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Abdelhak Maghchiche[1], Abdelkarim Haouam[1] and Barbara Immirzi[2]

EXTRACTION AND CHARACTERIZATION OF ALGERIAN ALFA GRASS SHORT FIBERS (STIPA TENACISSIMA)

[1]Laboratoire de pollution et chimie physique et analytique, Departement de chimie, Faculte des sciences, Universite Mentouri Constantine, 25000 Algeria
[2]Institute of chemistry and technology of polymers ICTP- Pozzuoli (Na) Italy
amaghchiche@yahoo.fr

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Abstract. Alfa fibers are cellulose-based fibers extracted from esparto grass leaves using alkaline procedure to remove noncellulosic substances such as pectin, lignin, and hemicelluloses. Morphological characterizations (length, width) of esparto fibers are analyzed. The cellulose fiber extracted was characterized by infrared spectroscopy, X-ray diffractometry, thermal analysis TG and DSC, optical and scanning electron micrographs (SEM). In this study, we attempted to characterize the esparto fiber obtained from a chemical extraction using sodium hydroxide then sodium hypochlorite as a bleaching agent. The characterization was based on the measurement of the morphological structure.

Keywords: cellulose, esparto grass fiber, fiber extraction, chemical treatment.

1. Introduction

In recent years, a number of bast-extracted fibers, alternative to the most used ones, such as jute, flax, hemp and kenaf, have been also proposed as reinforcement for plant fiber composites: these fibers are mainly from herbaceous plants.

In general, trying to broaden the number of botanical species from which fibers are extracted may present interest. Alfa is the Arab name of esparto grass. *Stipa tenacissima* plant is a hardy perennial grass from the family of mentioned grass. This is an endemic of the western Mediterranean, which grows on the semi arid grounds of North Africa and South Spain. It is constituted of stems with a cylindrical shape which have a maximum height of about 1 m. Fiber differentiation is closely related to the vegetative cycle. Fibers are short and wide at the leaf base (basal level) and grow longer and thinner above the leaf [1].

Alfa stem consists of strong, stiff and light cellulosic fibers which are mostly used in the production of high quality papers and for decoration, cigarettes and dielectric applications. The stem is also traditionally used in manufacturing ropes and carpets [2].

The more recent information estimated the esparto covered surface of approximately 3 million hectares in Algeria [3].

Due to its short fiber length, paper from Alfa retains its bulk and takes block letters well. The esparto grass seems to prefer the calcareous soil, not very deep and permeable, with the texture dominated by a high sand dust rate covered the stem of esparto grass.The quality of cellulose contained in this plant, ***i.e.*** the flexibility, the smoothness and the mechanical resistance imparts required properties for papermaking [4].

In addition to their multiple uses, natural fibers have shown many efficient properties such as resistance or flexibility which give them wide ranges of applications in a textile field. In fact they are recyclable and nature-friendly and nowadays they are exploited in automobile and medical applications.

The fiber wastes from esparto grass offer a certain potential of liquid absorption and may be used as an absorbent fiber in hygienic products even in blends with fuff pulp [5].

In recent years, considerable attention has been given to the development and utilization of natural fibers. The main application of these materials as composites has been directed towards the automotive industries [6]. Composite materials were prepared using unsaturated polyester resins reinforced by Alfa fibres [7]. Esparto grass fiber was evaluated for bleached pulp production [8].

The main purpose of this work is to characterize Alfa fibers in order to use them as reinforcement for structural composites materials. This choice is supported by the multiple advantages of natural fibers: they are available, renewable and biodegradable, and they have a low price and represent an economic interest for the agriculture sector.

To propose their application in composite materials, the question of chemical treatment is crucial: a preliminary study of chemical treatments to improve the properties of composite laminates [9]. As a consequence, a profound need exists for a sounder investigation of the morphological modifications produced by a wide range of chemical treatments on esparto fibers. At the same time this would expose as much as possible the cellulose structure to increase the number of reaction sites at fiber surface [10]. This is achieved by removing as much as possible non-structural matter, *i.e.* hemicelluloses, lignin and pectin [11]. However, alkaline treatment has been recognized capable of regenerating cellulose by addition of hydroxyl groups, dissolving microscopic pits or cracks on the fibres, *e.g*. in a study of kenaf fibres [12].

