




Review

# Investigating the Routes to Produce Cellulose Fibers from Agro-Waste: An Upcycling Process

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**Abstract:** The agriculture and agri-food sectors produce substantial amounts of plant-based waste. This waste presents an identifiable research opportunity to develop methods for effectively eliminating and managing it in order to promote zero-waste and circular economies. Plant-based waste and by-products are acknowledged as valuable sources of bioactive compounds, including cellulose fibers. Direct application of these fibers in non-food sectors such as textiles can reduce the environmental impact of secondary raw materials. This review aims to provide an overview of novel concepts and modern technologies for efficiently utilizing plant-based waste and by-products from the agricultural and agro-industrial sectors to extract fibers for a variety of final applications, including the fashion industry. Two major routes are identified to produce cellulose fibers: the extraction and purification of natural cellulose fibers and the extraction and purification of cellulose pulp that is further processed into manmade cellulosic fibers. Scalability of experimental results at the laboratory or pilot level is a major barrier, so it is critical to develop closed-loop processes, apply standardization protocols, and conduct life cycle assessments and techno-economic analyses to facilitate large-scale implementation.

**Keywords:** agricultural waste; agro-industrial waste; cellulose fiber; textiles; extraction process; dissolving pulp; regeneration; upcycling; agro-waste valorization



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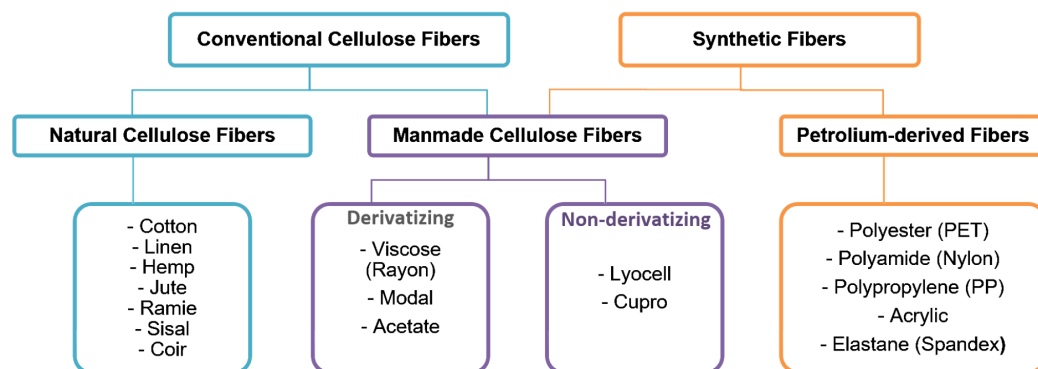
## 1. Introduction

Cellulose was first isolated by the French chemist Anselme Payen in 1838. It is considered to be the most renewable and abundant polymer that can be found in nature today, and the most common organic compound on earth, as several billion tons are biosynthesized annually [1]. The cellulose polymer consists of glucose monomers that are connected in a unique way, joined at  $\beta$ -1,4 linkages, to make cellulose very resistant to breakdown. Thus, cellulose is the structural part of the cell wall of plants and many forms of algae [2]. In the textile industry, the most important and dominant feedstocks were cellulose-based fibers until the boom in oil-based synthetic fibers during the 1950s [3]. Oil-based synthetic fibers are less expensive; however, cellulosic fibers, both cotton and regenerated cellulose fibers, have not been replaced by synthetic fibers due to their unique physical properties that enhance the comfort of textile products. Cellulose-based fibers have higher breathability, better moisture management, recyclability, biodegradability, and good mechanical properties [4].

