



Spanish Broom Fibres Properties and Extraction for Application in Composites: A Review

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Abstract

The development of a number of natural fibre products for textiles was attempted during the period of Italian autarchy (1935–1943) under the direct support of the Fascist regime. Among these fibre products, Spanish broom fibre is derived from the stem of *Spartium junceum*, a spontaneous plant in large parts of Italy, especially in the Southern region of Calabria. The extraction of the fibre proved cumbersome though, especially for the hardness of the stem. Therefore, different retting processes were developed, mechanical, chemical and also bacterial and, in the latter case through the use of *Clostridium felsineum*, it was proved to be effective to a point allowing the production of textiles. The historical events linked to the fall of the dictatorship led to some oblivion and also to the possible loss of archive materials. The objective of this review is to elicit ideas and developments concerning an effective extraction and degumming of Spanish broom fibres and evaluate the more recent applications to the field of biocomposites.

Keywords: Enzymatic treatment, Biocomposites, Spanish broom, Natural fibers

1 Introduction

Over the last decades, the re-introduction of some traditional fibres in the textile sector has been attempted, with variable success. In the specific case of Italy, in the period known as “autarchy” (1935–1943), a number of textile industry patented products were developed, such as milk whey fabrics (Lanital) [1], hybrid cellulosic/protein fibres (Cisalfa), and viscose fibres from perennial cane cellulose (*Arundo Donax*), patented as Arundax: all these products had a limited success over time. As far as the direct use of natural fibres is concerned, the main developments regarded hemp (*Cannabis sativa* L.), mulberry (*Morus Alba* L.) (gelsolino), and finally Spanish broom (*Spartium*

junceum L.), [2]. In the latter case, Spanish broom was cultivated mostly in non-productive and marginalised terrains, such as along the railway lines. Historical events linked to the fall of the dictatorship led to some oblivion and also to the possible loss of archive materials, which is not to deny the value embedded in some of those ideas for the textile industry, especially as far as fibre extraction is concerned.

It is worth mentioning though that the more recent development of natural fibre composites (NFCs) involved the need for the production of textiles to be coupled with polymer resins considered with some degree of attention all the above-mentioned plant fibres, demonstrating in general terms that their prospective application would possibly concern also

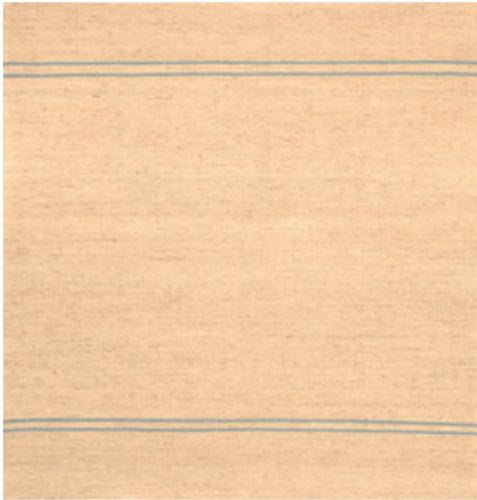


Figure 1: Spanish broom fibre textile.

the field of NFCs. These possibilities included also the above-mentioned fibres. More specifically, the use of hemp in composites is now widespread [3]. As regards the other fibres object of the autarchy experiments, these have been the attention of specific studies, such as it is the case for Donax cane [4], mulberry [5], and regenerated casein fibres [6]. This has been over time also the case for Spanish broom.

In particular, Spanish broom had at the time a particularly significant development, which resulted in the constitution of over 60 industries for the production of Spanish broom fibre textiles in all regions of Italy (an example is shown in Figure 1), with a distinct prevalence in the Calabria region [6]. The initial objective was the production of highly fashionable textiles. However, obtaining other products is also possible from these plants. This is the case e.g., for seed oil [7], aromatic water with anti-tumoral activity [8], cellulose fibres for chemicals (Bisphenol A) removal from polluted water [9], anti-bacterial wound dressing [10], [11], also compared with hemp and flax [12] or remediation of aquifers from heavy metals [13]. In terms of achieving a higher crystalline content, Spanish broom provided also the raw material for the extraction of cellulose nanocrystals, compared with other fibres from spontaneous plants, such as nettle [14].

