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Spanish broom Production Chain Improvement with a View to Sustainable Development

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Highlights

What are the main findings?

- Broom plants are a source of fiber obtained by a natural method, thus moving away from the use of sodium hydroxide, which has been the conventional method for fiber extraction.
- The rehydration time, of the plant, 4 days, is crucial for the recovery of significant amounts of cellulose. Additionally, the residual biomass from the broom plant is a significant source of pectin, comprising approximately 6%.

What is the implication of the main finding?

- Broom plants do not need any treatment; they are wild plants that do not need irrigation.
- It is possible to reduce the environmental impact by eliminating the use of sodium hydroxide and by extracting additional soluble fiber, pectin, from the plant's waste. Pectins obtained are valuable polysaccharides widely utilized in the food industry as gelling agents, thickeners, and stabilizers, thereby enhancing the economic viability of broom cultivation.
- This discovery not only promotes sustainable practices but also highlights the potential for utilizing natural resources more efficiently!

Abstract: The extraction of *Spanish broom* fibers presents significant commercial opportunities. However, the traditional production process is associated with a high environmental impact and considerable waste. This work demonstrates how to address the limitations of alkaline maceration by employing a natural maceration process. This innovative method not only reduces environmental harm but also facilitates the extraction of large quantities of pectin (6%). Notably, pectin has been obtained from the waste product of broom processing, creating a dual source of profit: both cellulose and pectin. This means that not only can the fibers be utilized for various applications, but the by-products can also be transformed into a valuable marketable product. Pectin, a valuable polysaccharide widely used in the food industry as a gelling agent, thickener, and stabilizer, can significantly increase the economic viability of broom cultivation. Moreover, the high yield of pectin from Spanish broom underscores the plant's potential as a sustainable resource, making it an attractive alternative to more environmentally damaging crops. Pectin obtained has been characterized by Fourier Transform Infrared Spectroscopy (FT-IR) and Scanning Electron Microscopy (SEM), providing valuable insights into its structural and morphological properties.

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1. Introduction

Until the end of the 20th century, research in the fields of chemistry, physics, and business related to materials and the circular economy, primarily focused on enhancing plastic systems derived from polymers synthesized from petroleum derivatives [1–4]. Firstly, there is a concern regarding the limited availability of fossil fuel resources [5]. In 2005, Hirsch [6] estimated that oil reserves were projected to be depleted within 25 years. Secondly, and perhaps more critically, there is a growing awareness of the irreversible ecological damage [7,8] caused by the indiscriminate use of non-biodegradable materials sourced from oil, which hinders the achievement of sustainable development goals [9–11]. In response to these challenges, both the scientific and industrial sectors have dedicated the past fifteen years to diversifying the sources of raw materials, focusing on abundant and renewable resources [12–16]. Among these new sources, two molecular species derived from the plant kingdom—cellulose and lignin—have garnered significant attention [17,18]. These compounds are synthesized by plants using water, carbon dioxide, and sunlight, making them ecologically compatible organic materials. Particularly when extracted from fast-growing plant species, cellulose and lignin present promising opportunities for research and practical applications in sustainable material development [19,20].

The production of *Spanish broom* fiber came to a halt after World War II due to the labor-intensive nature of the fiber extraction process, which rendered the costs uncompetitive compared to other fibers. The plant's branched structure made it impossible to extract cellulose fibers using mechanical processes alone [21]; instead, a combination of chemical and mechanical methods was required, which could not be automated at that time [22,23]. This presented a significant commercial and technical disadvantage, despite the numerous advantages of *Spanish broom*.

This plant grows without the need for pesticides, irrigation, or fertilizers, and when cut 20 cm above the ground, it can regenerate to the same height within a year. This cycle of regrowth and harvesting can continue for up to thirty years without compromising the plant's vitality. Establishing new plantations within a short supply chain could position the broom plant as a strategically valuable resource for various industrial sectors. For instance, the broom groves in Calabria alone could potentially supply the market with 200,000 tons per year of cellulosic fibers and lignins [24].