Since the world is now turning to more ecological, sustainable, and environmentally friendly solutions, extracting cellulose or cellulose fibers from waste with various methods is a good alternative to cover the needs for industrial uses. Of particular interest is the

utilization of the large quantities of lignocellulose waste produced as a result of agricultural and food processing activities, which require further handling and treatment [5]. On a global scale, agriculture generates more than 6.3 petagrams (Pg) of dry matter per year in the form of organic waste products. Crop residues, rich in cellulose content, account for more than half of this amount [6]. The management of these wastes mainly consists of land application for erosion prevention and nutrient recycling. On a smaller scale, collection, transportation, and disposal are implemented, and to a limited extent, their utilization is for compost production, animal feed, or energy generation. Alternatively, the garbage is incinerated [7]. Inappropriate disposal of agricultural residue results in the emission of greenhouse gases (GHGs) such as carbon dioxide (CO<sub>2</sub>), nitrous oxide (N<sub>2</sub>O), and methane (CH<sub>4</sub>), endangering both human health and the natural environment [8,9]. Moreover, certain inherent properties of agricultural and agro-industrial waste make their handling difficult: (a) the biological instability and rapid growth of pathogens may quickly lead to unacceptable sanitary conditions; (b) the high moisture content increases transportation costs, while water removed by mechanical means requires management as liquid waste; (c) rapid auto-oxidation may occur, resulting in the emission of malodorous fatty acids; and (d) the enzymatic activity of many by-products of the food processing industry can further enhance their prompt decomposition [10]. Engaging in upcycling can help avoid harmful activities like burning agricultural waste, which results in the emission of carbon dioxide (CO<sub>2</sub>). Fibrous applications are highly prominent among the various uses and waste valorization possibilities due to their potential for high value addition. These applications can include industrial wastes from the juice or compote industries, such as bagasse and peels.

Simultaneously, global fiber production continues its increasing trend after a small decline during the COVID-19 pandemic to reach a new record high in 2021 that corresponds to the production of 14.3 kg per person. Synthetic, petroleum-derived fibers dominate the market, making up 64% of global production, followed by natural cellulose fibers (NCFs) with a market share of around 28%. Manmade cellulose fibers (MMCFs), such as viscose, acetate, and lyocell, account for 6.4% of the global market [11]. They are synthetic polymers made from natural sources through regeneration processes [12]. MMCFs exhibit superior mechanical qualities, including elevated tensile and flexural strength, intrinsic durability, and decay resistance [13]. Especially for the textile industry, it is essential to search for new resources, methods, and technological applications to produce more environmentally friendly products that nevertheless meet consumers' needs [14] and align with the circular economy model. This trend has gained popularity as a means to mitigate the adverse effects of textile production on the environment and climate [15]. The environmental impact of textile products is significantly influenced by the production phase, in addition to the use phase. The garment production process accounts for roughly 80% of total climate change impacts, primarily due to fossil fuel consumption. Within this 80%, fiber production contributes 16% to the impact of climate change. The cultivation phase of conventional cotton significantly contributes to water scarcity, accounting for 87% of the impact [16]. Consequently, certain advancements focus on reducing fossil fuel reliance by replacing synthetic fibers with renewable options, while others aim to minimize water and land usage by substituting cotton with alternatives [5]. However, the utilization of first-generation feedstock, such as food crops or intentionally cultivated fiber crops, results in eutrophication, ecotoxicity, land use, and water consumption issues. When crops replace petrochemical inputs, the demand for primary inputs significantly increases due to necessary modifications in feedstock and processes [17]. Thus, the use of lignocellulose agricultural by-products can potentially increase the production and market share of bio-based textiles while avoiding negative climate effects, the expansion of arable land covered by fibrous crops [18], and the contribution to deforestation via the use of uncertified wood pulp [11]. Figure 1 shows the categorization of conventional cellulose and synthetic fibers.



**Figure 1.** Categorization of conventional cellulose and synthetic fibers.

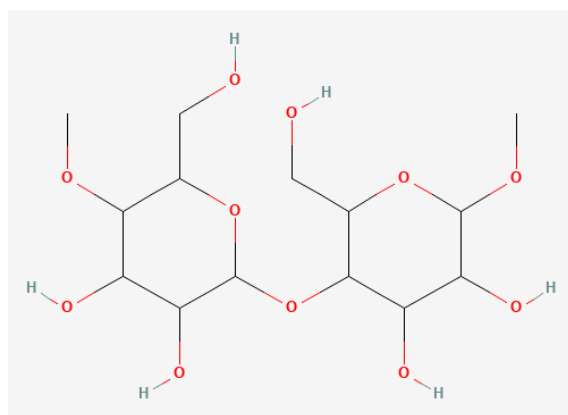
Depending on the initial plant source and fibrous material content, two major approaches to producing cellulose fibers are identified by reviewing the literature: (1) the extraction and purification of NCFs, and (2) the extraction and purification of cellulose pulp (CP), which is transformed into MMCFs through an additional regeneration process.