These initial considerations offer to the productive system characteristics closer to a circular economy (no-waste) concept, which has been recognised in literature [15]. The objective of this work is to update



Figure 2: Spanish broom vermenes.

to this moment the ideas concerning the production of high-quality textiles from Spanish broom fibres and to try to elicit and recover as much as possible this knowledge, especially on fibre extraction, applying it to the production of composites. The review is divided into three parts, first discussing the general properties of Spanish broom fibres and the productive system connected, and then concentrating on the extraction process, with particular reference to the bacterial one, based on the use of *Clostridium felsineum* [16], and finally discussing the applications so far in composites and the possible developments. This enables Spanish broom fibres entering the field of less-diffuse plant fibres for the filling and prospective reinforcement of polymers, a field that has demonstrated a wide activity over the last two decades or so, intensifying over the last few years, also in view of a higher sustainability of materials for commodity and structural uses [17]–[20].

2 Spanish Broom Characteristics

Spanish broom, also known as Weavers' broom, belongs to the peas' family. Data in literature suggest a wide variability of the cellulose content across the plant: some work suggests that the whole stem has cellulose content between 67 and 76% [21], although for the plant branchlets, also referred to as "vermenes", this might be down to 44% [22]; this difference might be due to the fact that the whole of structural polysaccharides (cellulose and pentosans) might have been considered together in [21]. In Figure 2, an image of the vermenes is depicted.

The density of the fibres has been measured to be in the region of 1.45 g/cm³, quite close to that of the

most diffuse ones used in composites, such as hemp or flax [23]. Also, further data from [22] suggest that the branchlets contain also a significant amount of pectin, over 13%, which appears to be quite separated and noticeable. This constitutes an important difference e.g., from hemp, where pectin and lignin are strictly bound together so that in practice the former is completely removed by alkali boiling treatment [24]. In Table 1, the amounts of the different chemical components in Spanish broom branchlets, hence before fibre extraction, which would remove the extractives (pectins, waxes, etc.) from different studies are reported.

Table 1: Composition of untreated Spanish broom vermenes (branchlets)

Component	Content (%)	
Cellulose	44.5 ± 0.2	50.5
Pentosans	16.3 ± 0.1	15.3
Lignin	18.5 ± 0.3	14.4
Pectins	13.3 ± 0.1	16.2
Waxes	3.4 ± 0.1	
Ash	4 ± 0.2	3.6
Ref.	[21]	[25]

Harvesting the fibres by hand allowed obtaining in the case of Spanish broom fibre bundles with diameters in the region of 50 µm, as bundles of ultimate cells, especially connected by lignin: they show a stiffness of 21.5 ± 5 GPa, and a strength of up to 400 MPa, with a limited elongation to break from 3 to 5%, reduced with strain gauge. This suggests that in these conditions the introduction of Spanish broom fibres in polymer composites can be advisable only for thermosetting or quite hard (glassy) matrices [25].

The extraction of the fibres from the stem does therefore represent a notable issue for the possible weaving in textiles and the insertion as reinforcement in composites. In some cases, this has been avoided at all, for example for the application of Spanish broom into adobe bricks to provide tensile resistance [26]. In this case, tensile properties of the sprigs have been measured, obtaining 41.53 ± 4.13 MPa for ultimate stress and 2.72 ± 0.19 for ultimate strain.

3 Extraction Methods

Due to the importance of extraction method in tailoring the properties of Spanish broom fibres obtained,

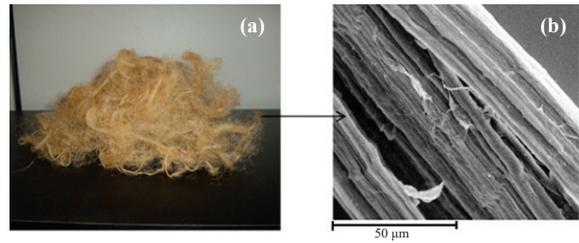


Figure 3: (a) Spanish broom fibres extracted by physico-chemical process; (b) SEM micrograph (1000x magnification) [10].

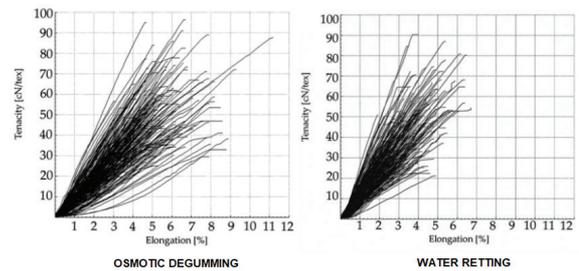


Figure 4: Tenacity vs. Elongation of Spanish broom fibres extracted through different methods (extracted from [27]).

different methods have been proposed and applied. The aspect of fibres obtained after physico-chemical extraction is represented in Figure 3 [10]. A study by Kovačević compared two possible retting procedures: water retting and osmotic degumming, evaluating the fineness of the fibres obtained, bearing in mind that for Spanish broom fibres the maceration times were longer than for flax ones in view of the higher hardness of its stem [27]. The dispersion of fineness values and therefore also of mechanical performance was larger for osmotic degumming than for water retting, as indicated in Figure 4, although in practice water retake under a moisture content of 65% resulted comparable for the two cases.

