

CHEMICAL AND STRUCTURAL ANALYSIS OF
LIGNOCELLULOSIC BIOMASS OF *AMPELODESMOS*
MAURITANICUS (DISS) AND *STIPA TENACISSIMA*

CHENAH MAY, AMRANI MOUSSA

M'HAMEDBOUGARA UNIVERSITY, FACULTY OF SCIENCE LABORATORY OF SOFT TECHNOLOGY,
RECOVERING, AND SUSTAINABLE DEVELOPMENT
BOUMERDES, ALGERIA

(RECEIVED SEPTEMBER 2017)

ABSTRACT

Chemical composition, anatomical characteristics and cell wall structure of *Ampelodesmos mauritanicus* (Diss) and *Stipa tenacissima* (Esparto grass) fibers were analyzed. The results reveal the % (W/W) holocellulose content of Diss and Esparto grass was found to be 54.39 % and 51.8 %, respectively. Esparto grass had the greatest % (W/W) lignin content of 32.2 % while Diss had the last lignin content of 24.95 %. Estimation of % (W/W) extractives contents in Diss and Esparto-grass were observed to be 12.03 % and 13.5 % in that order. According to XRD data, Diss showed, as expected, a lower crystallinity index (CI) 52.5 % when compared to Esparto grass samples 55.2 %. The results of the thermal decomposition of natural fibers are shifted to higher temperatures with increasing the cellulose crystallinity.

Based on the results of the chemical composition of Diss and Esparto grass, it was confirmed that straw is rich in cellulosic fibers and, therefore, a valuable raw material for the paper and manufacture industry.

KEY WORDS: Diss, esparto grass, lignin, cellulose, holocellulose, cristallinity.

INTRODUCTION

In recent years, more attention has been given to lignocellulosic biomass for sustainable development and environmental preservation (Khalil et al. 2012, Reddy et al. 2013, Khan et al. 2014). There has been spectacular development and rapid growing interest in renewable biopolymers derived, especially from lignocellulosic biomass for a wide range of applications (biodegradable packaging materials, automotive industries, agriculture, pharmaceuticals...) (Belgacem and Gandini 2008, Ahmadzadeh et al. 2009, Ali et al. 2009, Thomas et al. 2009).

The major components of lignocellulosic biomass are cellulose, hemicellulose, and lignin, in which cellulose and hemicellulose are generally referred to as polysaccharides. Lignin is made of phenolic polymers that consist of three types of phenylpropane units: p-coumaryl alcohol, coniferyl alcohol, and sinapyl alcohol (Cordeiro et al. 2004, Foyle et al. 2007, Sorek et al. 2014). Cellulose is a polymer of beta-D-glucopyranose moieties linked via beta-glycosidic bonds (Klemm et al. 2005, Moon et al. 2011). The degree of polymerization of cellulose chains in nature ranges from 10000 glucopyranose units in wood to 15000 in native cotton (Fengel and Wegener 1984, Zablackis et al. 1995, Desvaux, 2005). The reactions and properties of cellulose are determined by the using isolation process, the number of inter and intramolecular hydrogen bonds, the chain lengths, the chain length distribution, the crystallinity and by the distribution of functional groups within the repeating units and along the polymer chains (Saha 2003, Cordeiro et al. 2004, Laureano et al. 2005, Pratima 2016). These important parameters make cellulose a unique material. Therefore, wood remains one of the most important lignocellulosic biomass sources for obtaining cellulose, but annually renewal plants can be used as well. In recent years, annual plants have been considered as a potential source of raw material for industrial transformation. Annual plants, are low-cost raw materials, therefore, they are interesting sources of alternative fibers to wood cellulose fibers (Sridach 2010, Run Cang 2010, Plazonic et al. 2016).

The benefit of annual plants as fiber sources is their fast annual growth. Therefore, these raw materials represent an annual fiber resource available in abundant quantities in many regions all over the world.

Annual plants, Diss (*Ampelodesmos mauritanicus*) and Esparto grass (*Stipa tenacissima*), can be also used as an alternative to wood for cellulose-based materials. These plant species belong to the grass family, an endemic of the west Mediterranean, which grows on the semi arid grounds of North Africa and South Spain. They were localized, especially in the western part of the Mediterranean region (Paiva 2006, Akchiche 2007, Merzoud 2007, Bourahli 2014, Sellami 2015). In order to evaluate completely the potential of these plants for new applications, a detailed and, comprehensive study of chemical and physical characteristics is necessary.

The aim of this work was to characterize the structural fibers of Diss and Esparto grass by using Fourier transform infrared (FTIR) spectroscopy to determine the functional groups present in both plants, X-ray fluorescence (XRF) to estimate the quantity of elemental oxides, X-ray diffraction (XRD) to determine the degree of crystallinity in the plants, thermogravimetric analysis (TGA) of the thermal properties of the plants and scanning electron microscopy (SEM) to investigate the structure and morphology of the plants. The research result can also lead to a better understanding and knowledge of chemical composition.