Spanish broom fiber has significant market opportunities [25–28]. For instance, India, the second-largest producer of brooms in the world after China, has a substantial market for broom products (Figure 1A,B). The Indian broom market was valued at USD 74.8 million in 2020 and is projected to grow at a compound annual growth rate (CAGR) of 5.6% during the forecast period from 2020 to 2026 [29]. The increasing demand from end users, including households, hotels, restaurants, hospitals, shopping malls, and educational institutions, is a significant driver of growth in this market [30–32]. Furthermore, rising disposable incomes among Indian consumers have led to a heightened demand for high-quality brooms at affordable prices, further stimulating the growth of India's broom industry in recent years. As of 2023, the Indian broom market has recorded a Herfindahl–Hirschman Index (HHI) of 5322, which represents a slight decrease from the HHI of 6266 in 2017. This indicates a trend towards market concentration. The Herfindahl index is a measure of market competitiveness, with values ranging from 0 to 10.000. A lower index value signifies a

larger number of market participants or exporting countries, while a higher index value indicates fewer players or countries in the market.

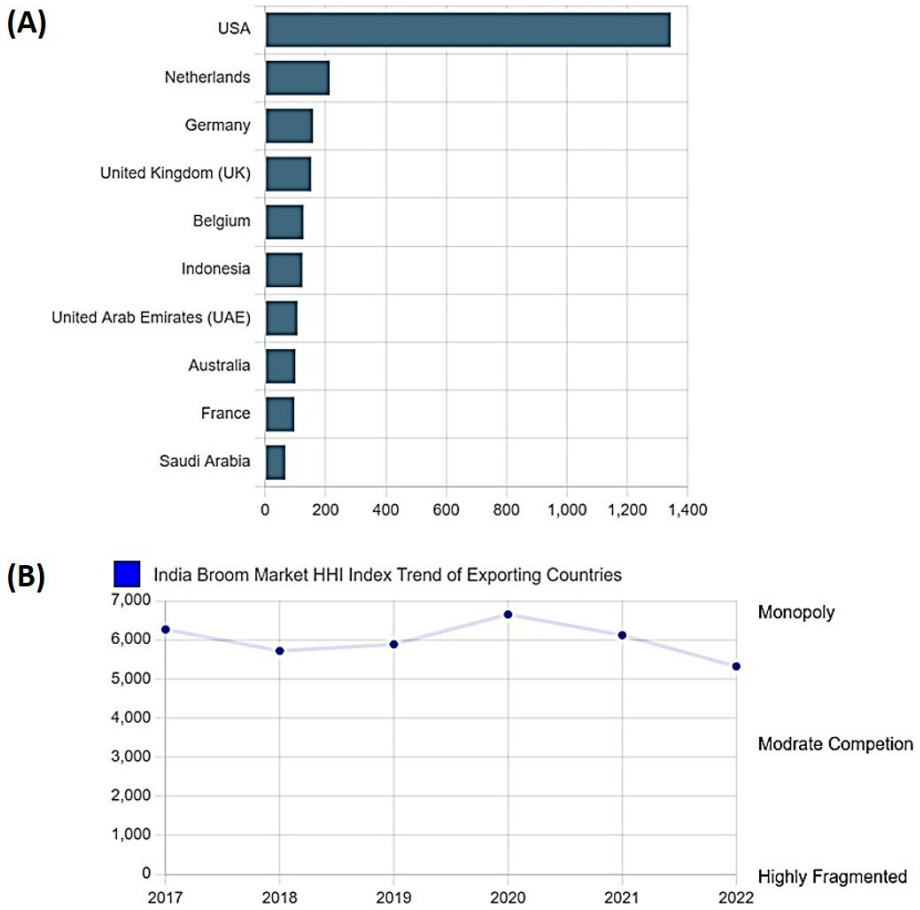

Figure 1. (**A**) India broom market trend; (**B**) India broom export [33].

The Indian market is striving to align with eco-friendly initiatives aimed at reducing environmental pollution caused by the traditional use of non-biodegradable synthetic fibers in the manufacturing process [34–36]. However, the Indian market is also characterized by high raw material costs. The manufacturing process relies on materials such as plastic molding resins, wood pulp derivatives, and coconut fibers, which constitute a significant portion of the total production costs. This situation makes it challenging for manufacturers to maintain profit margins, thereby diminishing their competitiveness against rivals who utilize lower-cost alternatives like bamboo or recycled plastics. Consequently, this leads to pricing pressure on producers with high input costs.