Some of them, such as alkali-treatment, *e.g.* with sodium hydroxide (NaOH), bleaching with sodium hypochlorite (NaClO) or chlorite ($NaClO_2$), are applied for the fiber bundles trying to obtain the technical fiber, which is the one that affords loading in tension and twisting, to be possibly used in textile products [13].

In North Africa, the esparto grass constitutes an essential element of fight against the turning into a desert and an essential factor of the maintenance of balance pastoral, thanks to its well-developed root system that retains and protects the ground [14].

2. Experimental

2.1. Materials

2.1.1. Plant samples

The raw material having been the subject of our study comes from the Algerian steppe region. Plant materials must be clean and free of extraneous substances including soil and dust particles that may influence analytical results. For analyses of esparto grass we prepared approximately 10 g of finely crushed plant with particles of homogeneous size, sifted on sieve No. 24 and No. 27.

2.1.2. Pretreatment of esparto grass

First of all the stems are being carded mechanically to refine their diameter. Before specific treatments were held, the fiber was first rinsed with distilled water to remove dirt on the fiber surface. Washed esparto fibers were left to dry at room temperature and finally dried in the oven for 5 h at 333 K. Afterwards they are submerged in 35 g/l salt water during 24 h at 333 K or 12 h at 353 K, to dissolve the waxes, a layer on the surface to protect the plant against heat by limiting the evaporation of water.

2.1.3. Extraction of cellulose fibers

Esparto grass stems are treated with chemical products to degrade and eliminate the two main linking components, lignin and pectins. As the objective is to produce fibres, the hemicellulose doesn't need to be eliminated because it sticks the cellulosic filaments together to form fibres. Alfa fibres are cellulose-based fibres extracted from esparto grass. The cellulose was extracted from Alfa plant with 400 ml toluene/ethanol mixture (2/1, v/v**)** for 6 h using Soxhlet apparatus and treated with NaOH (1M) for 8 h at 298 K [15, 16]. After filtration the cellulose was obtained and the filtrate contains the lignin and hemicelluloses. This is mainly due to the reduction of lignin that binds the cellulose fibrils together.

2.2. Analyses Methods

2.2.1. Plant analysis

The concentration of nutrients in plant tissues was measured in a plant extract obtained from fresh plant material .Plant samples were washed in distilled water; dried in the oven at 333 K for 48 h, weighed, and then ground to 0.1 mm before chemical analysis. To determine the organic, mineral and dry matter the elemental analysis was used [17-19].

Esparto grass fibres were characterized by elemental analysis, IR spectroscopy, thermal analysis, optical microscopy, scanning electron microscopy (SEM) and X-ray diffractometry (XRD).

To determine the organic, mineral and dry matter of esparto grass plant we used the elemental analysis.

FT-IR spectra were recorded on Perkin-Elmer Paragon 500 FT-IR spectrophotometer in the range of 4000–400 cm^{-1} using thin film by solution casting *via* air evaporation and KBr pellets for sample preparation.

Thermal analysis was carried out with Mettler TA TC 11 thermal analyzer. Both thermo gravimetric analysis (TGA) and differential scanning calorimetry (DSC) of all samples were performed up to the temperature of 873 K, starting from room temperature in nitrogen atmosphere. A heating rate of 10°/min was maintained in all cases and flow rate of 30 ml/min nitrogen.

Esparto grass was subjected to XRD analysis, the sample was packed into a hole of 2 mm diameter in a small container made of perplex about 1.5 mm thick. PW 1830 diffractometer and P3020 X-ray generator (Phillips, Holland) were used for this study producing CuKα radiation; the scattering angle (2θ) was varied from 283 to 318 K.

SEM micrographs were taken using Philips XL20 (Philips analytical Inc., the Netherlands) Samples was coated by gold before examination (cathode dispersion). Fibers obtained from esparto grass plant composed mainly by cellulose filaments were characterized by SEM.

2.2.2. Morphological characteristics

The dimensions of the fibers, and especially the length, are largely related to the quality of the pulp [20]. A manual method was used employing a microscope equipped with an ocular micrometer.

3. Results and Discussion

3.1. Plant Analysis

After esparto grass extraction and bleaching, the cellulose fibers were obtained (Fig. 1). The fibers are better separated from one another. This causes the release of fibers encrusting substances (lignin, hemicellulose, pectin, *etc*.).