MATERIAL AND METHODES

Materials

Diss (Fig. 1) and Esparto grass (Fig. 2) were collected from the north east of Boumerdes area, Algeria and from the south west of Saida area, Algeria, respectively. Both biomass materials were harvested in early June 2016. The sample was washed with water to remove dirt, dried at 60°C for 8 h in an air oven and reduced to about 2 cm in length, with a razor blade, for effective milling to a size less than 500 µm. The residues after this milling were used for the compositional analysis.



Fig. 1 : Tuft of Diss. Fig. 2: Tuft of Esparto grass.

Methods

Solvent extractives

Into the cellulose thimble, loaded 2.5 g of dried raw biomass. With the Soxhlet extractor set up, 150 mL of mixture of toluene ethanol ($C_6H_6 - C_2H_5OH$) solvent in a ratio of 2:1 was used as solvent for extraction for 6 hours. After extraction, the sample was air dried at room temperature for few minutes. Constant weight of the extracted material was achieved in a convection oven at 105 °C. The % (w/w) of the extractives content was evaluated as the difference in weight between the raw extractive-laden biomass and extractive-free biomass (TAPPI T204 cm-07). (Blasi, 1999).

Lignin

In glass test tubes, 0.3 g of dried extracted raw biomass was weighed and 3 mL of 72% H_2SO_4 was added. The sample was kept at room temperature for 2.5 h with careful shaking at 30 min intervals to allow complete hydrolysis. After the initial hydrolysis, 84 mL of distilled water was added. The second step of hydrolysis was made to occur in an autoclave for 1 h at 121°C. The slurry was then cooled at room temperature. Hydrolyzate was filtered through vacuum using a filtering crucible. The acid insoluble lignin was determined by drying the residues at 105°C and accounting for ash by incinerating the hydrolyzed samples at 575°C in a muffle furnace. The acid soluble lignin fraction was determined by measuring the absorbance of the acid hydrolyzed samples at 320 nm. The lignin content was calculated as the summation of acid insoluble lignin and acid soluble lignin (TAPPI T222 om-11) (Sluiter 2008).

Hemicelluloses

Into a 250 mL Erlenmeyer flask, 1 g of extracted dried biomass was transferred and 150 mL of 0.5 mol/L NaOH was added. The mixture was boiled for 3.5 h. It was filtered after cooling through vacuum filtration and washed until neutral pH. The residue was dried to constant weight at 105°C in a convection oven. The difference between the sample weight before and after this treatment is the hemicelluloses content (% w/w) of dry biomass (Sluiter 2008).

Cellulose

The cellulose content was determined following Kurschner Hoffner approach which consists of treating 5 g of extractives-free samples with 125 ml of alcoholic nitric acid solutions under reflux during four cycles of 1 h. After each cycle, the alcoholic nitric acid solution is removed and a fresh volume is added. The alcoholic nitric acid solution consisted in mixing one volume of 65% (w/w) solution of nitric acid with four volumes of 96% purity ethanol; at the end of the four cycles, the cellulose was washed, dried and weighed (Ouensanga 1989).

Mineral analysis

The elemental analysis of samples was conducted by X-ray fluorescence (XRF) on PHILIPS PW 1480 spectrometer that has used a dispersive technical wave length. Before chemical analysis, each sample was heated at 1000°C for 2 hours, and the decrease in mass was taken as the loss on ignition (LOI).

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectra were obtained through a Bruker spectrometer (Thermo Scientific, Waltham, MA, USA). Raw material powder samples of each species (5 mg) were dispersed in a matrix of KBr (100 mg), followed by compression to form pellets. The sample collection was obtained by using 32 scans, in the range of 4000 to 400 cm^{-1} , at a resolution of 4 cm^{-1} .

X-ray Diffraction

XRD patterns of the samples were obtained at room temperature on a Bruker AXS D-8 diffractometer using Cu-K α radiation in Bragg-Brentano geometry (θ - 2θ). The diffraction patterns were obtained at diffraction angles between 5 and 50° with a scanning rate 0.02 °/min at room temperature.

The crystallinity index (CI) of samples was determined following the method of Segal et al. (1959). (Gümüskaya 2003, Popescu 2011), which is:

$$CI = (I_{002} - I_{am}) / I_{002} \quad (1)$$

Where I_{002} and I_{am} were the intensity of the peak at $2\theta = 22^\circ$ and of the peak at $2\theta = 18^\circ$, respectively. Where I_{002} is the maximum intensity of the (002) lattice diffraction; and I_{am} is the intensity diffraction of the amorphous band.

Thermogravimetric analysis (TGA)

The combustion performance of Diss and Esparto grass was monitored with a thermogravimetric analyzer (SDT Q600 - TA instrument). The mass loss of the sample was measured as a function of time and temperature as the material is combusted. The rate of mass loss (DTG) was also evaluated in order to determine the exact temperature where evaporation occurred. The sample was heated at temperatures ranging from 50°C – 800°C at a rate of 10°C·min⁻¹ heating rate under constant nitrogen flow (100 mL·min⁻¹). The temperature measurements of both the TGA and DTG were simultaneously recorded during the analysis as well as the time degradation of the sample. Approximately, 4 mg of each sample were used.