The Italian experience shows that textile and fashion companies are increasingly focused on ecological considerations and sustainable production practices. As a result, they are increasingly supportive of using textile fibers derived from broom, as its production does not require chemical inputs. Economic analysis indicates that managing and harvesting a broom grove of about 200 hectares necessitates at least 20 work units. Once harvested, the broom is sent to a fiber extraction plant, which requires an additional 12 work units for processing. The development of a broom supply chain could significantly increase employment opportunities for skilled workers, including researchers, agronomists, foresters, engineers, designers, and other professionals. This growth would not only benefit the agricultural sector but also have positive implications for various production industries, such as clothing, automotive, furniture, and civil construction [37–40]. *Spanish broom*, in particular, has the potential to make a significant impact in the fabric and waste sectors,

as its production does not involve the use of polluting and harmful substances [41]. In contrast, cotton production is currently responsible for a quarter of all insecticides and herbicides used globally. As a result, there is a growing interest in alternative fibers like linen and hemp, which are being adopted by various brands looking to replace cotton. These factors present a unique opportunity for Italy, especially in regions with extensive areas covered by spontaneous broom groves and land that is otherwise unused or abandoned. The advantages of using these natural resources are the following: (1) broom can be grown on very poor soil that is not suitable for other crops, improving its landscape appearance [42]; (2) the roots of the broom sink into the ground to a depth of several meters, giving great stability to the slopes against landslides, and giving the crops resistance even against fires, which do not affect the roots; (3) it requires neither the irrigation during the growth cycle nor the use of insecticides and fungicides; (4) after cutting, it grows back spontaneously; (5) a broom grove that is systematically subjected to annual cuts remains stable over time for at least 30 years; (6) the cellulose fibers extractable from broom have excellent mechanical and chemical-physical qualities, comparable and even in some aspects (elasticity, breathability) superior to those extractable from other bast plants [43–46].

The objective of this study is to explore the sustainable processes involved in the extraction of *Spanish broom* fiber.

2. Materials and Methods

2.1. Isolation of Cellulose

In a first defibration laboratory process, developed during this research, bunches of brooms were previously macerated for half an hour in a 5% NaOH solution, at a temperature of 80–90 °C and then defibrated by rotating cylinders having longitudinal rows of harmonic steel teeth [47]. This type of maceration generates significant quantities of aqueous waste, which should be purified with stage filtration techniques, to recover both the process water and the basic substances from combustion of the filtered solids [48]. The overall process ends up becoming too expensive, when compared to the revenues obtainable from the marketing of the produced fibers.

This research illustrates how to overcome the drawbacks linked to the use of alkaline maceration of the plant by means of a natural maceration process. Such a process, although slower, does not compromise the speed of the industrial fiber production process if, as we will see, the collection of the plant is brought forward by one week compared to the start of processing the macerated material for the production of the fiber.

Broom was harvested in bunches of 200 g each, ensuring the use of plants that had been cut at a height of 20 cm from the ground the previous year. In this way, the new brooms all had a bushy appearance with central twigs having a trunk less than 4 mm, and lateral twigs having an average diameter of the order of 2 mm. The bunches were left to dry in the sun for three consecutive days, until the twigs showed signs of losing their typical flexibility and becoming so rigid that they were easily broken transversally due to bending. At this point, 12 dehydrated bunches were rehydrated with water sprayed through appropriate spray nozzles, in an apparatus that allowed the recycling of the used water. The decks were then extracted from the rehydration circuit three at a time, at intervals of 24, 48, 72, and 96 h, to be subjected to a manual defibering process. This process was performed in two stages.

The first stage had the aim of delicately removing the superficial cuticle of the brooms by means of brushes with plastic bristles soft enough not to penetrate between the cellulose fibers underlying the cuticular layer. The objective was to remove the superficial layer of the macerated cuticle which, being rather rubbery and adhesive, would remain glued to the fibers, compromising their quality and degree of purity, if not previously removed [49]. The

second stage of the defibration consisted in the actual extraction of the fibers, brushing the brooms with brushes with harder teeth than those of the brushes used in the first stage, and such as to remove the fibers from the underlying woody part [50]. In the laboratory process, these two phases of the mechanical treatment of the plant were carried out manually, taking care to spray the action areas of the brushes with jets of water, so that all the residues of the superficial cuticle of the brooms, just removed by the mechanical action of the brushes, were removed from the aqueous flow.

The experimentation has shown that these residues settle easily, in times of the order of half an hour, when the water used is left in a settling tank, from which it can be taken back in a closed cycle for reuse in cleaning and defibering the brooms. The fibers removed manually by the extraction brushes were then dried at 80 °C and carded using a laboratory carding machine to dryly remove the last remaining encrustations. An example of fibers extracted in this way is shown in Figure 2.