#### Research Objectives

The purpose of this study is to identify the possible processing routes that can lead to the upcycling and valorization of lignocellulosic agricultural and agro-industrial waste for their exploitation as cellulose fibers. The research objectives of the present study are: (1) to identify the building units of the raw material that need to be preserved and/or regenerated to obtain cellulose fibers; (2) to identify the upcycling routes for fiber production; (3) to study the applicable processes and understand the advantages and drawbacks of each method; and (4) to map research results and commercial advances in order to offer a wide and thorough understanding of the studied subject and boost further research.

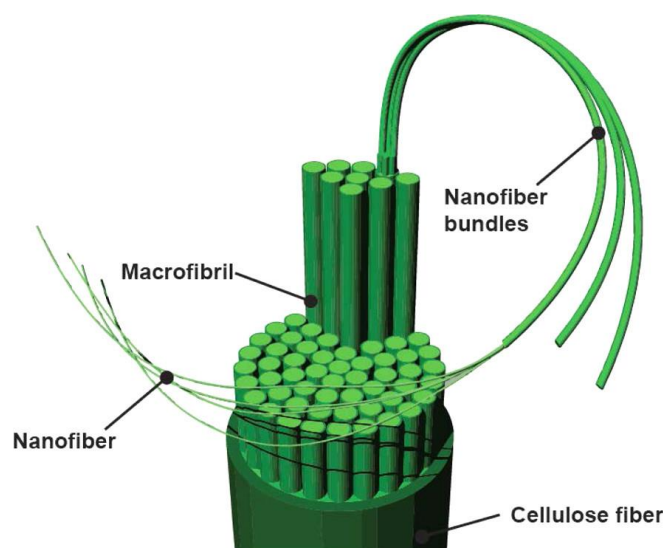
## 2. Cellulose and Cellulose Fibers

Cellulose ( $C_6H_{10}O_5$ )<sub>n</sub> (Figure 2) is a complex carbohydrate and a structural polysaccharide in plant cells. It consists of linear glycan chains, i.e., chains of several hundred to several thousand D-glucopyranose units (D-glucose) linked together by  $\beta$ -1,4-glucosidic bonds, each with a different degree of polymerization. All plant matter is composed, on average, of 33% cellulose, with 90–94% of cotton and around 50% of wood being cellulose [19–21]. The regular arrangement of hydroxyl groups along the cellulose chains leads to the formation of fibrous structures held together by intra- and intermolecular hydrogen bonds and intermolecular Van der Waals forces [22]. The strong hydrogen bonds that are developed in the macromolecular structure of cellulose make it insoluble in water and in common organic solvents, significantly limiting its use in various applications [23].



**Figure 2.** Chemical structure depiction of cellulose (PubChem Identifier: CID 14055602) [24].

The fibrous structures that form the aggregates of cellulose are of particular interest in terms of their arrangement in space (Figure 3). Cellulose macromolecules constitute elongated linear chains without bifurcations that allow them to form a parallel arrangement in space and, by extension, to form crystals or aggregates with a high degree of orientation. Therefore, along the microfibrils of cellulose, both regions of high orientation (crystals) and low orientation (amorphous regions) are found. Gaps are also observed between the microfibrils, which are occupied by other plant tissue molecules such as hemicelluloses, lignin, and pectin [25]. Up to 36 cellulose chains can form the elementary fibril (alternatively nanofibril or nanofiber) in plants, while several such fibrils can form micro- and macrofibrils. Bundles of many macrofibrils form cellulose fibers [26].



**Figure 3.** Size hierarchy: from cellulose fiber to cellulose nanofiber. Reprinted with permission from [27]. Copyright 2015 American Chemical Society.