Scanning electron microscopy

The Scanning Electron Microscope type FEI Quanta 650 was used to characterize the morphological investigations of Diss and Esparto grass fibers at the accelerating voltage of 30 KV. Samples were prepared by mounting them on specimen stubs using double-coated tape. Excess material was gently blown off before SEM measurement.

RESULTS AND DISCUSSION

Mineral composition

The minerals are known to vary widely depending on the agronomical factor and amount of contaminated soil. The concentration of particular mineral substance in a plant varies depending on

plant age or stage of development plant species, and climate conditions during the growth phase of plant (McManus et al. 1977). The data of XRF analysis (Tab. 1) show that the main elemental constituents of Diss and Esparto grass minerals were Si, Ca, K, Na, Mg and S, with smaller amounts of P, Fe, Mn, Ti and Al.

Tab. 1: Mineral composition of Esparto grass and Diss Samples (% W/W).

Sample	SiO ₂ (%)	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	TiO ₂	MnO	P ₂ O ₅	SO ₃
Esparto grass	27.6	0.22	0.14	2.16	1.57	0.49	0.89	<0.05	<0.05	<0.05	1.15
Diss	13.77	<0.05	0.09	1.21	0.97	1.47	3.5	<0.05	<0.05	0.13	0.97

The content of SiO₂, Al₂O₃, Fe₂O₃, CaO, SO₃ and MgO were greater in Esparto-grass than Diss. However, P, K and Na concentrations were great in Diss. The major mineral components of Esparto grass and Diss is silica, with a SiO₂ content of 27.6 % and 13.77% respectively. These were in agreement with the observation of McManus et al. (1977) as the major minerals found in grasses plant.

Chemical composition

Tab. 2 shows the percentage of various chemical components present in the fibers used in this study. As expected, ash content determined at 525°C was relatively high in both analyzed straws, considering that it is a distinguishing quality for all non-woody raw materials. Diss fibers exhibited the highest ash content (8.63%), compared to Esparto-grass fibers (2.5%). The result obtained for the ash content compared adequately with what was observed for stalk fibers by Hurter (1988).

Tab. 2: Compositional analysis of Esparto grass and Diss (% W/W).

Content	Esparto grass (%)	Diss (%)
Moisture content	14	12
Minerals matter	2.5	8.63
Organic matter	97.5	91.37
Extractives	13.5	12.03
Holocellulose	51.8	54.39
Lignin	32.2	24.95
Hemicellulose	19.63	26.26
Cellulose	32.17	28.13

The holocellulose content was determined to be around 51.8 % for Esparto grass and 54.39% for Diss. These two percentages were at a satisfactory level (above 60%) for each type of non-woody considered in present study. However, the Esparto grass fibers had the highest lignin content with around 32.2 %, while Diss fibers had the lowest at 24.95%. Generally, the percentage of lignin content in Diss fibers such as in Esparto-grass was still lower than that of wood fiber (25 – 37 %). (Matheus et al. 2014, Augustine et al. 2015). From results in Tab. 2, the amount of total extractive was found 12.03 % and 13.50 % in Diss and Esparto grass respectively. That present amounts of extractives (Tab. 2) were similar to those encountered in tropical woods, such as *Dipteryx odorata* and *Mezilaurus itauba* which are three times higher than temperate woods, such as *Eucalyptus grandis* and *Pinus elliottii* (Poletto et al. 2014).

FT-IR spectra

The FTIR spectra for vegetal fibers studied are shown in Fig. 3. It can be observed that there is a strong broad band at around 3400 cm^{-1} , which is assigned to different O–H stretching modes, and another two bands at around 2920 and 2850 cm^{-1} , related to asymmetric and symmetric methyl and methylene stretching groups present in the spectra of all of the fiber components of Diss and Esparto grass.

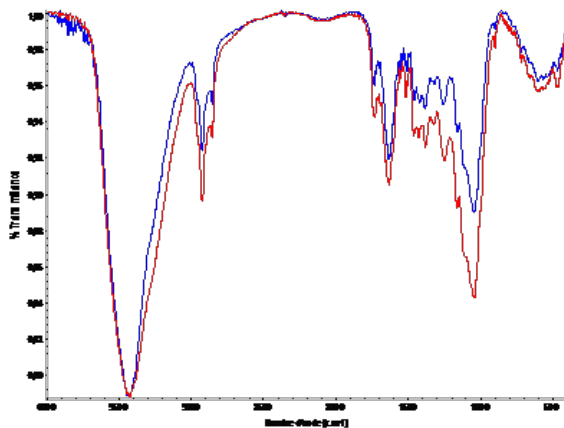


Fig. 3: FTIR for non woody (a) Diss and (b) Esparto gras.

