Journal of Applied Sciences

ISSN 1812-5654

Journal of Applied Sciences 6 (11): 2450-2455, 2006
ISSN 1812-5654

Physical and Mechanical Characterization of Technical Esparto (*Alfa*) Fibres

[1]L. Ghali, [1]M. Zidi and [2]S. Roudesli
[1]Laboratory of Mechanical Engineering, National School of Engineering of Monastir, Tunisia
[2]Laboratory of Polymers Bio Polymers Organic Materials, Faculty of Science of Monastir, Tunisia

Abstract: The aim of this study is to give scientific data about the physical, morphological and mechanical proprieties of esparto fibres. The use of esparto fibres for reinforcing thermoplastics is a novel way. That is why a better investigation of their proprieties and surface characteristics is necessary to maximize their potential use. In this study, different methods of extraction were conducted on the fibres using mechanical separation (At1), aqueous solution of NaOH 3N (At2) and chemical modification by acetylation (At3). Esparto fibres were characterized through technical analysis such as SEM and FTIR. The result of microscopic analysis shows that esparto fibres have a similar structure to that of a composite material in which the ultimate fibres of cellulose constitute the reinforcement. The pycnometric measurement shows that the density of esparto fibres is similar to those of jute and *Agave americana* L. The FTIR spectra of various esparto fibres show a decrease of the number of carboxyl groups after (At2) fibres acetylating. This result proves the contribution of the chemical modifications for the hydrophobic improvement of the proprieties of fibres. In addition a comparison between properties of esparto fibres and other vegetal ones has been integrated. The results of this study indicate that technical esparto fibres can have promising properties especially in reinforcement of composite materials

Key words: Esparto fibre, extraction, characterization, physical properties, mechanical properties

INTRODUCTION

A critical discussion about the preservation of natural resources and recycling has recently renewed the interest in natural materials especially renewable raw materials. Natural fibres derived from varied natural resources, have special characteristics such as biodegradability, recycling, low density and good mechanical resistance that can be used in a diversity of applications.

Cellulose fibres, for instance, played a significant role in the human history. They were often used for clothing and construction materials (Roger *et al*., 2000). Nowadays, a renaissance in the use of natural fibres, as a reinforcement of technical applications, is taking place mainly in the automotive and construction industries. In fact, Joseph *et al*. (1999) showed that sisal fibres permit to have a composite of good properties. This composite material has interesting implications for the automotive and transport industries. In fact Bledzki and Gassan (1999) studied the influence of the treatments of vegetal fibres on their physical and mechanical properties in order to use them for composite materials. Herrara-Franco *et al*. (2004) studied the mechanical properties of the composites reinforced by continuous natural fibres (long fibres). Abdelmouleh *et al*. (2004) studied the effect of the cellulose fibre modification by organosilanes on the fibre-matrix adhesion.

Li *et al*. (2000) studied the properties of fibres of sisal and the properties of composites reinforced by these fibres. Properties of the hybrid composites with reinforcement of sisal and glass fibre were also reported. Viviana *et al*. (2004) studied the influence of chemical treatments on mechanical properties of bio composite of sisal fibre.

In spite of the various applications of these natural fibres, the fields of application of some remain limited. The esparto (*Stippa tenacissima* or *alfa*) particularly can provide long fibres belonging to the family of "hard" natural fibres which can be used in diverse technical applications. Han (1998) showed that the ultimate fibres from esparto grass are lignocellulosic fibres ranging from 0.2 to 3 mm in length and between 6 and 22 μm in diameter. These fibres are mainly used for the production of paper pulp.

The purpose of this study is to report the proprieties of technical esparto fibres, which are long fibres extracted from leaves of esparto grass. The esparto (*alfa*) is a

Corresponding Author: M. Zidi, Ecole Nationale d'Ingénieurs de Monastir, Département Génie Mécanique, Avenue Ibn Eljazzar 5019 Monastir, Tunisie Tel: 00 216 98 93 90 24 Fax: 00 216 73500514

monocotylic plant belonging to the family of graminaceous in which leaves source of lignocellulosic consist of approximately 33-38% of cellulose, 21% of lignin, 27-32% of hemicelluloses and 6-8% of ash (Han, 1998).

