Chemical Composition, Morphology and Tensile Properties of Spanish Broom (Spartium junceum L.) Fibres in Comparison with Flax (Linum usitatissimum L.)

Abstract
Flax has been usually used to produce natural cellulose fibres with properties suitable for composite, textile, and other high-value fibrous applications. Spanish Broom, extracted by a physical-chemical process, is a source of cellulose fibres that can be used, as well as flax, in various fields (textile, paper, composites etc.). The aim of this study was to describe the chemical composition, morphology and tensile properties of Spanish Broom fibres in comparison with flax. The morphology of both fibres was established by optical microscopy (OM). The chemical composition and tensile properties of Spanish Broom fibres were determined according to conventional methods. The results show that Spanish Broom fibres have a higher cellulose content (91.7%) and better tensile properties than flax fibres. Moreover, Spanish Broom fibres have a smaller diameter (7-10 μm) than flax (17-24 μm), as observed by OM, and the cross-sections show an irregular polygonal shape with well defined lumen. Spanish Broom fibres were found to have very good tensile properties as well as thermal stability and could successfully replace flax in many applications.

Key words: cellulose, Spanish Broom fibres, fibre extraction, flax fibres.

Introduction

Recently there has been increasing worldwide interest in the use of lignocellulosic fibres (hemp, flax, ramie, sisal and jute) as an alternative to synthetic fibres for various applications in the textile industry [1 - 6] such as a reinforcement for composite materials [7 - 9] or raw materials for thermal insulations [10]. The use of bast fibres in many applications depends on their composition, extraction process and physical properties [11].

Generally natural fibres present important advantages such as low density, appropriate stiffness and mechanical properties as well as high disposability and renewability in comparison with synthetic fibres [12, 13]. Moreover they are recyclable and biodegradable. Bast fibres such as flax, hemp and Spanish Broom are some of the oldest sources of cellulosic fibres. Currently flax is widely used by the textile industry as speciality fibres to create a distinctive fabric with unique characteristics, while the paper and composite industries utilise this strong bast fibre to improve strength [14]. In contrast, the use of Spanish Broom fibres has been limited due to the low efficiency of the extraction process. In the past, people used Spanish Broom fibres mainly for coarse fabrics and cordage. Recently, fibres from Spanish Broom were extracted by an easy, efficient, convenient and fast physical-chemical process, increasing the possibility of extensive application of these fibres on a large scale in various fields, including the textile industry [15].

Spanish Broom is a small shrub available in Mediterranean countries, where it grows spontaneously. In comparison with flax and hemp, Spanish Broom grows in the most unfavourable limestone soil and once planted it can be used during a period of up to twenty years, whilst hemp and flax demand high quality soil each year. Taking into account this information, in this work we report the chemical composition, morphology and tensile properties of Spanish Broom fibres in comparison with flax.

Materials and methods

Materials

Spanish Broom shrubs were grown in a research field at Calabria University, Arcavacata di Rende (CS), Italy. The Spanish Broom was used without any pretreatment or purification. Flax was supplied by Linificio & Canapificio Nazionale (LCN), Bergamo, Italy.

Fibre extraction

Fibres were extracted by a physical-chemical process, as reported in our previous work [15]. Briefly, fibres were extracted by treating vegetable branches with a 15% (w/w) sodium hydroxide solution at 100 °C for 15 min. The hot sprigs were washed in water to obtain rough fibres that were further cleaned from lignin by air oxidation into an autoclave.

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at 120 °C and 1 MPa pressure for 3 h. The fibres were finally washed and dried.

**Fibre composition**

The amount of cellulose in the fibres was determined using the colorimetric method with the anthrone reagent [16]. Pentosans and lignin were determined according to the TAPPI T223 hm 84 (1984) [17] and TAPPI T222 om 02 (2002) [18] methods, respectively. The ash content of the fibre was determined by weighting the residue remaining after ignition at 575 °C for 3 h [19]. Pectins were determined colorimetrically using the carbazole method [20].

**Fibre morphology**

A Leitz DB-MR microscope was used to observe the cross sections of Spanish Broom and flax fibres. The diameter of the fibres was measured according to UNI 5423/64.

**Colour measurement**

The colour coordinates of Spanish Broom and flax fibres were determined using a Minolta CM 2600-D colorimeter and CIE Lab colour system. Each sample was analysed five times.

**Tensile properties**

The tensile properties of the fibres in terms of tenacity, percentage of elongation at break and initial modulus were determined using an Instron 6021/5500 tensile testing machine according to UNI EN ISO 5079 at room temperature (20 °C) and 65% relative humidity. About 100 fibres were tested and the average and standard deviations recorded.