This might be attributed to the higher extractive contents in these two fibers, as we see in Tab. 2, since some compounds in organic extractives, such as fatty acid methyl esters and phenolic acid methyl esters, contain methyl and methylene groups (Yokoi et al. 2003, Ishida et al. 2007). The carbon double bond region will appear around $1800 - 1500\text{ cm}^{-1}$. These carbonyl bonds are attributed to the lignin network (Wada and Okano 2001). The band at 1730 cm^{-1} is attributed to the acetyl group of hemicelluloses uronic ester or the carboxylic ester group of the ferulic ring and p-coumaric acid of lignin and/or hemicelluloses and the band at 1634 cm^{-1} is attributed to C-Ph which is generally found in the lignin aromatic structure (Wada and Okano 2001). The bands at 1552 cm^{-1} , 1514 cm^{-1} are assigned to C=C and C–O stretching or bending vibrations of different groups present in lignin (Wada and Okano 2001, Mészáros 2007). A peak near 1250 cm^{-1} associated with C–C, C–O and C=O stretchings in the lignin; and a prominent band in the rang $1200 - 900\text{ cm}^{-1}$, attributed to the presence of polysaccharides. The high intensity of this band in all samples shows that polysaccharides are present in large amounts. The band observed in the spectra of Diss at 1040 cm^{-1} and of esparto grass at 1045 cm^{-1} is due to the C–OH stretching vibration and the peak near 896 cm^{-1} indicates C–H rocking vibration of cellulose present in the samples. The band at 1159 cm^{-1} is assigned to C–O–C stretching vibration (Kondo and Sawatari 1996, Matheus et al. 2014) and peak can be observed at 896 cm^{-1} is attributed to the C–H deformation in cellulose (Chen et al. 2010).

X-ray diffraction

Fig. 4 shows the XRD results for Diss and Esparto grass. From this figure, we can observe that the shape of the 002 peak at $2\theta=22^\circ$ in all samples is asymmetric. According Wada et al. (2001), the $2\theta = 22^\circ$ and the $2\theta = 14.6^\circ$ reflection assigned to the (002) and (110) crystallographic plan of cellulose, and the $2\theta = 18^\circ$ reflection assigned to the (110) amorphous phase.

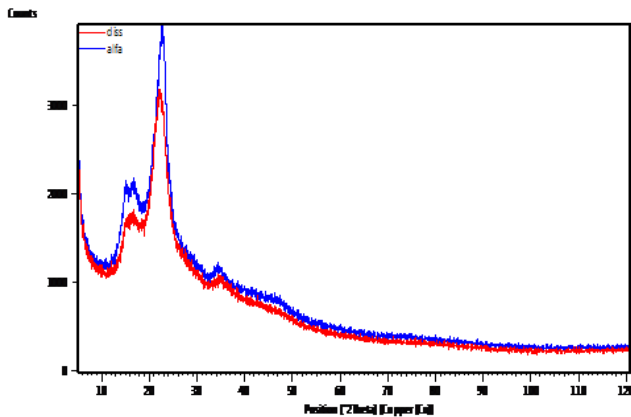


Fig. 4: XRD diffractograms Diss and Esparto grass.

The degree of cellulose crystallinity is one of the most important crystalline structure parameters. The rigidity of cellulose fibers increases and their flexibility decreases by increasing the ratio of crystalline to amorphous regions. The crystallinity is strongly influenced by the composition of biomass; the relative amount of lignin, hemicelluloses and cellulose varies according to the nature of the biomass (Gümüşkaya et al. 2003, Kim et al. 2010). The intensity of the peaks of Esparto grass was significantly higher than that of the Diss (Fig. 4). The crystallinity index was obtained from the ratio between the intensity of the 002 peak ($2\theta = 22^\circ$) and the minimum intensity diffraction ($2\theta = 18^\circ$) according to the Segal et al. (1959) Eq. 1. The results show the crystallinity of the esparto grass was higher than that of the Diss, as presented in Tab. 3.

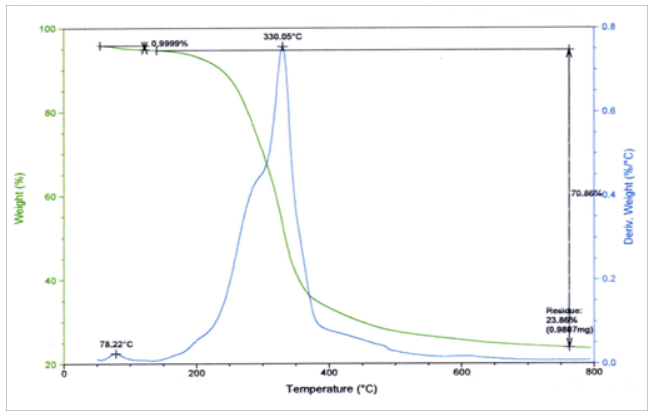
Tab. 3: Crystallinity Index of Esparto grass and Diss Based on XRD.