Figure 2. Cellulose fiber extracted from *Spanish broom*.

2.2. Isolation of Pectin

The cuticular wastes generated through the fiber extraction process were freeze-dried (Telstar freeze-dryer, mod. Cryodos) at −20 °C, then grilled to 120 µm, and subjected to further extraction for recovering pectin to be used in the pharmacological field for their antimicrobial activity [51]. The extraction of pectin from waste powder was carried out according to the experimental conditions reported by La Torre et al. [52]. The waste powder (2 g) from rehydration period of the broom stems equal to 2, 3, and 4 days (2D, 3D, and 4D, respectively) was added with 10% (*w*/*v*) citric acid solution until the pH of two was reached. The suspension was magnetically stirred for 1 h at 90 °C, cooled, and centrifuged (5000 rpm, t = 30 min). Then, the supernatant was added with an equal volume of absolute ethanol and kept for 16 h at 4 °C in order to allow for pectin flotation. The centrifugation (Universal 320, Hettich Zentrifugen, Merck, Italy) allowed to separate the pectins that were rinsed with absolute ethanol, solubilized in distilled water, added to an equal volume of acetone for further purification, and left to stand at 4 °C for 12 h. Afterwards, the supernatant was separated by centrifugation (5000 rpm, t = 15 min), and the gelatinous residue was dried under a vacuum. The extraction procedure was repeated three times and data expressed as mean ± standard deviation. All samples were characterized by FTIR (Fourier Transform Infrared Spectroscopy) and SEM (Scanning Electron Microscopy).

2.3. Determination of Esterification Degree

The esterification degree of pectin is defined as the ratio of esterified carboxy groups to the total number of carboxy groups. It was determined by using two methods: the titrimetric method (DE) and the instrumental FT-IR method (DM).

2.3.1. Potentiometric Titration Method

The esterification degree of pectin (DE) was evaluated by applying the protocol previously described by Fazio et al. [53]. The dried pectin (20 mg) was dissolved in distilled water (5 mL). The suspension was heated at 45 °C under magnetic stirring for 30 min to facilitate the solubilization of the sample. The resulting solution was titrated with 0.01 N NaOH using three drops of 1% phenolphthalein as the indicator, and the volume consumed was recorded as V1 once a pale pink color appeared. V1 indicated the number of free carboxy groups. Then, 3 mL of 0.01 N NaOH was added, and the solution was stirred at room temperature for 2 h in order to saponify the esterified carboxy groups of the polygalacturonic acid. The unreacted sodium hydroxide was neutralized by 3 mL of 0.01 N hydrochloric acid (HCl), and the excess HCl was retro titrated by 0.01 N NaOH in the presence of phenolphthalein. The titration volume consumed representing the esterified group number in pectin sample, was recorded as V2. The DE was calculated as follows:

$$\mathrm{DE}(\%) = \frac{\mathrm{V2}}{(\mathrm{V1} + \mathrm{V2})} \times 100 \quad (1)$$

2.3.2. Instrumental FT-IR Method

The methoxylation degree (DM) of pectin was determined by analyzing the FT-IR spectra, taking into account specific bands at 1747–1746 cm^{-1} and 1633–1621 cm^{-1}, corresponding to esterified and free carboxyl groups, respectively. FT-IR spectra of pectin were obtained from the analyses performed using a Bruker ALPHA FT-IR (Bruker, Billerica, MA, USA) spectrometer equipped with a A241/D reflection module in 4000–400 cm^{-1} range and applying the standard KBr method [54]. The pectin was ground with KBr in the 1:40 ratio, and the resulting powder was hard-pressed into tablets. DM was proportional to the ratio of the area from the band at 1747–1746 cm^{-1} over the sum of the areas from the bands at 1747–1746 cm^{-1} and 1633–1621 cm^{-1}:

$$\mathrm{DM}(\%) = \frac{\mathrm{A\ 1750\ cm^{-1}}}{\mathrm{A\ 1650\ cm^{-1}} + \mathrm{A\ 1750\ cm^{-1}}} \times 100 \quad (2)$$

2.4. Scanning Electron Microscopy (SEM)

The surface morphological investigations were performed by a scanning electron microscope (SEM) (Field Emission SEM FEI Quanta 200, Thermo-Fisher Scientific, Hillsboro, OR, USA) and Electron Probe Micro Analyzer (EPMA)-JEOL-JXA 8230t (JEOL Ltd, Kyoto, Japan). The analyses were carried out as reported by Fazio et al. [55].