MATERIALS AND METHODS

Materials: The raw material used in this study is collected from Tunisia during the summer season when the leaves of esparto reach their maximum state of growth. The fibres operated are extracted from the esparto leaves by mechanical and chemical processes.

Mechanical extraction: The fibres obtained by simple mechanical separation are indicated by (At1) or gross fibres. They are extracted from the leaves by scraping. In the first time, the esparto leaves were steeped in water during 24 h and calendared. In the second time, the residue is dried to the ambient air for 8 h and brushed by a metallic brush.

Chemical attack: The mechanically extracted fibres of esparto (At1) are immersed in a soda solution (NaOH, 3N) during two hours at a temperature of 100°C and under backward flow. The esparto fibres are then picked up and rinsed 4 to 5 times with water in order to eliminate excess of soda. The residue obtained is then bleached using a 40% diluted sodium hypochlorite solution during one hour in ambient temperature. Finally, the sample is rinsed with water several times and then dried at 60°C during 8 h. The fibres obtained by this process are indicated by (At2).

Acetylation of (At2) fibres: The fibres (At3) are obtained by acetylation treatment of fibres (At2). Then the alkali-treated fibres were soaked in glacial acetic acid for 1 h. Fibres were separated from solution and soaked in acetic anhydride containing two drops of concentrated H_2SO_4 for 2 min (Manikandan *et al*., 2001; Paul *et al*., 1997). These fibres were then taken out, washed well with water and dried in an oven at 80°C for 6 h.

Test methods: The esparto fibres obtained from the three techniques mentioned above are characterized by means of physical and mechanical analysis in order to define their properties.

The measurement of linear density of esparto fibres, obtained by the different processes, is described according to the French standards NF G 07-007(1983). The test was carried out on a batch of conditioned fibres to a normal atmosphere of relative humidity of 65±2% and a temperature of 20±2°C. The density of the esparto fibres was determined by the method relating to the French standards NF T20-053 while using the carbon tetrachloride (CCl_4) as reference solution (d = 1.595). The moisture regain was also determined with reference to the French standards NF G 08-001.

The specimens of esparto fibres were covered with gold and observed through scanning electron microscopy using a Phillips scanning electron microscope. The infrared spectra of all esparto fibres varieties were carried out using a Fourier Transform spectrophotometer Nicolet 510M.

The tensile tests were performed using an LLOYD universal testing machine according to the requirements of the French standard NF G 07-002.