Samples	Crystalline Intensity I_{22} (at 2θ Scale)	Amorphous Intensity I_{18} (at 2θ Scale)	CI (%)
Diss	3178	1508	52,5
Esparto grass	3890	1743	55,2

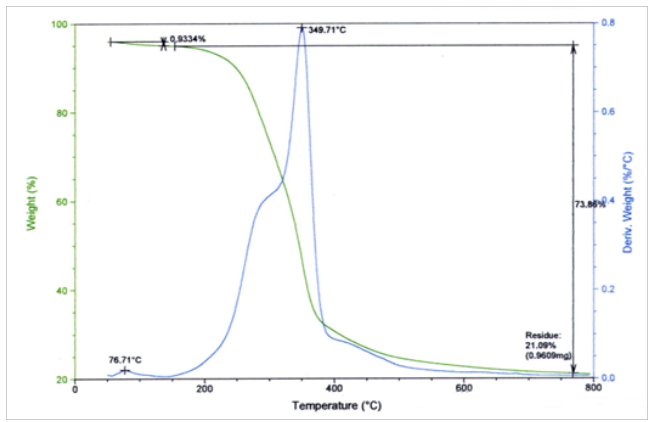
These results indicate that esparto grass contains a more ordered cellulose structure than Diss. (Akerholm 2004).

Thermogravimetric analysis

Thermogravimetric tests in inert atmosphere (N_2) were achieved to evaluate the pyrolytic behavior of biomass fibers. Fig. 5 display the thermogravimetric, TGA (in wt %) and differential thermogravimetric, DTG (in wt % / $^\circ C$) curves for the Diss (a) and Esparto grass (b). The TGA curves show three weight loss steps and their decomposition occurs in two main stages. The initial weight loss, 5.28% for Diss observed around at room temperature to $192.54^\circ C$ (Fig. 5 a) and that 5.05 % for Esparto grass observed around at room temperature to $180.22^\circ C$ (Fig. 5 b), is attributed to the evaporation of the moisture and of different low molecular compounds from the biomass fibers samples.



(a)



(b)

Fig. 5: TGA and DTG of Diss (a) and Esparto grass (b).

According to Tab. 4, the first stage thermal degradation temperature, T_1 around 192.54°C to 280.12°C with weight loss 23.57% of the Diss and T_1 around 180.22°C to 300.04°C with weight loss 33.08% of the Esparto grass is attributed to hemicelluloses. The second stage thermal degradation temperature, T_2 around 280.12 °C to 390.01°C, corresponds to 47.29% of weight loss of cellulose and lignin present in the Diss and T_2 around 300.04°C to 400.01°C, corresponds to 40.78% of weight loss of cellulose and lignin present in the Esparto grass.

Tab. 4: TGA Results of Diss and Esparto grass .

Samples fibers	1 st stage degradation in TGA curve		2 nd stage degradation in TGA curve		Residual mass % at 800°C (%/ °C)
	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
Esparto grass	180.22 to 300.04	33.08	300.04 to 400.01	40.78	21.09

The weight loss, 23.86% for the Diss observed after 390.01°C up to 800°C, and 21.09 % for Esparto grass observed after 400.01°C up to 800 °C is due to the degradation of lignin. In a recent study, Mazlan et al. (2015) showed that the depolymerization of hemicellulose occurs between 180 and 350°C, the random cleavage of the glycosidic linkage of cellulose between 275 and 350°C and the degradation of lignin between 250 and 500°C. The higher activity of hemicelluloses in thermal decomposition might be attributed to its chemical structure. Hemicellulose has a random amorphous structure, and it is easily hydrolyzed (John and Thomas, 2008). In contrast, the cellulose molecule is a very long polymer of glucose units, and its crystalline regions improve the thermal stability of lignocellulosic fibers (Yang 2006). Lignin is different from hemicellulose and cellulose, because it is composed of three kinds of benzene-propane units, being heavily cross-linked and having very high molecular weight (John and Thomas 2008 and Yang 2006). The thermal stability of lignin is thus very high, and it is difficult to decompose (Bridgwater, 2012).

These results are confirmed by DTG curves of Diss and Esparto grass fibers (Fig. 5) where are shown three main peaks. The first peak appear at 78.22°C in Diss curve and at 76.71°C in Esparto grass curve corresponds to evaporation of moisture and different low molecular compounds. The second peak observed at 330.05°C in Diss curve and at 349.71°C in Esparto grass curve assigned to cellulose decomposition. A hemicelluloses shoulder at 280°C for the Diss and 320°C for the Esparto grass. The last peak appears around 475°C in curve obtained with sample of Diss and Esparto grass attributed to lignin decomposition.

The DTG curve for Esparto grass was showed the higher temperatures (349.71°C) of cellulose decomposition than that of the Diss (330.05°C). According to Kim et al. (2010b) this behavior suggests that higher crystallite size celluloses.

SEM image analysis

The cross sectional and longitudinal views of morphology of Diss and Esparto-grass are depicted in Figs. 6 a. and b.

