480 spectrometer with a dispersive technical wavelength. Before the chemical analysis, each sample was heated to 1000 ° C for 2 hours, and the decrease in mass was taken as loss during ignition.

Analysis by Fourier Transform Infrared Spectroscopy (FTIR)

The plant powder was analyzed by infrared (IRTF), using a "Bruker Thermo Scientific Waltham, MA, USA" spectrometer, by preparing KBr pellets containing 1% by weight of the plant powder. The spectra were recorded between 400 and 4000 cm-1 at a resolution of 4 cm^{-1}. The recorded spectra are the average of 32 scans.

X-ray diffraction analysis (DRX)

The plant powder samples were scanned for the wave number 0 - 4000 cm^{-1} and degree 2θ (0-50 °). The X-ray diffractometer used is of the D8 Advance BRUKER type, using monochromatic Kalpha1 radiation of copper (CuKα-radiation) (λ = 0.154 nm) at 45 kV and continuous scanning. In order to determine the degree of crystallinity of the cellulose, the crystallinity index was determined by the empirical method of the height of the DRX peak developed by Segal., *et al*. [30] which examines the DRX spectra. The crystallinity index (CI) was calculated from the ratio of the peak height of 002 (I002) to the height of the minimum value (IAM) between 002 and the tips 101, using equation (1).

$$CI = (I002 - IAM)/I002) \times 100 \quad (1)$$

CI: Crystallinity Index

I002: Maximum intensity of the diffraction peak

IAM: Intensity diffused by the amorphous phase of the sample

Thermogravimetric analysis (TGA)

The combustion performance of Diss was monitored with a thermogravimetric analyzer (SDT Q600 - TA instrument). To know the temperature where devolatilization occurs, we measure the mass loss of the Diss sample during the combustion of the material. The sample was heated at temperatures ranging from 50° The recording of the temperature measurements and the time degradation of the sample for the two analyzes TGA and DTG are done at the same time. The amount of powder sample used in this analysis is 4 mg.

Scanning electron microscopy

This technique capable of producing images of the surface of the studied sample using the principle of electron-matter interactions. The apparatus used is a scanning electron microscope of the FEI Quanta 650 type which makes it possible to analyze non-conductive samples such as plants, operating at 30 kV. The micrographs obtained make it possible to observe the microstructure of the surface of the vegetable powder.

Results and Discussion

Mineral composition

Several factors influence the concentration of a mineral compound in a plant such as the development stage, growth phase and age of the plant. Mineral compounds vary significantly depending on soil and climatic conditions [31]. According to the results of the FRX, the majority mineral element was silica with a content of 13.77%. This result is in agreement with that found from MacManus., *et al.* (1977) in grass plants.

Sample	Mineral composition (%)										
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	MnO	P_2O_5	SO_3
Diss	13.77	<0.05	0.09	1.21	0.97	1.47	3.5	<0.05	<0.05	0.13	0.97

Table 1: Mineral Composition of Diss Sample (% W/W).

Chemical composition

Table 2 shows the results of the different chemical compounds in the sample. The ash content was 8.63%, a high content as is the case for all non-wood raw materials. The result obtained for the ash content was adequate with what was observed for stalk fibers by Hurter (1988) [32].

The percentage of the The Küschner–Hoffer cellulose was at a satisfactory level (28.13%), it is advantageous for the pulp and paper industry because it yield higher pulp content after cooking process. The first fiber components responsible for the behavior of initial thermal degradation and associated with the moisture content

are hemicelluloses. The Diss fibers had the total lignin (Klason and acid soluble lignin) content with around 24.95%. The lignin content of the Diss sample is 24.95%, a content slightly lower than that found in wood (25-37%) [33,34]. According to Table 2, the amount of total extractive was found 12.03 %. The extractives content was 12.03% (Table 2), this content is similar to the content found in tropical woods (*Dipteryx odorata* and *Mezilaurus itauba*) [33].

Sample	Compositional Analysis of Diss (% W/W)							
	Moisture	Minerals matter	Organic matter	Extractives	Klason Lignin	Acid soluble lignin	Hemicellulose	Cellulose
Diss	12	8.63	91.37	12.03	22.08	2.87	26.26	28.13

Table 2: Compositional Analysis of Diss (% W/W).

FT-IR spectra

Figure 1 shows the FTIR spectra of the Diss sample. The results show:

- A wide band at 3400 cm^{-1} relating to the O-H stretching modes.
- Two bands at 2920 and 2850 cm^{-1} relating to the asymmetric and symmetrical methyl and methylene stretching groups present in the spectrum of all the components of the Diss sample. This is due to the high content of extractives which are represented by fatty acid methyl esters and phenolic acid methyl esters [35,36].
- A band at 1800-1500cm^{-1} relating to the lignin network [37].
- A band at 1730 cm^{-1} relates to hemicelluloses (acetyl group) and to lignin (uronic ester or to the carboxylic ester group of the ferulic cycle and of p-coumaric acid).
- Two bands at 1552 cm^{-1} and 1514 cm^{-1} are assigned to the groups C = C and C-O present in lignin [38].
- A band at 1200-900cm^{-1} assigned to the polysaccharides is strongly intense proves that the polysaccharides in the sample are present in large quantities.
- A band at 1040 cm^{-1} is due to the group C-OH
- A band at 1159 cm^{-1} is allocated to the vibration C-O-C [33] and
- A peak at 896 cm^{-1} due to the C-H deformation of the cellulose [39].