A comparative study concerned in particular four extraction methods: after soaking in seawater for 21 days (fibres yield 12 wt%); in sodium hydroxide at 120°C for 3 h (yield 30 wt%); in sodium hydroxide at 100°C for 15 min, then after rinsing, in autoclave at 120°C at 10 atmospheres (yield 30 wt%); in an alkali solution and then in a 900 W microwave oven, for a period of 10 min, then rinsed in hot and cold distilled water (yield 30 wt%) [28]. In terms of quality of

extraction, the one performed with microwaves requires the least amount of energy and the shortest time.

It has also been clarified that the type of extraction performed on the fibres would substantially vary their composition, as reported in Table 2 below. The two treatments applied in [21] were a physico-chemical (PC) process, which involved a treatment with 15% sodium hydroxide solution at 100°C for 15 min and a mechanical (MEC) process of decortication and hand brushing. While the former left basically no other components than cellulose, the latter left reasonably intact the lignin-pectin complex: it might be suggested that physico-chemical process would be more suitable for the production of paper or cottonized fibres, while for composites the mechanical extraction would in contrast be more appropriate.

Table 2: Composition of extracted Spanish broom fibres by physico-chemical (PC) or mechanical (MEC) process

Component	PC	MEC
Cellulose	91.7 ± 0.1	66.9 ± 0.1
Pentosans	4.1 ± 0.3	7.2 ± 0.3
Lignin	3.2 ± 0.4	11.7 ± 0.2
Pectins	0	12 ± 0.2
Waxes	1 ± 0.2	1 ± 0.1
Ash	0	1.2 ± 0.1

On the other side, the high level of crystallinity of Spanish broom fibres would contribute to its brittleness. This has been reduced by fatty acid grafting of the fibres in [29], which was demonstrated to be particularly effective with olive oil, reducing the crystallinity index from 55.8% to 42.4% and increasing the first onset of the degradation peak from 177 to 198°C.

4 Enzyme Retting of Spanish Broom

This section deals particularly the results of retting via pectinolytic enzymes, which were synthesized by micro-organisms, such as *Bacillus felsineus*, first used on flax for pectin fermentation, around 1915 [30], [31], then extended to hemp, and patented by Carbone in 1925 [32]. During autarchy, the *Bacillus felsineus* was also used also on Spanish broom. After the first studies, which were extended throughout the world, it was noticed that *Bacillus felsineus* released spores,

so that it was renamed as *Clostridium felsineum* [33].

Basically, the idea is that these bacteria are able to dissolve pectin present in the bundles, therefore untying them into fibres, the specific reaction is called pectin lyase: however, these bacteria were not mentioned amongst the agents producing this reaction, also in a recent review [34]. It is worth observing that obviously these are not the only possibility for microbial retting, for example for hemp. In particular, it was proved that the signal intensity of Fourier Transform infrared spectroscopy (FTIR) measurements at 1734 cm⁻¹ [35] of the non-cellulosic components (hemicellulose esters and pectic substances), normalized with the internal standard at 2916 cm⁻¹ of the sample subjected to 12 days of retting, was remarkably lower than after only 3 days [36]. In other cases, white rot fungi *Ceriporiopsis subvermispora* and *Phlebia radiata* Cel 26 proved effective during water retting, also reducing porosity, dissolving lignin and pectin, with a relatively low loss of cellulose [37]. It might also be the case that the number of agents for this process would be much larger than recognised so far. Attempts to use *Bacillus felsineus* also on some other fibres have also been proposed in recent years, such as ramie, in the perspective of its introduction in the Mediterranean context [38]. One study related with *Bacillus felsineus* compared two bacterial strains, which increased the crystallinity index and provided a higher tensile strength, particularly on a younger crop [39].

In Table 3, some approximate properties of Spanish broom fibres, as reported above, are summarised, as obtained from the most effective fibre retting (osmotic or bacterial).

Table 3: Spanish broom fibres properties

Average Properties	Values
Density	1.45 g/cm ³
Tenacity	65 cN/tex
Maximum elongation	6%

5 Spanish Broom Composites

Recovering some information about Spanish broom fibres and the most suitable methods for cultivation in order to improve their quality in prospective textiles and therefore composites has been the object of specific studies, such as [40], which in particular correlated its use to other infesting crops, such as nettle (*Urtica*

Dioica L.). The use of fibres from infesting crops has in effect been recommended as a possibility for future development of plant fibre composites, for their relatively easy availability and their less strict requirements in terms of seasonality and plantation ground [41].