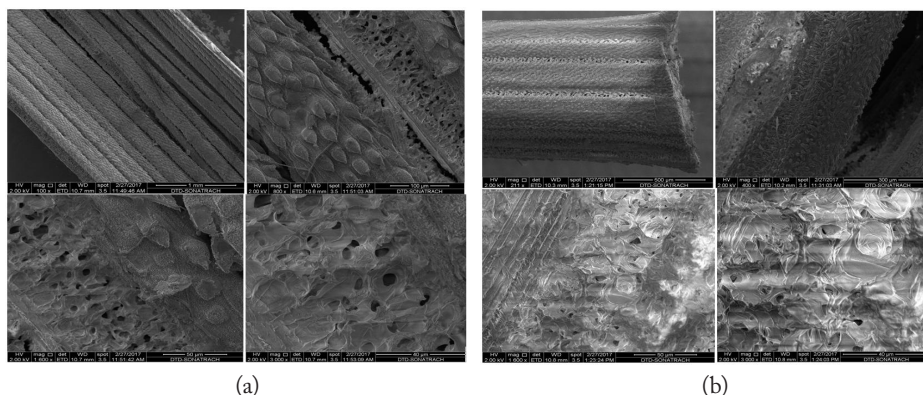


Fig. 6: SEM images of Diss (a) and Esparto-grass (b).

The samples showed some irregularities and defects at the surface of the fibers, which does not allow observation of fibrillar structure of the material in longitudinal view. However, in cross section we see that Diss and Esparto grass have a circular and fibrillar structure, as well as a highly porous and considerably more uniform and isotropic structure throughout. However, some lignin or lignin carbohydrate complexes may be condensed on the surface of cellulose fibers. (Zhu et al. 2009 Dhanalakshmi 2014).

CONCLUSIONS

The methods used to characterize the Diss and Esparto grass studied was found to be appropriate to evaluate differences in the structures of the fiber components. The analysis by SEM, TAG-TDG, XRD, FTIR spectroscopy and chemical method showed differences amongst the Diss and Esparto-grass. The FTIR spectroscopy analysis indicated that the higher extractive contents in Diss and in Esparto grass fiber might be associated with the prominent bands at around 2920 and 2850 cm^{-1} . The X-ray diffractometry results showed that esparto-grass contain much more cellulose chains in a highly organized form with a higher crystal size than Diss, which can lead to higher crystallinity. Also the Scanning Electron Microscopy (SEM) shows a network structure where the fibrils are bound together by hemicelluloses and lignin. It clearly shows the presence of longitudinally oriented unit cells with almost parallel orientations. The result of the thermogravimetric analysis in order to determine the α -cellulose and hemicellulose amounts in Diss and esparto grass samples successfully correlated between the component contents issued from chemical extraction methods. Conducted chemical component analysis showed that Diss and Esparto grass contain a high amount of cellulose and α -cellulose, which justifies their use as a source of fibers in several industry. It could be concluded from the obtained results, that the Diss as the Esparto grass represent an alternative non-wood raw material.

REFERENCES

1. Augustine, O., Ayeni, O. A., Adeeyo, O. M., Oresegun, T. E., 2015: Compositional analysis of lignocellulosic materials: Evaluation of an economically viable method suitable for woody and non-woody biomass, American Journal of Engineering Research 4(4): 14-19.
2. Ahmadzadeh, A., Zakaria, S., Rashid, R., 2009: Liquefaction of oil palm empty fruit bunch (EFB) into phenol and characterization of phenolated EFB, Industrial Crops and Products 30(1): 54-58.
3. Akkiche, O., Messaoud, B. K. 2007: Esparto grass (*Stipa tenacissima*), raw material of papermaking. First part, Chimia rastitelnoy syria 4: 25-30.
4. Åkerholm, M., Hinterstoisser, B., Salmén, L., 2004: Characterization of the crystalline structure of cellulose using static and dynamic FT-IR spectroscopy, Carbohydr. Res. 339: 569-578.
5. Ali, I.S., Sapuan S.M., Zainudin, E.S., Abdan, K., 2009: Kenaf fibres as reinforced for polymeric composites: A review, International Journal of Mechanical and Materials Engineering 4: 239-248.
6. Belgacem, M.N., Gandini, A., 2008 : Monomers polymers and composites from renewable resources. Elsevier, Amsterdam, 4 pp.
7. Blasi, C. D., Signorelli, G., Di Russo, C., Rea, G., 1999: Product distribution from pyrolysis of wood and agricultural residues, Industrial Engineering Chemistry Research 38(6): 2216-2224.
8. Bourahli, M. E. H., 2014: Caractérisation d'un composite verre/époxy. Thèse de magister. Université Ferhat Abbas, Setif, Algérie.
9. Bridgwater, A. V., 2012: Review of fast pyrolysis of biomass and product upgrading, Biomass and Bioenergy 38, 68-91.