2.5. Statistical Analysis

The experiments were conducted in three replications, and the averages were subjected to variance analysis using GraphPad Prism 10.3.1 software (GraphPad Software, San Diego, CA, USA). The study opted for the two-way ANOVA followed by Tukey's test to determine the differences between the means ± standard deviations of the pectin extraction yields and for the degree of esterification and methoxylation of pectin.

3. Results and Discussion

3.1. Cellulose Extraction

The cuticle residues, sedimented in the washing water, were recovered to extract useful materials. This material, which at first glance could only be considered a waste material from the fiber extraction process, actually appears to contain non-negligible quantities of pectin, which acts as a glue between the surface cuticle and the underlying fibers. In this experiment, the quantity of pectin obtainable as a by-product of the broom signature extraction process was also evaluated. Table 1 shows the quantity of cellulose fiber (without further purification treatments) and of dry cuticular residues. The percentage yields were calculated on the basis of the starting amount of green broom equal to 200 g.

Table 1. Amount of cellulosic fiber and waste obtained after different maceration time.

Maceration Time (h)	Cellulosic Fiber (g) I	Fiber/Green Plant (%) II	Dry Cuticle Waste (g) III	Dry Cuticle/Green Plant (g) IV
24	2.70	1.30	1.85	0.92
48	10.20	5.10	8.90	4.50
72	15.80	7.90	14.60	7.30
96	17.20	8.60	16.80	8.40

The best yield of the fiber extraction process, using the process described in this work, is obtained for broom rehydration times of 96 h, i.e., 4 days. It could be seen that beyond this time no improvements in extraction yield were obtained, as after this rehydration period there were no longer any fibers attached to the woody part of the brooms after combing with the extraction brushes. Even though the maceration process takes much longer than the one which can be achieved with hot alkaline solutions, there is no slowdown in the extraction of the fiber during the main process, involving the actual defibering of the plant. The time gap in the maceration process is overcome by placing several maceration towers at the defibration plant, so that the defibration machine can process the material from a single tower without interruption, as it gradually reaches the adequate level of maceration, moving on to treat the material from the subsequent towers in the following days. This type of plant maceration can therefore be considered sustainable and entirely "green", as it requires no chemical reagents and produces no dangerous processing waste (only small quantities of modest organic sludge with no odorous effects). Additionally, it can be disposed of within the same production chain as fertilizers. Furthermore, the process consumes very little water: only what is lost through evaporation during the rehydration phase of the plant (minimal, however, given that the process takes place at room temperature).

3.2. Pectin Extraction

Pectin recoveries from dried wastes obtained from broom at 2, 3, and 4 days of rehydration were 14.9 $\pm$ 2.4 mg/g (1.46% yield), 31.1 $\pm$ 5.4 mg/g (3.1% yield), and 63.3 $\pm$ 8.5 mg/g (6.2% yield), respectively (Figure 3). These results showed that the pectin content significantly changed as the rehydration time of dried brooms. In particular, the recovery of pectin increased with increasing hydration period of the plant stems. In fact, the extraction yield of 3D was doubled (3.1%), while 4D yield quadrupled (6.2%) compared to 2D yield (1.46%).

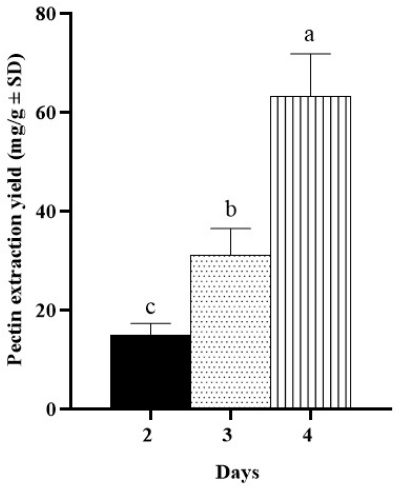

Figure 3. Extraction yields of pectin at 2, 3, and 4 days of dried broom rehydration. The letters on the bars indicate significance.