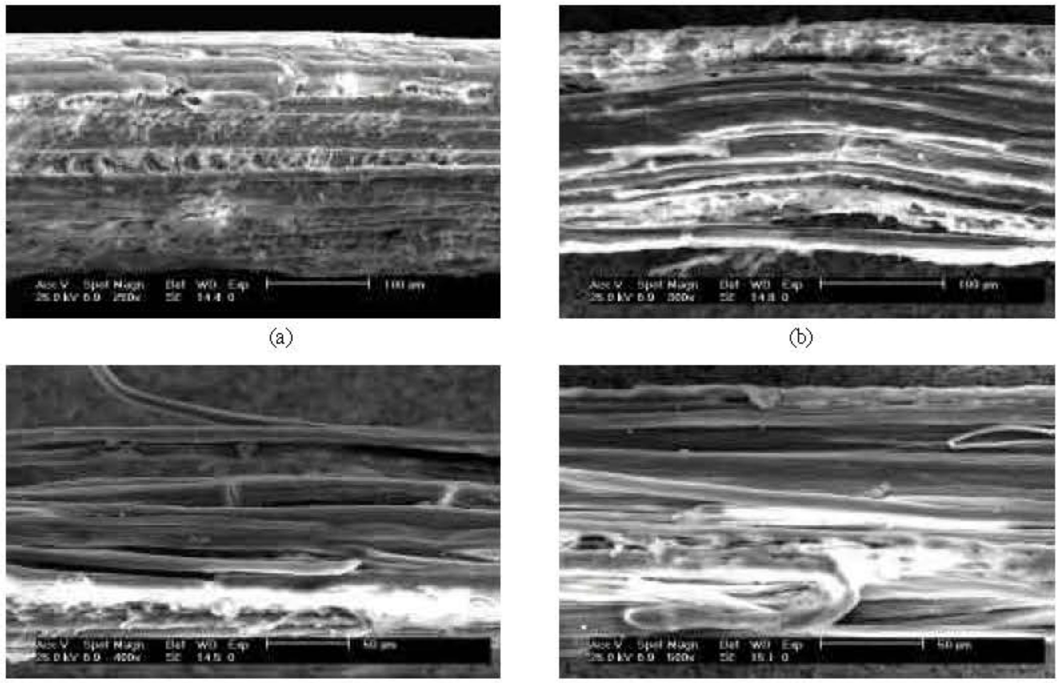
RESULTS AND DISCUSSION

Physical properties of esparto fibres: The physical characterization of esparto fibres obtained by the various processes described above is limited to the morphological properties, linear density, density and moisture regain. These various tests are carried out according to international standards in order to compare the results obtained for esparto fibres with those of other vegetal fibres, studied already.

Structure of fibres: Observations using a scanning electron microscope on longitudinal sections of the multiple esparto fibres varieties were carried out. These observations showed that the fibres At1, At2 and At3 have similar structures but their surface aspects differ according to treatments undergone by the fibres. Figure 1 illustrates the morphological structures of esparto fibres (At1, At2 and At3). Figure 1c shows that the esparto fibres At2 present a similar structure to that of a natural composite of which ultimate cellulose fibres constitute the fibrous reinforcement while the woody and gum substances constitute the matrix.

The comparison of Fig. 1a and b shows that the chemical treatment improves the aspect of surface of esparto fibres and eliminates the gummy and waxy substances persisting on rough fibres (At1). Then the alkali treatment removes the hemicelluloses and lignins components of (At1) fibres (Bal *et al*., 2004). The analysis of Fig. 1d shows the existence of the ramifications on acetylated esparto fibres.

Figure 1 shows that the ultimate cellulose fibres have a cylindrical structure whose diameter lies between 6 and 22 μm and length ranging from 0.2 to 3 mm (Han, 1998).



(a) (b) (c) (d)

Fig. 1: Esparto fibres observed by SEM (a) At1 fibre (b, c) At2 fibre and (d) At3 fibre

The analysis of this figure also shows that the cellulose fibres are glued by a substance (lignin) which resists to the chemical extraction treatments. The ultimate packages of fibres are present in the form of a structure perfectly directed in length. This result confirms the works of Thielemans *et al.* (2004) which showed that the hard fibres, are presented in beams ultimate fibres that are tied up by gummy and waxy substances.

Density, linear density and moisture regain: Among the properties which characterize the vegetal fibres and their behaviour are smoothness, density and moisture absorption. These properties highlight the relationship between the morphology of fibre, its chemical composition and its behaviour towards the physical and mechanical aggressions.

However, the complex structure of fibres and the difficulty of identifying their geometry pose a problem in determining the esparto fibre fineness. So the fineness of technical fibres is not evaluated by means of diameter measurements but by means of linear density or title measurement. (title(tex) = 1000mass(g)/length(m))

The measures of linear density of different batches of fibres show medium values ranging from 13.31 tex for fibres (At2) to 21.95 tex for fibres (At1). We also note that the chemical modification by acetylation has an influence on the fineness of the esparto fibre. In fact, their title increases from 13.31 tex to 19, 71 tex, respectively for fibres (At2) and (At3). This title increase is due to the weight increase after grafting by introduction of the acetyls groups.

Table 1: Densities and linear densities of esparto fibres

		At1	At2	At3
Density (g cm^{-3})		2.142	1.350	1.531
Linear density	Average (tex)	21.95	13.31	19.71
	Number of samples	100	100	100
	Standard deviation	2.78	1.3	2.6

Another important characteristics of the vegetal fibres is their lightness. This characteristic is expressed by a low density compared to other used fibres. Table 1 indicates the results of densities and linear densities obtained for the different varieties of esparto fibres.