10. Chen, H., Ferrari, C., Angiuli, M., Yao, J., Raspi, C., Bramanti, E., 2010: Qualitative and quantitative analysis of wood samples by Fourier transform infrared spectroscopy and multivariate analysis, *Carbohydrate Polymers* 82(3): 772-778.
11. Cordeiro, O.N., Belgacem, M.N., Torres, I. C., Moura, J. C., 2004: Chemical composition and pulping of banan pseudo-streams, *International journal crops and products* 19: 147-154 .
12. Del Río, J. C., Marques, G. , Lino, A. G. , Lima, C. F. , Colodette, J. L., Gutiérrez, A., 2015: Lipophilic phytochemicals from sugarcane bagasse and straw, *Industrial Crops and Products* 77: 992-1000 .
13. Desvaux, M., 2005: *Clostridium cellulolyticum*: model organism of mesophilic cellulolytic clostridia. *FEMS Microbiol Rev* 29: 741-764.
14. Dhanalakshmi, S. P. , Ramadevi, B., Basavaraju, P, R., Raghu, C. V., 2015: Srinivasa, Physical characterization of natural lignocellulosic single areca fiber, *Ciência and Tecnologia dos Materialis* 27: 121-135.
15. Fengel, D., Wegener, G., 1984: *Wood chemistry: Ultrastructure, Reactions*. Walter de Gruyter, Berlin and New York, 613 pp.
16. Foyle, T., Jennings, L., Mulcahy, P., 2007: Compositional analysis of lignocellulosic materials: Evaluation of methods used for sugar analysis of waste paper and straw, *Bioresource Technology* 98: 3026-3036.
17. Gümüşkaya, E., Usta, M., Kirei, H. , 2003: The effects of various pulping conditions on crystalline structure of cellulose in cotton linters, *Polymer Degradation and Stability* 81: 559-564
18. Hurter, A.M., 1988: Utilisation of annual plants and agricultural residues for the production of pulp and paper. *Proceedings of TAPPI pulping conference 1988*. New Orleans, LA, USA Book 1, Pp 139-160.
19. Ishida, Y., Goto, K., Yokoi, H., Tsuge, S., Ohtani, H., Sonoda, T., Ona, T., 2007: Direct analysis of phenolic extractives in wood by thermochemolysis-gas chromatography in the presence of tetrabutylammonium hydroxide, *J. Anal. Appl. Pyrolysis* 78: 200-206.
20. John, M. J., Thomas, S., 2008: Biofibres and biocomposites, *Carbohydr. Polym.* 71: 343-364
21. Katri Sajjonkari-Pahkala, K., 2001: Non-wood plants as raw material for pulp and paper. *Thesis Faculty Agricultural and Forest, University of Helsinki in Finland*, 101 pp.
22. Kim, U.J., Eom, S.H., Wada, M., 2010: Thermal decomposition of native cellulose: Influence on crystallite size, *Polymer Degradation and Stability* 95(5): 778-781.
23. Khalil, A., Bhat, H. P. S., Ireana Yusra, A. F., 2012: Green composites from sustainable cellulose nanofibrils: A review, *Carbohydrate Polymers* 87: 963-979.
24. Khan, A., Huq, T., Khan, R.A., Riedl, B., Lacroix, M., 2014: .Nanocellulose-based composites and bioactive agents for food packaging, *Critical Reviews in Food Science and Nutrition* 54 (2): 163-174.
25. Kim, U. J., Eom, S.H., Wada, M., 2010b: Thermal decomposition of native cellulose: influence on crystallite size, *Polym. Degrad. Stab.* 95: 778-781.
26. Klemm, D., Heublein, B., Fink, H.P., Bohn, A., 2005: Cellulose: Fascinating biopolymer and sustainable raw material, *Angew. Chem. Int. Ed.* 44: 3358-3393.
27. Kondo, T., Sawatari, C., 1996: A Fourier transform infra-red spectroscopic analysis of the character of hydrogen bonds in amorphous cellulose, *Polymer* 37: 393-399.
28. Laureano-Perez, L., Teymouri, F., Alizadeh, H., Dale, B.E., 2005: Understanding factors that limit enzymatic hydrolysis of biomass: Characterization of pretreated corn stover, *Applied Biochemistry and Biotechnology* 121-124: 1081-1099.