Figure 1: FTIR of Diss.

X-ray diffraction

The XRD results are summarized in Figure 2. From these results it is observed that the peak 002 is of an asymmetrical shape. According Wada and Okano (2001), the 2θ =22° and the 2θ = 14.6° reflection assigned to the (002) and (110) crystallographic plan of cellulose, and the 2θ =18° reflection assigned to the (110) amorphous phase.

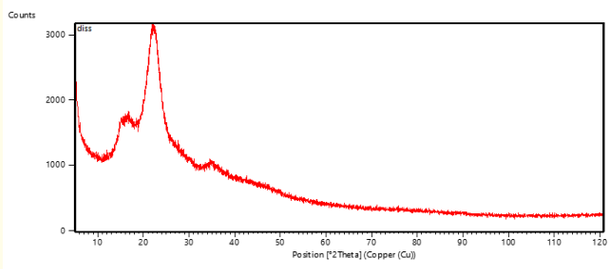

Figure 2: XRD diffractogram of Diss.

The crystallinity is influenced by the compounds of the raw material such as cellulose, hemicellulose and lignin which vary according to the botanical nature of the plant [40]. The crystallinity index was obtained from the ratio between the intensity of the 002 peak (2θ = 22°) and the minimum intensity diffraction (2θ = 18°) according to the Segal., *et al.* (1959) equation (1) [30]. The results show that crystallinity of the Diss was high, as presented in table 3. These results indicate that Diss contains a ordered cellulose structure [41].

Sample	Crystalline Intensity I_{22} (at 2θ Scale)	Amorphous Intensity I_{18} (at 2θ Scale)	CI (%)
Diss	3178	1508	52,5

Table 3: Crystallinity Index of Diss Based on XRD.

CI : Crystallinity Index.

Thermogravimetric analysis

The pyrolytic behavior of the fibers in the sample was evaluated. The results of TGA and DTG of Diss are shown in figure 3. The results of TGA weight loss in three stages and decomposition in two. An initial weight loss at a temperature of 192.54°C evaluated at 5.28% due to the volatilization of humidity and low molecular weight compounds. A 23.57% weight loss due to the decomposition of hemicelluloses around the degradation temperature of the first stage T1 from 192.54°C to 280.12°C. The second stage thermal degradation temperature, T2 around 280.12°C to 390.01°C, corresponds to 47.29% of weight loss of cellulose and lignin present in the Diss. Between 390°C and 800°C, a weight loss of 23.86% due to the degradation of lignin was observed. The hemicelluloses depoly-

merize between 180°C and 350°C, the glycosidic bonds of cellulose between 275°C and 350°C and the lignine degrades between 250°C and 500°C according to a recent study by Mazlan *et al* [42]. The differences between the degradation temperatures are explained by the differences in chemical structures, therefore; hemicelluloses which are of an amorphous structure are easily hydrolyzable [43], while cellulose which is of a more crystalline structure improves its thermal stability [44]. Highly crosslinked lignin with a high molecular weight generates very high thermal stability which makes it difficult to decompose [45].

The curves in Figure 3 which presents three main peaks confirm the results found. At 78.22°C (first peak) the humidity begins to volatilize. The second peak observed at 330.05°C in Diss curve assigned to cellulose decomposition. The last peak appears around 475°C in curve obtained attributed to lignin decomposition.

Figure 3: TGA and DTG of Diss.

Samples fibers	1st stage degradation in TGA curve		2nd stage degradation in TGA curve		Residual mass % at 800°C (%. °C^{-1})
	Temperature range (T_1) (°C)	Weight Loss (%)	Temperature Range (T_2) (°C)	Weight Loss (%)	
Diss	192.54 to 280.12	23.57	280.12 to 390.01	47.29	23.86

Table 4 : TGA Results of Diss.

SEM image analysis

Figure 4 shows the morphology of the Diss sample. In longitudinal section, there are defects on the surface of the fiber which prevents an appreciation of the fibrillar structure of the sample. On the other hand, in cross section, a considerably uniform circular and fibrillar porous structure is observed. However, some lignin or lignin carbohydrate complexes may be condensed on the surface of cellulose fibers [46].

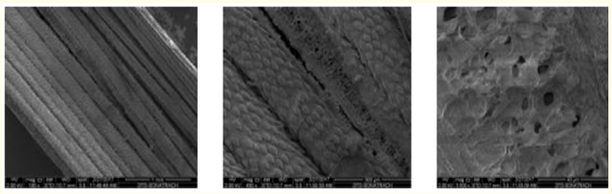

Figure 4: SEM images of Diss.

Conclusion

The physical, chemical and thermal characteristics of Diss fibers, were discussed in this study.

The chemical analysis revealed that the samples of the contain a higher quantity of extractives and a lower quantity of cellulose and hemicelluloses than wood. The results of the FTIR demonstrated a significant amount of polysaccharide associated with the bands 1200-900cm^{-1}. The Scanning Electron Microscopy (SEM) shows that the raw fibers have smooth and homogenous surface with highly porous texture and clearly shows the presence of longitudinally oriented unit cells with almost parallel orientations.

Obtained results indicate us, that the Diss can be an alternative non-wood raw material for pulp, paper, and hydrolysis chemistry.

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