The chemical composition of esparto grass is shown in Table 1. Cellulose is the major component, followed by hemicelluloses and lignin. The smallest components are extractives and ashes. The silicate in esparto grass accounts for 2.03 %. The lignin contents were around 20 wt %.

Fig. 1. Esparto grass after chemical treatment

We note that the fibers contain a high remarkable SiO_2 and CaO. The silica is present in the composition of esparto grass; it constitutes even one of the reasons for which the delignification of this grass is carried out by the alkaline processes, with soda.

Percentages of esparto fibres humidity absorption were found to be 67 % at 298 K.

Table 1

Composition of esparto grass

Composition	% of dry plant
Dry matter	94.25
Organic matter	17.78
Mineral matter	1.22
Extracted with ebullient water	4.06
Crude fiber	28.75
Cellulose rate	33.81
Lignin rate	18.20
Ash content	5.75
Silica	2.03
Moisture	12.30

Note: losses on the ignition is 48.23 % at 1373 K.

Esparto grass ashes contain mineral components which are listed in Table 2.

Table 2

Mineral components of esparto grass ashes

Element	%
SiO_2	32.5
CaO	7.25
MgO	2.40
K_2O	1.32
Na_2O	0.40
P_2O_5	0.60
Fe_2O_3	2.60

3.2. FT-IR Spectra

In FT-IR spectrum of esparto fiber (Fig. 2) a broad absorption band at 3274–3500 cm^{-1} is mainly due to OH-groups in the existing structure of the fibers. We also note the presence of a band at 1050 cm^{-1}, and a second one at 1630 cm^{-1} which indicates the existence of single C–O and double C=O bonds. Wave number 920 cm^{-1} corresponds to the vibrations of H-aliphatic chains.

3.3. Thermal Analyses

Thermal stability and degradation patterns were determined by employing TG and DTA. To examine the thermal stability of esparto grass fibers thermo gravimetric analysis under nitrogen flow was used.