3.3. FT-IR Analyses

The FTIR spectra of waste pectin samples 2D, 3D, and 4D are reported in Figure 4. Typical bands appearing in the spectra confirmed the structure of pectin. The bands characterizing the structure did not show significant differences, except for their relative absorption intensities. The spectra showed a large band in 3426–3454 cm^{-1} range. It was generated by OH stretching arising from inter- and intramolecular hydrogen bonding of the galacturonic acid backbone. The C–H stretching determined the presence of a small band in the range of 2921–2853 cm^{-1}. An important region was between 1748–1628 cm^{-1}, where the bands allowed the detection of esterified vs. un-esterified functional groups, that is to say the methoxylation degree of pectin. In fact, the first one was due to the C=O ester carbonyl group stretching, and the second one is generated by COO^{-} carboxylate ion one [52]. The extracted pectin from dried broom 2D (black line) and 3D (yellow line) highlighted in this spectral region the bands at 1748 cm^{-1} and 1628 cm^{-1} with greater intensity of the carboxylate ion band than of the esterified group, while 4D sample did not show any band at 1748 cm^{-1} confirming the absence of esterified carboxyl groups, The Raman bands occurring in 1160–1019 cm^{-1} were generated from C-C stretching, COH deformation, and asymmetric C-O-C stretching vibration.

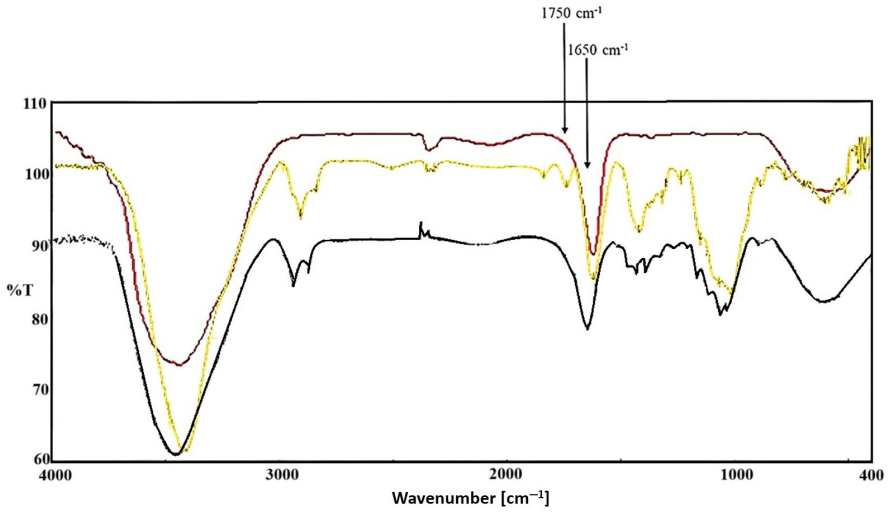

Figure 4. FTIR spectra of pectin from dried broom 2D (black line), 3D (yellow line), and 4D (red line) in the 400–4000 cm^{-1} region.

3.4. Esterification Degree

Pectin is a heteropolysaccharide containing at least five different sugar moieties, but 80–90% of its dry weight is galacturonic acid (Gal*A*), which is mainly present as a linear chain of homogalacturonan in which the carboxyl group is substituted in varying proportions by introducing Gal*A* subunits containing carboxymethyl groups at position 6 [56]. It is crucial in characterizing the esterification degree of pectin, which is an important parameter conditioning its functional properties in food [57]. Pectin can be classified into two types: high-methoxyl (HM) form, characterized by a percentage of esterified groups greater than 50%, and low-methoxyl (LM) form, in which the percentage is less than 50%. The esterification degree of pectin from dried broom at 2, 3, and 4 days of rehydration (2D, 3D, and 4D, respectively) was determined by both classical titrimetric method (DE) and instrumental FTIR method (DM) (Figure 5). Each sample was analyzed in triplicate by titrimetric and instrumental methods, and the results were reported as mean ± standard deviation. The titrimetric values of DE for pectin 2D were found to be 52.3%, but decreased to 6.6% for pectin 4D. The instrumental technique, based on FTIR spectroscopy, provided slightly lower values for 2D (52.9%) and 3D (46.2%), while 4D highlighted the absence of esterified groups in the homogalacturonan backbone. The degree of esterification of 4D was significantly lower than that of 2D and 3D, while among the three pectin samples, 2D was also significantly higher than 3D.