We notice that the chemical modification by acetylation of these esparto fibres (At2) increases their density up to 1.53 g cm^{-3}. This is due to the introduction of the acetyls groups into the reactive sites of esparto fibre, which involves an increase in the density of modified fibre. Some techniques such as Fourier Transform Infrared spectrophotometry (FTIR) as well as the techniques of measurement of the contact angle

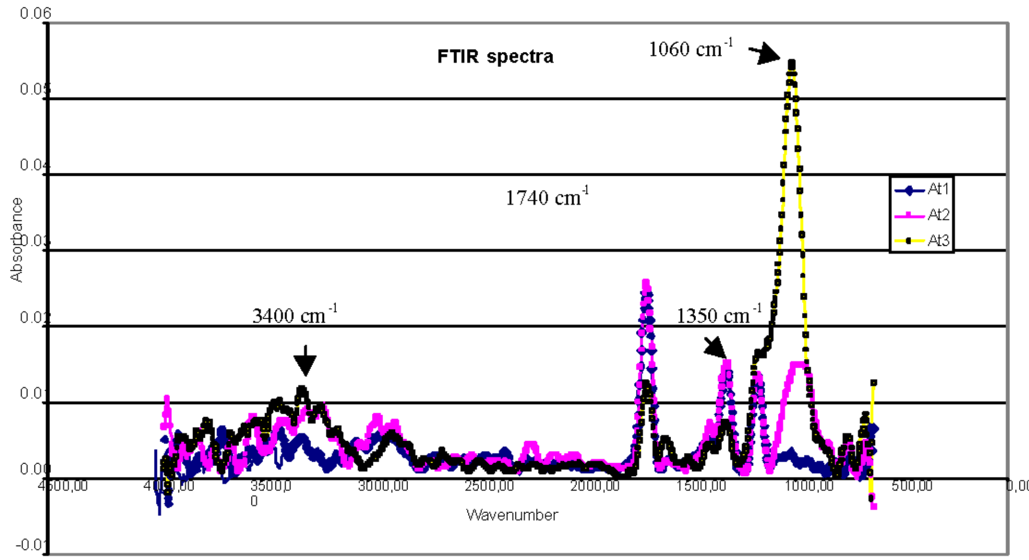


Fig. 2: FTIR spectra of esparto fibres

enable us to visualize the chemical modification of fibre and justify the preceding results. The esparto fibres, as cellulose fibres, have an absorbent feature that is defined by their capacity of moisture absorption. This feature is evaluated by the measurement of moisture regain which informs about the quantity of water that the fibre can contain under well defined conditions of temperature and moisture. The experimental result of moisture regain, for esparto fibres (At2), is: R = 8.84%. The esparto fibres show a moisture regain similar to that of sisal and slightly higher than that of the flax. Consequently, the use of these fibres as thermoplastic reinforcement requires pre-treatment of their surfaces in order to improve the adhesion between the fibre and the matrix.

The esparto fibres showed a light and hydrophilic composite structure. These physical properties are going to dictate the nature of the mechanical behaviour of the fibre after a tensile test.

FTIR characterization: A comparison between the FTIR spectra of the At2 fibre and that of At1 fibre (Fig. 2) shows that the intensity of the peak at 3400 cm^{-1} increased. This increase is due to the formation of more hydroxyl groups resulting from the rupture of bonds that develops lignin and hemicelluloses with cellulose after alkali treatment.

As shown in Fig. 2 the treatment of At2 esparto fibres with acetic acid led to the appearance of the increment of the absorbance in the region 1730-1740 and 1050-1210 cm^{-1} (Tserki *et al*., 2005; Valcineide *et al*., 2005). The peak at around 1740 cm^{-1} for the treated fibres (At3) indicates that the acetyl groups are involved in an ester bond with the hydroxyl groups of the fibres. The esterification reaction is also confirmed by the appearance of a new peak in 1060 and 1210 cm^{-1}.