**Moisture regain and water absorption**

The moisture regain of Spanish Broom and flax fibres was determined at different temperatures and humidities. Water absorption analyses were determined gravimetrically. The specimens were immersed in distilled water, took out of the water and excess water was removed before weighting.

**Thermogravimetric analysis**

The fibres were heated in Netzsch 429 Thermogravimetric analysis (TGA) apparatus at a rate 20 °C min⁻¹ from room temperature to 500 °C. TGA was performed in static air.

**Results and discussion**

**Fibre composition**

The chemical composition of Spanish Broom fibres is reported in Table 1 in comparison with that obtained from flax. As seen from the table, the cellulose content is higher in Spanish Broom fibres than in flax. The lignin content in Spanish Broom fibres is similar to that of flax fibres. The pectin and pentosan content is lower in Spanish Broom fibres than in flax fibres. The chemical composition differences observed among Spanish Broom fibres is similar to that of flax fibres. The pectin and pentosan content is lower in Spanish Broom fibres than in flax fibres. The chemical composition differences observed among Spanish Broom and flax fibres can be attributed to various factors such as the composition of the plant and also, not insubstantially, to the conditions used for extracting the fibres [21, 22]. Thus it is possible to modify extraction conditions (NaOH concentration, time) to remove further hemicelluloses and lignin from fibres.
Table 1. Comparison of chemical composition of Spanish Broom and flax fibres.

<table>
<thead>
<tr>
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<th>Spanish Broom fibres</th>
<th>Flax fibres</th>
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<tbody>
<tr>
<td>Cellulose, %</td>
<td>91.7 ± 0.1</td>
<td>75.3 ± 0.3</td>
</tr>
<tr>
<td>Lignin, %</td>
<td>3.2 ± 0.4</td>
<td>4.2 ± 0.1</td>
</tr>
<tr>
<td>Pentosans, %</td>
<td>4.1 ± 0.3</td>
<td>16.3 ± 0.2</td>
</tr>
<tr>
<td>Pectins, %</td>
<td>0.0 ± 0.0</td>
<td>3.2 ± 0.3</td>
</tr>
<tr>
<td>Ash, %</td>
<td>1.0 ± 0.2</td>
<td>1.0 ± 0.1</td>
</tr>
</tbody>
</table>

Table 2. Tensile properties of Spanish Broom fibres compared with flax fibres.

<table>
<thead>
<tr>
<th>Fibre properties</th>
<th>Spanish Broom</th>
<th>Flax</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, g/ml</td>
<td>1.36</td>
<td>1.41</td>
</tr>
<tr>
<td>Tenacity, cN/tex</td>
<td>35.9</td>
<td>16.9</td>
</tr>
<tr>
<td>Strain at break, %</td>
<td>5.8</td>
<td>1.9</td>
</tr>
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Morphological structure

Figure 1 shows a optical micrograph of a transversal cross-section of the Spanish and flax fibres. Both fibres are arranged in bundles of single cells called elementary fibres. Although the number of elementary fibres is the same for Spanish flax, it was observed that Spanish Broom fibres are slightly smaller than flax fibres. In particular, the diameter of Spanish Broom varies in the range of about 7 - 10 μm, while the flax diameter varies in the range of about 17 - 24 μm. These differences influence the properties of fibres such as water absorption.

Moreover Spanish Broom as well as elementary flax fibres have an irregular polygonal shape with well defined lumen.

Colour measurement

Fibre colour is an important quality in design applications, for example in the automotive and textile sectors [23]. The colour values of Spanish Broom as well as flax fibres are presented in Figure 2.

From the colour values, it was observed that Spanish Broom fibres were less bright and yellower than flax. In fact, the L value of Spanish Broom samples is lower than that of flax, decreasing the brightness, while the b value (which represents variations in yellowness to blueness) is higher than that of flax. These differences in colour can be attributed to various factors such as the extraction process [24], but also to raw materials and storage conditions.

Tensile properties

The tensile properties of Spanish Broom fibres compared with flax fibres are reported in Table 2. Spanish Broom fibres have a higher tenacity and strain at break than flax fibres. These data indicate that Spanish Broom fibres will be more soft and flexible than flax fibres.

Like other lignocellulosic fibres (hemp, sisal, kenaf) [25], Spanish Broom as well as flax fibres are characterised by low density, which is highly appreciated in the automotive industry [26].

Moisture regain

Table 3 shows the moisture regain at different contents of relative humidity (RH) of Spanish Broom and flax fibres. The moisture regain is slightly lower in Spanish Broom fibres than in flax, suggesting that apparel made from Spanish Broom fibres are more absorbent and would be comfortable to wear. The higher moisture regain of flax fibres can be attributed to the presence of non-cellulosic substances, especially pentosans (16.3%) and pectin (3.2%), which are more hydrophilic with respect to cellulose [27].