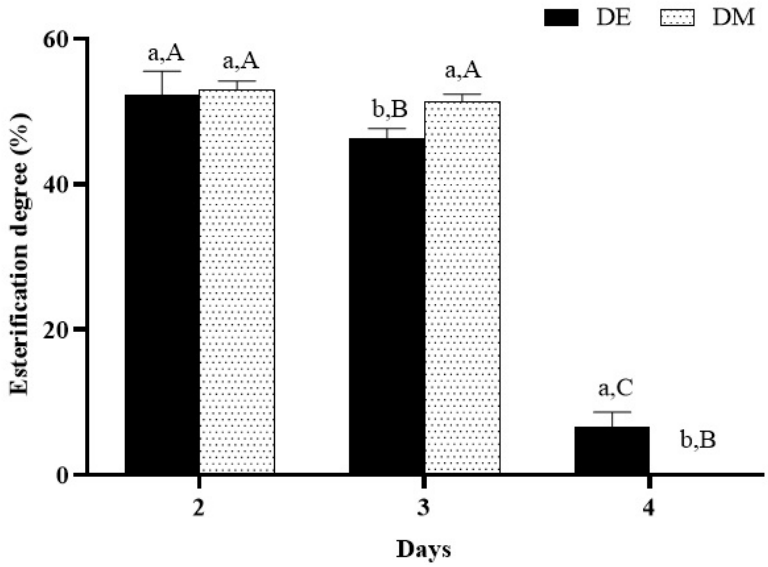

Figure 5. Esterification degree of waste pectin from dried brooms at 2, 3, and 4 days of rehydration determined by titrimetric (DE) and instrumental methods (DM). Error bars represent standard deviation ($n = 3$). Small letters indicate significance between DE and DM at the same day, while capital letters indicate significance in the same sample (DE or DM) at different days. Values with the same letter were not significantly different, while values with different letters were significantly different.

3.5. Scanning Electron Microscopy (SEM) of Pectin

The morphological characteristics of pectin were investigated by SEM at three magnifications equal to 100×, 200×, and 1000×, with a scale equal to 800, 300, and 80 µm, respectively. SEM images showed different surface structures, depending on the rehydrating period of dried brooms, i.e., 2D, 3D, and 4D (Figure 6). Sample 2D (Figure 6A) showed an irregular, micro-fractured surface similar to flakes. In fact, at low magnifications, the flake structures appeared connected to each other. At high magnifications, an irregular surface with microfractures and smooth regions was visible. In addition, it was possible to see a few medium-sized random cavities (pores). They were distributed in an irregular

pattern. Sample 4D (Figure 6C) showed a filamentous structure and a microporosity evenly distributed over the entire surface and the absence of flake-like microfractures observed on the surface of sample 2D. Sample 3D (Figure 6B) was an intermediate surface structure compared to samples 2D and 4D, exhibiting an irregular and rough surface and the presence of some filaments. In all samples, crystalline fragments were distributed over the entire surface.