Alkali treatment before acetylation may destroy the hydrogen bonding in cellulosic hydroxyl groups and cause partial removal of lignin, thereby making them more reactive. After acetylation, the hydrophilic nature of the fibre is decreased (Valdez-Gonsalez *et al*., 1999).

Mechanical properties: The mechanical tests on the esparto fibres allow us to determine their behaviour and their resistance to simple or compound requests. In fact, several tests were made to characterize the behaviour of the esparto fibres.

The materials' properties generally, depend, on their morphologic structures and on their chemical compositions. While taking into account the variability of the structure and the physical properties examined in a batch of esparto fibres, the characteristics of resistance such as the initial module, the load of break and the elongation of break, present a high coefficient of variation.

Figure 3 shows the evolution of the specific load (F (cN)/title (Tex)) according to the elongation during tensile tests realized for three esparto fibres (At1), (At2) and (At3). A careful examination has indicated that the curves describing the behaviour of the esparto fibres are characterized by two zones: the first one describes a reversible behaviour or an elastic behaviour of which the slope to the origin defines the initial module and the second zone describing an irreversible behaviour which ends by a fragile break of the fibrous material.

Table 2: Mechanical properties of esparto fibres

	At1	At2 Average	At2 No. of sample	At2 Standard deviation	At3
Elongation (%)	2.84	4.08	50	0.88	3.8
Tenacity (cN/tex)	28.06	19.6	50	3.16	18.5
Initial module (N/tex)	10.12	8.29	50	1.38	6.8

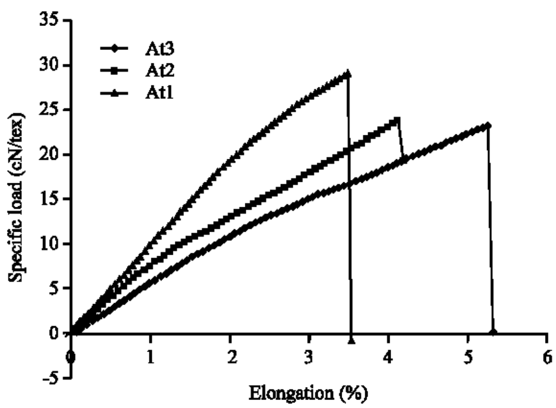


Fig. 3: Tensile curve of the various types of esparto fibres

The experimental results obtained by the series of tests made on fibres (At1), (At2) and (At3) are presented in Table 2. The tenacity is defined by the ratio of the maximum loading which the test-tube can support with its linear density whereas the initial module is defined by the slope at the origin of the tensile curve (Fig. 3).

It stands out from Table 2 that the nature of the fibre extraction process influences its mechanical resistance. In fact, the esparto fibres (At1) are more rigid than those extracted by soda (At2). In addition, the chemical modification by acetylation increases the density and the linear density of esparto fibre (At2) but it does not influence considerably its mechanical properties. In fact, the acetyls groups grafted on cellulose have a transverse orientation and do not contribute to the resistance of modified fibre.

Comparison of the esparto fibres with other vegetal fibres: In technical applications such as composites, the contribution of vegetal fibres is reflected in their low density and resistance. The esparto fibres extracted by chemical process (At2) have a lower density than those of vegetal fibres such as the flax, the sisal, cotton and the henequen of which the densities are close to 1.5 g cm^{-3}. They have a low average tenacity compared to those of other vegetal fibres. In order to highlight the importance of fibres obtained from the esparto plant the tenacity/density ratio of various vegetal fibres was carried out.

Table 3: Vegetal fibres compared to the τ/d ratio

Fibre	Density(g cm^{-3})	Tenacity (cN/tex)	τ/d
Cotton	1.5-1.6	26-44	17.33-27.5
Jute	1.3	26-51	20-39.23
Flax	1.5	23-24	15.33-16
Agave americana L.	1.36	28.3	20.8
Sisal	1.5	36-45	24-30
Glass E	2.5	75	30
Esparto fibre (At1)	2.142	28.06	13.09
Esparto fibre (At3)	1.531	18.50	12.08
Esparto fibre (At2)	1.350	19.6	14.51

Table 3 illustrates a comparison between esparto fibres (At1, At2 and At3) and those used in composites on the level of the τ/d ratio; where τ is the tenacity (N/Tex) and d is the density. This comparison shows that the esparto (*Alfa*) fibres have the weakest ratio. The τ/d ratio of the esparto fibre (At2) is close to that of the flax but is definitely lower than that of sisal and glass E. This leads us to optimize processes of extraction in order to get esparto fibres with a low density and a good resistance compared with other fibres such as sisal, agave, glass E etc.