Green plants, algae, tunicates, and some bacteria exhibit the cellulose I modification in its natural form [28]. According to Pérez and Samain [23], cellulose I can be transformed irreversibly into a stable crystalline form, namely cellulose II. Two different processes, mercerization and regeneration, both applicable in the textile industry, can achieve the conversion into this new allomorph. Immersing cellulose in a concentrated aqueous solution of sodium hydroxide (NaOH) mercerizes it, causing intracrystalline swelling and subsequent recrystallization after washing. Regeneration occurs after the coagulation and recrystallization of a cellulose or cellulosic intermediate derivative solution. Cellulose I exhibits a parallel arrangement of cellulose chains, whereas cellulose II displays an antiparallel arrangement. Chemical treatment with ammonia or certain amines produces cellulose III from cellulose I or II, and the treatment of cellulose III with glycerol at high temperatures results in cellulose IV.

Cellulose fibers consist of highly oriented cellulose macromolecules. Most NCFs are staple fibers with relatively short lengths ranging between 3 and 20 cm. Conversely, MMCFs are initially produced as filament fibers of indefinite length. Distinct methods of processing the fibers are then chosen to form a continuous strand of yarn [29].

### 3. 1st Route Towards Cellulose Fibers: Extraction and Purification of NCFs

Different methods have been employed to extract fibers from their native microenvironment, which is composed of wax and other substances such as lignin, hemicellulose, and pectin, causing them to remain attached and to have an uneven appearance [30]. Processing techniques that can be applied both to conventional and agro-waste fibers include mechanical, chemical, and biological treatments that remove the impurities and improve the morphology, processability, and final properties of the NCFs. The success of fiber

extraction is determined by the acquisition of long, fine, and pure fibers with low instances of breakage [18].

Different types of processes applicable for the extraction of natural fibers are presented in Table 1.

**Table 1.** Established processes used to produce NCFs.

Method	Purification Agent	Short Description	Pros	Cons	References
Dew retting	Fungi such as <i>Aspergillus</i> sp., <i>Penicillium</i> sp., and <i>Cladosporium</i> sp.	Fibrous plant parts are placed uniformly across the crop field. Air, dew and sunlight support fungal colonization that deconstructs plant tissue towards the research of fibers.	<ul style="list-style-type: none"> <li>- Easy removal of pectin.</li> <li>- Low cost.</li> <li>- No need for technical facilities.</li> <li>- Applied in areas with water scarcity.</li> </ul>	<ul style="list-style-type: none"> <li>- Reduced strength.</li> <li>- Inconsistent quality.</li> <li>- Properties of the final product influenced by climate and weather.</li> <li>- End point difficult to define.</li> <li>- Over-retting results in poor fiber quality.</li> <li>- Time consuming (2–3 weeks).</li> </ul>	[31–34]
Water retting	Bacteria such as <i>Clostridium</i> sp., and <i>Bacillus</i> sp.	Fibrous plant parts are immersed in water (fresh water or seawater) that causes swelling and allows the development and activity of decay-causing bacteria. The process conditions are monitored periodically.	<ul style="list-style-type: none"> <li>- Produces fiber of greater uniformity and high quality.</li> <li>- Possible use of different water systems such as rivers, ponds, or tanks.</li> <li>- Low cost.</li> </ul>	<ul style="list-style-type: none"> <li>- Extensive water contamination.</li> <li>- Presence of anaerobic bacteria.</li> <li>- Wastewater treatment is required.</li> <li>- Difficulties in controlling the retting conditions.</li> <li>- Time consuming (7–25 days).</li> <li>- Final qualities are time dependent.</li> </ul>	[32–34]
Enzymatic process	Enzymes such as pectinases, xylanases, cellulases	The enzymes cause degradation of the components separating the cellulosic fiber from non-fiber tissues. Conditions are controlled based on the enzymes applied.	<ul style="list-style-type: none"> <li>- High-quality fibers.</li> <li>- Final properties depend upon selection of enzymes, conditions and process duration.</li> <li>- Considerably less duration, less than 24 h</li> </ul>	<ul style="list-style-type: none"> <li>- High cost of commercialized enzymes.</li> </ul>	[32,33,35–39]
Decortication	Impact, shear, and compressive forces	Squeezing and breaking are used to separate fibers from other unwanted plant tissue.	<ul style="list-style-type: none"> <li>- No use of chemicals or need for wastewater treatment. Capability for extensive fiber purification.</li> </ul>	<ul style="list-style-type: none"> <li>- Lower yields.</li> <li>- Shortening and/or damage to fiber resulting from applied stress.</li> </ul>	[37,40,41]
Steam explosion	Steam	Fibrous plant parts are subject to steam boiling, followed by a phase of explosion when the pressure drops. Temperature and pressure are controlled.	<ul style="list-style-type: none"> <li>- Cost-effective.</li> <li>- Environmentally friendly without the use of chemicals.</li> <li>- Time efficient (few minutes).</li> </ul>	<ul style="list-style-type: none"> <li>- Utilization as pretreatment process: non-completion of the extraction.</li> </ul>	[42–46]
Chemical process	Chemicals such as sodium hydroxide, hydrogen peroxide, sodium percarbonate, sulfuric acid, and oxalic acid	Treatment with chemical agents. Time, temperature, and concentrations are controlled. May include pre-treatment or post-treatments.	<ul style="list-style-type: none"> <li>- Fiber of superior quality and consistent uniformity.</li> <li>- Minimum processing time (approx. 2 h or less)</li> </ul>	<ul style="list-style-type: none"> <li>- Reduction of fiber properties, including length, if overprocessed.</li> <li>- Higher processing cost.</li> <li>- Production of wastewater that requires treatment.</li> </ul>	[37,44,47,48]