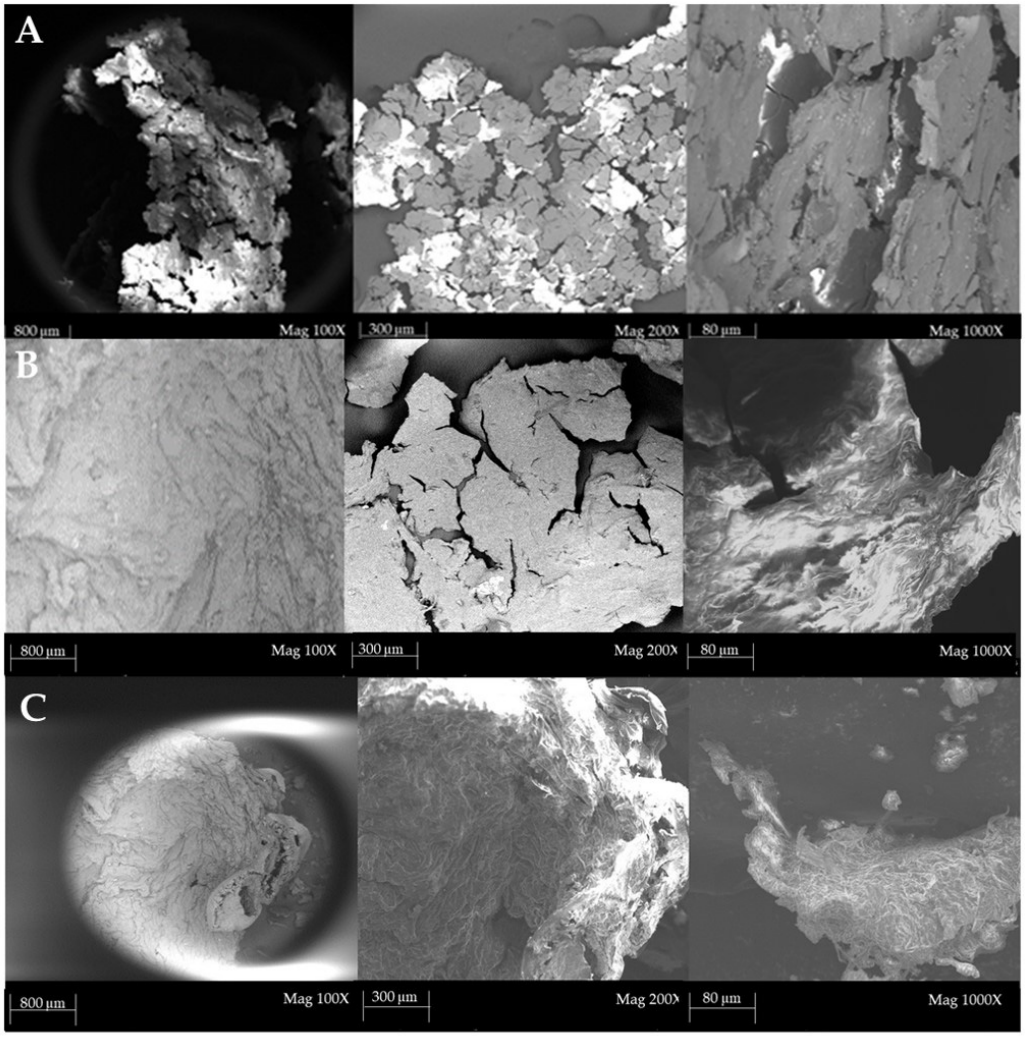

Figure 6. Scanning electron images of pectin from dried brooms 2D (**A**), 3D (**B**), and 4D (**C**) at three different magnifications and scales (100×, 800 µm), (200×, 300 µm), and (1000×, 80 µm).

4. Conclusions

The research aimed to achieve a substantial yield of cellulose from the defibering of broom, an invasive plant, utilizing a more environmentally friendly methodology compared to existing techniques. The conventional methods employ sodium hydroxide, which necessitates costly disposal procedures to mitigate pollution risks. The newly developed extraction process efficiently produces not only cellulose but also significant quantities of pectin from the cuticles of broom, which are collected in the washing water during the defibering process. The results obtained show that the rehydration period of dried broom influences both the extraction yield and the degree of methoxylation and the morphological characteristics of the extracted pectin. In any case, the highest extraction yield was obtained from the cuticular residues of broom rehydrated for 4 days. Current market prices of pectin indicate that its commercialization could generate revenue comparable to, or even

exceeding, that derived from fiber sales. The establishment of an initial industrial plant, characterized by a high degree of automation, is now feasible. This plant is essential for developing a broom fiber production chain and promoting this high-quality fiber within the natural textile sector. As the fiber extraction techniques mature, we can anticipate the rapid formation of broom fiber production chains that will engage various stakeholders, including farmers responsible for ensuring a steady supply of raw materials, the metalworking industry for the construction of defibration facilities, and companies involved in spinning and weaving. This process and the results of its implementation contribute to the achieving of the following Sustainable Development Goals (SDG):

- SDG 6 "Clean Water and Sanification" (target 6.4 "By 2030, substantially increase water-use efficiency across all sectors and ensure sustainable withdrawals and supply of freshwater to address water scarcity and substantially reduce the number of people suffering from water scarcity");
- SDG 9 "Industry, Innovation and Infrastructure" (target 9.4 "By 2030, upgrade infrastructure and retrofit industries to make them sustainable, with increased resource-use efficiency and greater adoption of clean and environmentally sound technologies and industrial processes, with all countries taking action in accordance with their respective capabilities";
- SDG 17 "Partnerships for the Goals" (target 17.16 "Enhance the Global Partnership for Sustainable Development, complemented by multi-stakeholder partnerships that mobilize and share knowledge, expertise, technology and financial resources, to support the achievement of the Sustainable Development Goals in all countries, in particular developing countries").

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