29. Mazlan, M. A. F., Uemura, Y., Osman, N. B., Yusup, S. , 2015: Characterizations of bio-char from fast pyrolysis of meranti wood sawdust, *Journal of Physics: Conference Series* 622.
30. Mc Manus, W. R., Robinson, V.N.E., Grout, L.L., 1977: The physical distribution of mineral material on forage plant cell walls, *Australian Journal of Agricultural Research* 28: 651–662.
31. Merzoud, M. ,2007 : Elaboration et caractérisation d'un matériau composite à base de fibres de diss dans la fabrication de la maçonnerie. Thèse de doctorat d'Etat, Université Badji Mokhtar, Algérie.
32. Mészáros, E., Jakab, E., Várhegyi, G., 2007: TG/MS, Py-GC/MS and THM-GC/MS study of the composition and thermal behavior of extractive components of *Robinia pseudoacacia*, *J. Anal. Appl. Pyrolysis* 79: 61-70.
33. Mohanty, A.K., Misra, M., Hinrichsen, G., 2000: Biofibres, biodegradable polymers and biocomposites: An overview, *Macromol. Mater. Eng.* 276-277(1): 1-24.
34. Moon, R.J., Martini, A., Nairn, J., Simonsen, J., Youngblood, J., 2011: Cellulose nanomaterials review: Structure, properties and nanocomposites, *Chem. Soc. Rev.* 40:
35. Ouensanga, A., 1089 : Variation of fiber composition in sugar cane stalks. *Journal of the society of wood science and technology* 21(2): 105 – 111.
36. Paiva, M.C., Ammar, I., Campos, A.R., Cheikh, R.B., Cunha, A.M., 2006: Alfa fibres: Mechanical, morphological and interfacial characterization, *Composites Science and Technology* 67: 1132-1138.
37. Plazonić, I., Mikočević, Ž. B., Antonović, A , 2016 : Chemical composition of straw as an alternative material to wood raw material in fiber isolation, *Drvna industrija* 67 (2): 119-125.
38. Poletto, M., Ornaghi Junior, H.L., Zattera, A.J., 2014 : Native cellulose: structure, characterization and thermal properties, *Materials* 7: 6105-6119.
39. Popescu, M.C., Popescu, C.M., Lisa, G., Sakata, Y., 2011 : Evaluation of morphological and chemical aspects of different wood species by spectroscopy and thermal methods, *J. Mol. Struct.* 988(1): 65-72
40. Pratima, B., 2016: Pretreatment of lignocellulosic biomass for biofuel production, Chapter II Structure of lignocellulosic Biomass. Pp 7-9. Springer Briefs in Green Chemistry for Sustainability.
41. Reddy, M.M., Vivekanandhan, S., Misra, M., Bhatia, S. K., Mohanty, A. K, 2013: Biobased plastics and bionanocomposites: Current status and future opportunities. *Progress in Polymer Science* 38: 1653-1689.
42. Run Cang, S., 2010: Cereal straw as a resource for sustainable biomaterials and biofuels, ed. Elsevier, 289 pp
43. Saha, B.C., 2003: Hemicellulose bioconversion. *J Ind. Microbiol. Biotechnol.* 30: 279–279.
44. Sellami, A., 2015 : Elaboration des composites cimentaires à base de fibres végétales locales (le diss) : Caractérisation, Durabilité et Application au cas de la maçonnerie, Thèse de magister. universitebadjimokhtar – annaba, Algérie.
45. Sluiter, A., Hames, B., Ruiz, R., Scarlata, C., Sluiter, J., Templeton, D. , Crocker, D., 2008: Determination of structural carbohydrates and lignin in biomass, *Laboratory analytical procedure* 1617: 1-16 .
46. Sorek, N., Yeats, T.H., Szemenyei, H., Youngs, H., Somerville, C.R., 2014: The implications of lignocellulosic biomass chemical composition for the production of advanced biofuels, *BioScience* 64: 192-201.
47. Sridach, W., 2010: The environmentally benign pulping process of non-wood fibers, *Suranaree Journal of Science & Technology* 17(2): 105-123.

48. Run Cang, S., 2010: Cereal straw as a resource for sustainable biomaterials and biofuels, ed. Elsevier, 289 pp.
49. Thomas, S., Pothan, L., 2009: Cellulose fiber reinforced polymer composites. Old City Publishing, Philadelphia, 539 pp.
50. Wada, M., Okano, T., 2001: Localization of I α and I β phases in algal cellulose revealed by acid treatments, Cellulose 8: 183–188.
51. Yang, H., Yan, R., Chen, H., Zheng, C., Lee, D.H., 2007: Characteristics of hemicellulose, cellulose and lignin pyrolysis, Fuel 86: 1781-1788.
52. Yokoi, H., Nakase, T., Goto, K., Ishida, Y., Ohtani, H., Tsuge, S., Sonoda, T., Ona, T., 2003: Rapid characterization of wood extractives in wood by thermal desorption-gas chromatography in the presence of tetramethylammonium acetate, J. Anal. Appl. Pyrolysis 67: 191–200.
53. Zablackis, E., Huang, J., Müllerz, B., Darvill, A. C., Albersheim, P., 1995: Characterization of the cell-wall polysaccharides of *Arabidopsis thaliana* leaves, Plant Physiol 107(4): 1129-1138.
54. Zhu, Z., Sathitsuksanoh, N., Vinzant, T., Schell, D. J., McMillan, J.D., Zhang, Y.H.P., 2009: Comparative study of corn stover pretreated by dilute acid and cellulose solvent-based lignocellulose fractionation: enzymatic hydrolysis, supramolecular structure, and substrate accessibility, Biotechnology and Bioengineering 103: 715–724.

CHENAH MAY, AMRANI MOUSSA*

M'HAMEDBOUGARA UNIVERSITY

FACULTY OF SCIENCE

LABORATORY OF SOFT TECHNOLOGY, RECOVERING, AND SUSTAINABLE DEVELOPMENT

M'HAMEDBOUGARA UNIVERSITY 35000

BOUMERDES

ALGERIA

*Corresponding Author: moussaamrani@yahoo.fr

PHONE: 213 0560082355