### 3.1. Biological Processes

**Retting** is a simple process that separates the fiber bundles from the central stem by loosening the fibers from the woody tissue of the fiber crops [49]. However, excessive extraction of pectins and natural waxes would result in an undesirable fiber texture, making it dry and rough. Complete removal of pectins will result in the disintegration of fiber bundles into elementary fibers [50]. The retting process utilizes the action of microorganisms and moisture (**dew retting**) or water (**water retting**) to dissolve or decompose the cellular tissues and other unwanted substances that surround the fiber bundles. Water retting of hemp and flax has been demonstrated to be improved by the combined use of microwaves, which has also resulted in considerably shortening the retting time needed to 24 h [51].

Gradually, the term retting evolved to incorporate advanced processes, such as enzymatic, chemical, and mechanical processes; however, in this paper, they are discussed separately under their respective categories.

**Enzymatic treatment** is based on the enzymatic endohydrolysis of cellulose, hemicellulose, lignin, and pectin found in plants. Cellulases tend to break down the amorphous regions of the fibrils in priority rather than the crystalline regions. Thus, fibers with high crystallinity may be produced; however, a prolonged retting period will result in an extended decomposition of the fiber [35]. Cellulases can decrease roughness and increase smoothness, glossiness, and brightness [36].

Anoxic or anaerobic conditions were used by Jayaprabha et al. [52] and Wobiwo et al. [53] to successfully extract natural banana fibers from the pseudostem and peduncle, respectively. In particular, the bioextraction of banana pseudostem fibers using anoxic water demonstrated that the properties of the fibers were comparable to those of mechanical processing. However, a reduction in the tensile strength is observed with the prolongation of the extraction time from 6 to 12 days.

### 3.2. Mechanical/Physical Processes

**Decortication** is the mechanical removal of non-fibrous material from stalks, ribbons, or strips of the plant in order to collect the fibers [54]. This process may employ manual labor, hammer mills, fluted rollers, or willowing machines. Over the years, experts have created various decortication machine designs with varying capacities [40]. A common design consists of a spinning drum equipped with blades positioned around its circumference. These blades are attached to a shaft, which is then connected to an electric motor. The spinning of the drum produces a rhythmic and forceful motion that eliminates the pulpy substances [41]. Another popular design includes squeezing or feeding rollers and scraper rollers to extract the fibers [37,40,41]. In all cases, the extracted fibers are cleaned and dried, while further treatment is necessary to enhance the value of the fibers and reduce the presence of unwanted non-cellulosic residues [55].