In engineering terms, a typical application of plant fibres during last few years involves their insertion as fillers, and hopefully reinforcement, into biodegradable matrices, with the idea to produce a fully sustainable material. Among these, polylactic acid (PLA) is mainly popular, particularly for its relative similarity to some oil-based thermoplastics, which proved suitable for applications in composites, such as in the automotive sector e.g., polypropylene (PP) and polyethylene (PE). The introduction of Spanish broom into PLA proved partially successful, since it required improving the toughness on the produced composites, by adding further fillers, for example ceramics, such as montmorillonite, and sustainable crosslinkers, such as citric acid [42]. A more recent work suggested the application of Spanish broom flour as the filler for a PP matrix, in the view of treating the filler as the by-product of a possible productive system to be revived: in this case, to improve adhesion, flour was treated by alkali or silane [43]. Also, the effect of compatibilization of PP matrix with maleic anhydride was investigated on composites with silanised Spanish broom and pine cone flour, obtaining by the introduction of the two fillers a higher crystallinity of the composite [44].

The particular hardness and difficult extraction of Spanish broom fibres suggested also other possible applications for them, such as the introduction into ceramic matrices into cement mortar, therefore in competition with more diffuse fibres, such as sisal [45], or coir and oil palm [46]. In the specific case of serving as the reinforcement for cement mortar, particularly aggressive treatments were used, such as seawater followed by alkali treatment with sodium hydroxide, in the idea of resorting with a substantial matter removal. Also, the influence of harvesting time was also significant for this application [47]. In another case, different amounts of sodium hydroxide and of sodium hydroxide/sodium sulphate mixture were applied to these composites, again with a pronounced matter removal effect [48]. The need for a somehow intrusive chemical treatment is justified by the particularly complex structure of Spanish broom stem, as reported in Figure 5.

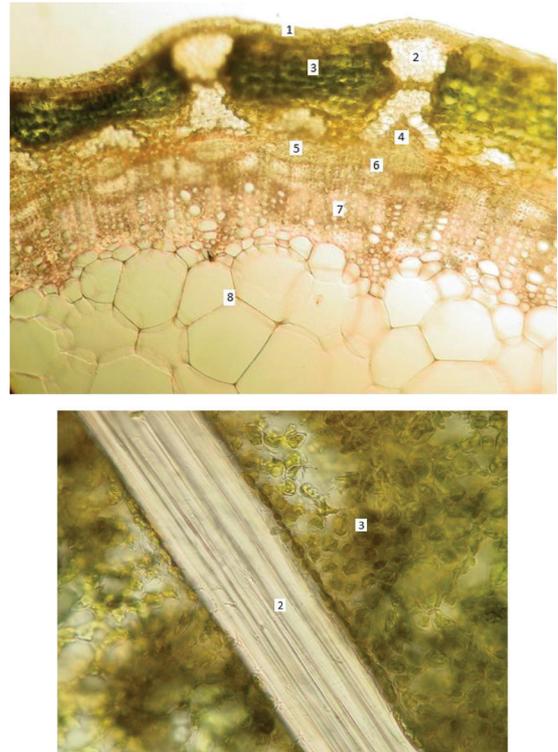


Figure 5: Cross section and longitudinal section of a Spanish broom (*Spartium junceum* L.) stem (1: epidermis with a cuticle, 2: sclerenchyma, 3: parenchyma with chloroplast, 4: primary phloem, 5: secondary phloem, 6: cambium, 7: secondary tree, 8: sapwood) section of Spanish broom (*Spartium junceum*) stem [47].

6 Conclusions

Spanish broom fibres have been proposed over time for application in composites, because of their infesting character, which make their growth quite easy in some regions, such as in Italy, in the Balkans and elsewhere in hilly areas not far from the Mediterranean Sea. When it comes to the possible production of textiles to be introduced in composites, this has proved very difficult so far, since the extraction of the fibres appears cumbersome and the variability of fineness obtained with different treatments is significant. Different retting methods have been applied, including chemical, mechanical and enzymatic, the latter using specific bacteria (*Clostridium felsineum*), which has revealed its effectiveness also on flax and hemp. Prospective interest exists for further use of these fibres in

biocomposites, to be considered as a by-product of a potentially vast productive system, based on seed oil, extracts and cellulose. Applications so far concerned mainly a few matrices, such as poly(lactic acid (PLA), polypropylene (PP) and also cement mortar reinforcement.

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