Water absorption

In Table 4 the values of water absorption of Spanish Broom compared with flax fibres are reported. Spanish Broom fibres have a higher water absorption value than flax fibres, suggesting the possibility to create more comfortable clothes. This behaviour can be attributed to the higher fineness of Spanish Broom fibres compared to flax, as confirmed by the morphological analysis.

Thermogravimetric analysis

Figure 3 shows thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of Spanish Broom and flax fibres. According to other studies on lignocellulosic fibres [28, 29], after the initial weight loss at 100 °C due to moisture evaporation of the fibres, significant weight loss for both fibres is observed over 200 °C, suggesting the thermal deg-
radation of hemicellulose, cellulose and lignin. In addition, DTG curves show two endothermic peaks at 335 °C and 472 °C for Spanish Broom and at 343 °C and 470 °C for flax, corresponding to the decomposition of cellulose and lignin, respectively. Therefore it can be concluded from these results that Spanish Broom as well as flax fibres show good thermal sta-

Conclusions
Natural fibres with a very high cellulose content, can be extracted from Spanish Broom spontaneously growing in Medi-
terranean countries. Spanish Broom fibre applications were very limited in the past, due to many difficulties of the extraction method. Nowadays the use of a physical-chemical process to extract Spanish Broom fibres allows to obtain fibres which can be conveniently used in various fields (textile, biochemical conversion, composites etc.). In this work, we compared Spanish Broom fibres with flax, which is one of the oldest sources of cellulose fibres. The results show that the morphology of both fibres is similar, but Spanish Broom fibres have a smaller diameter than flax. Moreover comparing Spanish Broom fibres with flax, it was observed that Spanish Broom fibres have very good tensile properties as well as thermal stability and could successfully replace flax in many applications.

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References
1. Liu Z, Erhan SZ, Akin DE, Barton FE. “Green” composites from renewable re-

sources: preparation of epoxidized soya-

soy beans and oil and flax fibre composites. J. Ag-

3. Joseph PV, Joseph K, Thomas S, Pillai CKS, Prasad VS, Groeninck G, Sark-

4. Fung KL, Xing XS, Li RKY, Tjong SC, MaiYW. An investigation on the pro-
cessing of sisal reinforced polypropy-
7. Eichorn SJ, Baillie CA, Zafeiropoulos N, Mwaikambo LY, Ansell MP, Dufresne A, Entwistle KM, Herrera-Franco PJ, Es-
12. Wambua P, Ivens R, Verpoest I. Natu-

13. Herrera-Franco PJ, Valadez-Gonzalez A. Mechanical properties of continuous natural fibre-reinforced polymer com-

14. Foulk JA, Chao NY, Akin DE, Dodd RB, Layton PA. Analysis of flux and cotton fiber fabric blends and recycled polyeth-
15. Gabriele M, Cerchiaro T, Salerno G, Chidichimo G, Vetere MV, Alampi C, Gallucci MC, Conidi C, Cassano A. A new physical-chemical process for the efficient production of cellulose fibers from Spanish broom (Spartium junce-

19. Han JS, Rowell JS. Chemical composi-
tion of fibers. In: Paper and Composites from Agro-Based Resources (Chapter 5); Rowell, R. M.; Young R. A.; Row-
20. McComb EA, McCready RM. Colorimet-
ric determination of pectic substances. Analytical Chemistry, 1952, pp. 1630-
1632.
21. Deleghini Bonatti P, Ferrari C, Focher B, Grippo C, Torri G, Casentino C. His-
tochemical and supramolecular studies in determinino qualità of hemp fibres for textile applications. Euphytica 2004; 140: 55-64.
23. Musig DJ, Stevens Ch. Industrial appli-
cations of natural fibres: structure, prop-
erties and technical applications. John Wiley & Sons Ltd., 2010: 373.
24. Akin EA, Epps HH, Archibald DD, Shek-
25. Kalia S, Kaith BS, Kaur I. Pretreatments of natural fibers and their application as reinforcing material in polymer composi-
26. Pauksztas D, Borysiak S. The influence of processing and the polymorphism of lignocellulosic fillers on the structure and properties of composite materials. A re-
28. De Rosa IM, Kenny JM, Puglia D, San-
tulli C, Sarasini F. Morphological, ther-
mal and mechanical characterization of okra (Abelmoschus esculentus) fibres as potential reinforcement in polymer composites. Composites Science and Technology 2010; 70, 1: 116-122.
29. Chen WH, Kuo PC. A study on torrefac-
tion of various biomass materials and its impact on lignocellulosic structure simu-

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