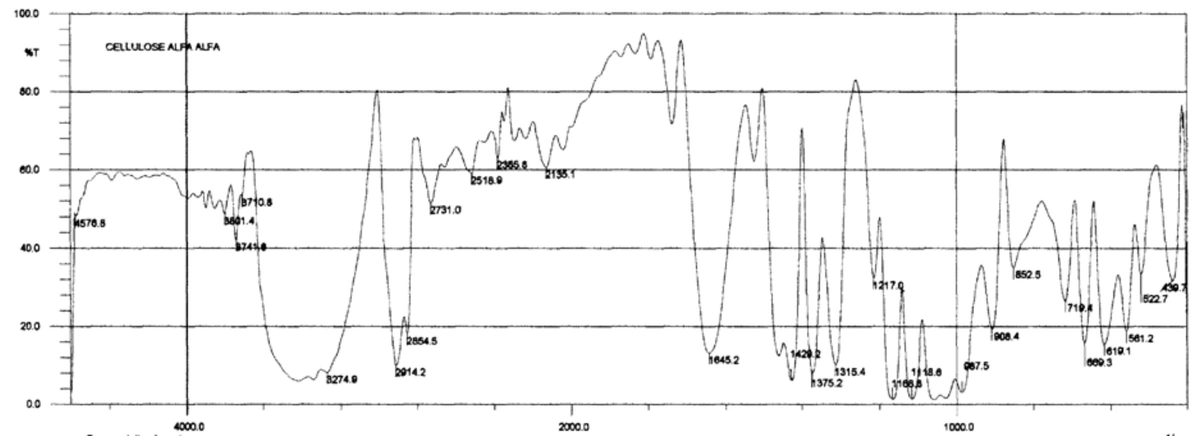

Fig. 2. FTIR spectrum of esparto grass fibers with KBr

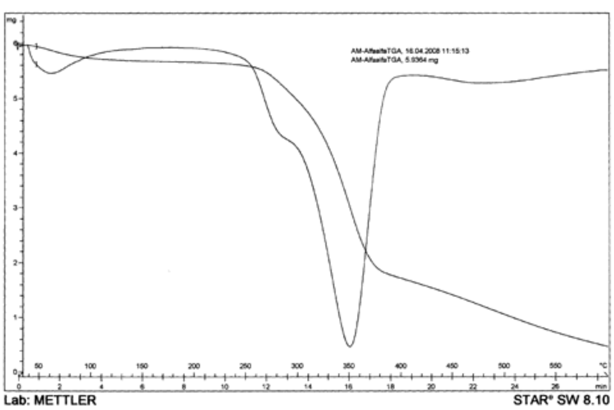

Fig. 3. TGA curve of esparto grass fiber

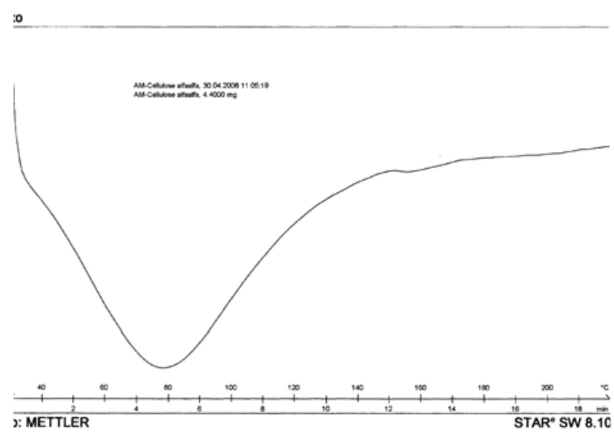

Fig. 4. DSC curves of cellulose Alfa-Alfa

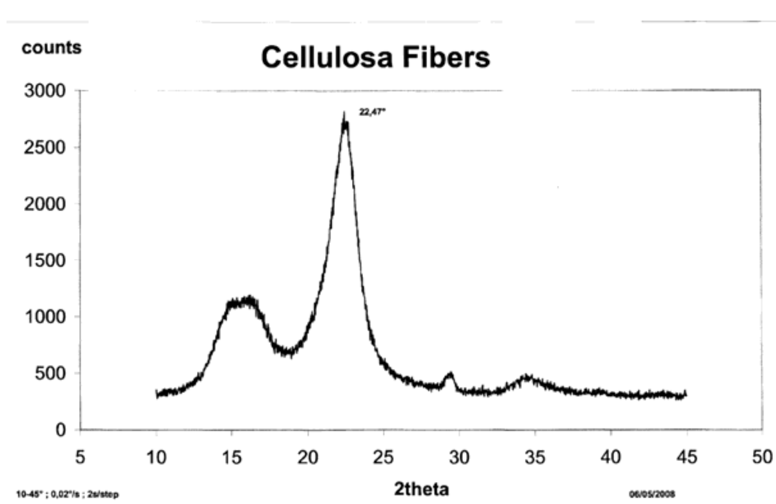

Fig. 5. X-ray diffractograms of esparto grass fiber

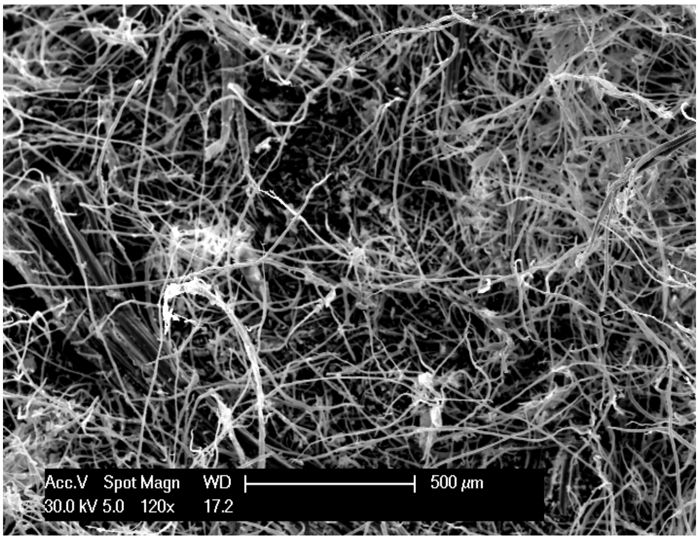

Fig. 6. Scanning electron micrographs of esparto grass fibers

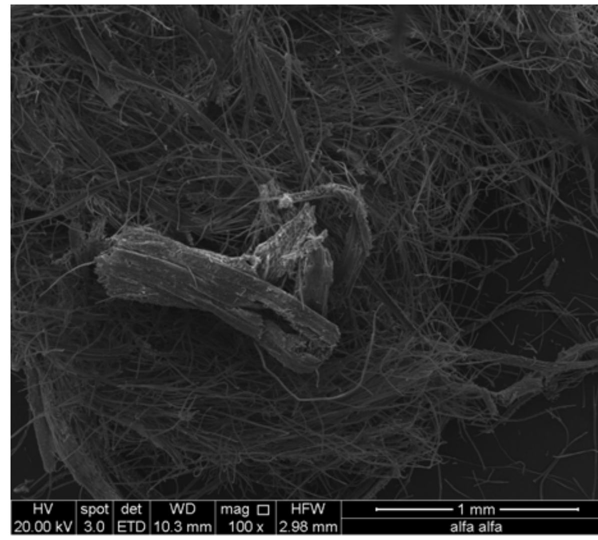

Fig. 7. SEM micrographs of extracted esparto grass fiber

Initial weight loss at 343 K is due to the presence of small amount of moisture in the sample, the second loss is due to the loss of CO_2 and the rate of weight loss increased with the increase in temperature till degradation at 623 K.

Thermal degradation was studied by differential scanning calorimetry under nitrogen flow. Cellulose shows one big endothermic curve at 353 K; caused by evolution of water entrapped by OH-groups present in cellulosic chains.

The cellulose decomposition takes place at the temperatures higher than 473 K.

3.4. X-Ray Analysis

Fig. 5 shows a diffractogram of esparto grass fibers, in which one can observe that esparto grass fibers extracted *via* the physico-chemical process have a very similar diffraction pattern. Crystalline peak appears at 22.47°. It is understandable that the cellulose content increases, whereas the amorphous hemicellulose content decreases during the physico-chemical process [21-22]. This is in agreement with FT-IR and chemical analyses.

3.5. Morphological Characteristics

The average dimensional specifications of the fibers sample which we had analyzed are summarized as follows:

Length	1.47 mm
Outside diameter *D*	0.015 mm

Esparto fibers are short with thick walls.

3.6. Scanning Electron Micrographs (SEM)

The morphology of the esparto grass fibers was investigated by SEM. Figs. 6 and 7 demonstrate that fibers are totally separated by chemical treatment.