CONCLUSIONS

In this study, we investigation the physical and mechanical properties of fibres extracted from the leaves of *alfa* (esparto). In fact, several techniques of characterization were used such as the scanning electron microscope (SEM), the FTIR spectrophotometer and the tensile test. The studied technical fibres were obtained by mechanical extraction (At1), chemical extraction (At2) as well as the other chemically extracted and acetylated (At3).

The study of the mechanical behaviour of the esparto fibres obtained by various methods of extraction has allowed us to position them with regard to the natural fibres already used in the industry such as flax, sisal and cotton. The treatment of fibre surfaces by various chemical processes can improve their characteristics and make a composite material having industrial performances. The characteristics of fibres/matrix interface can also be improved (Valdez-Gonsalez *et al*., 1999).

REFERENCES

Abdelmouleh, M. *et al*., 2004. Modification of cellulosic fibres with functionalised silanes: Development of surface properties. Intl. J. Adhesion Adhesives, 24: 43-54.

Bal, K.E. *et al*., 2004. Gross morphology and absorption capacity of cell-fibers from the fibrous vascular system of loofah (*Luffa cylindrica*). Textile Res. J., 74: 241-247.

Bledzki and Gassan, 1999. Composites reinforced with cellulose based fibbers. Pro. Polym. Sci., 24: 221-274.

Han, J.S., 1998. Proprieties of non-wood fibres. Fibber Property Comparison, TAPPI 1998 North American Nonwood Symposium Atlanta.

Herrara-Franco, P.J. *et al*., 2004. Mechanical properties of continuous natural fibre-reinforced polymer composites. Composites: Part A, 35: 339-345.

Joseph, K. *et al*., 1999. A review of sisal fibre reinforced polymer Composites. Revista Brasileira de Engenharia e Ambiental, 3: 367-379.

Li, Y. *et al*., 2000. Sisal fibre and its composites: A review of recent developments. Comp. Sci. Technol., 60: 2037-2055.

Manikandan, N.K.C., Sabu Thomas and G. Groeninck, 2001. Thermal and dynamic mechanical analysis of polystyrene composites reinforced with short sisal fibres. Comp. Sci. Technol., 61: 2519-2529.

NF, G., 07-007, 1983. Fibres tests-determination of the fibres linear density. Paris: AFNOR, pp: 272.

Paul, A. *et al*., 1997. Effect of surface treatments on the electrical proprieties of low-density polyethylene composites reinforced with short sisal fibres. Comp. Sci. Technol., 57: 67-79.

Roger, R.M. *et al*., 2000. Characterization and factors effecting fibre properties. Natural Polymers and Agrofibers Composites, Sãn Carlos-Brazil.

Thielemans, W. *et al*., 2004. Butyrated kraft lignin as compatibilising agent for fibre reinforced thermoset composites. Composites Part A: Applied Sci. Manufacturing, 35: 328.

Tserki, V. *et al*., 2005. A study of the effect of acetylation and propionylation surface treatments on natural fibres. Composites: Part A, 36: 1110-1118.

Valdez-Gonsalez, A. *et al*., 1999. Effect of fibres treatment on the fibre bond strength of natural fibre reinforced composites. Composites Part B: 30: 309-320.

Valcineide, O.A. *et al*., 2005. A comprehensive characterization of chemically treated Brazilian sponge-gourds (*Luffa cylindrica*). Polym. Testing, 24: 474-482.

Viviana, P.C. *et al*., 2004. Effect of chemical treatment on the mechanical properties of starch-based blends reinforced with sisal fibre. J. Comp. Mat., 38: 1387-1399.