4. Conclusions

The aim of this study was to extract cellulosic fibers from esparto grass (*Stipa Tenacissima L.*) for being used in different applications. This material is renewable, biodegradable and very ecological. In fact, it requires very small amount of water to grow and neither insecticides nor pesticides are needed.

To produce ultimate fibers, we must look for an appropriate method of extraction. In this paper, a method of extraction that gives cellulosic fibers without any damage was investigated, structure of technical esparto grass fibers is discontinuous, where cellulosic fibers are found in matrix; this contains pectin, lignin and hemicellulose. The ultimate fibers from esparto grass have a length between 0.3 and 2.5 mm and a diameter between 5 and 20 μm. This shows that ultimate fibers are very short but with interesting features and the process of esparto grass fiber results in an excellent quality of fibers .This study emphasizes the use of esparto grass fiber for different useable products in order to improve its value.

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ЕКСТРАГУВАННЯ І ХАРАКТЕРИСТИКА КОРОТКИХ ВОЛОКОН АЛЖИРСЬКОЇ ЕСПАРТО (*STIPA TENACISSIMA*)

Анотація. *Альфа-волокна – це волокна на основі целюлози, отримані з листя альфа трави (еспарто) лужним методом для видалення таких нецелюлозних речовин, як пектин, лігнін і геміцелюлози. Досліджено морфологічні характеристики (довжина, ширина) волокон еспарто. Екстраговане целюлозне волокно охарактеризовано за допомогою інфрачервоної спектроскопії, рентгенівської дифрактометрії, термічного аналізу, оптичної та скануючої електронної мікроскопії. Проведені дослідження волокон еспарто, отриманих хімічною екстракцією з використанням гідроксиду натрію і гіпохлориту натрію як відбілюючого агента. Визначено їх морфологічну структуру.*

Ключові слова: *целюлоза, волокно еспарто, екстраговане волокно, хімічне оброблення.*