The combined use of retting and mechanical means [56] corresponds to the process of **mechanical retting**. Here, the mechanical extraction of fibers is further supported by applying pre-retting that weakens the bonds between the fiber and the gum and facilitates their breakage by mechanical stress.

**Osmotic degumming** was applied by Konczewicz et al. [50] by implementing a continuous flow of water or frequent periodical changes. The method was applied to flax fiber and resulted in high-quality and fine fiber after a maximum of 96 days, indicating a reduced processing time as well. Water diffuses into the stem to cause swelling of the fiber, expansion of the pectin present, and dissolution of the soluble substances containing chromophores, bacteria, pectin, and mineral salts, while hydrostatic pressure further supports this process by weakening the bonds between the fiber and surrounding substances. The combined use of ultrasound shortens the time needed for optimum extraction to 48 h. A later study [51].

**Steam explosion** is an eco-friendly approach to degum different types of NCFs [42]. This treatment involves the utilization of highly pressurized saturated steam, which is introduced into a batch or continuous reactor along with lignocellulosic biomass for a brief

period of time (often a few minutes). It can be separated into two distinct phases: (1) a phase of steam boiling and (2) a phase of explosion. Pressures ranging from 0.5 to 1.5 MPa have been investigated in the case of kenaf and ramie [43,44] to induce hydrolytic degradation of the lignocellulosic matrix. The second phase of the process involves the transformation of heat energy into mechanical energy. This phenomenon entails an abrupt decrease in pressure, resulting in the expansion of vapor within the fibers and the disturbance of the fibrous arrangement [45]. As a result, it has the ability to eliminate a portion of the gummy substance from the raw materials, break down certain complex carbohydrates, and result into fiber bundles [46]. Nevertheless, the steam explosion process is not used as a standalone treatment to produce CNFs, as it results in the production of fiber bundles rather than NCFs and it is used as a highly effective pre-treatment process, which can be followed by chemical [43,44,46], enzymatic treatments or retting [56].

### 3.3. Chemical Processes

Chemical treatments for NCF production utilize a range of substances including alkalis (such as sodium hydroxide), hydrogen peroxide, salts (such as sodium percarbonate), organic acids, strong acids or a blend of both (such as oxalic and sulfuric acid) [37,47]. Chemical substances, commonly used as aqueous solutions, induce the degradation of hemicellulose, pectin, and lignin. Temperature, reaction duration, and chemical concentration [47] have been reported to impact the quality and effectiveness of chemical treatments for fiber extraction. Excessive treatment will cause NCFs to undergo degradation. The primary challenge in chemical processes is the removal and possible reuse of the chemicals, and the treatment and disposal of sewage.

### 3.4. Production of Agro-Waste NCF

Besides cotton, commercially exploited plant-based fibers include primarily jute, coir from coconut husks, flax, hemp, and sisal, accounting for over 6% of the global fiber market [18]. Abaca (manila hemp), kapok, ramie, agave fibers, and henequen have been explored as alternative sources to produce NCFs. At the same time, all agricultural crops generate residues such as stalks, leaves, husks, and straws in quantities that may surpass the primary agricultural product [57]. Table 2 shows research efforts to value agricultural residues and byproducts for NCF production. In the case of coir, mature coconut husks are the conventional source of coir fiber. Green husks, however, are readily accessible in large volumes and fall under the category of agro-waste [31].

**Table 2.** Paradigms of agro-waste NCFs in the literature.

Crop and Residue Raw Material	Production Process	Process Description	References
Banana Pseudostem	Anoxic biological extraction	Submersion of sheaths in anoxic water which is circulated via an anaerobic reactor for 6 or 12 days.	[52]
	Mechanical	(1) Use of a mechanical decorticator consisting of two feed and two scraper rollers; (2) Sun drying for 24 h.	
	Chemical	(1) Acid treatment with 2 g/L H <sub>2</sub> SO <sub>4</sub> solution at 55 °C for 2 h to remove wax; (2) Treatment with 7 g/L H <sub>2</sub> O <sub>2</sub> , 3% Na <sub>2</sub> SiO <sub>3</sub> , and 2% sodium polyphosphate at 95 °C for 1.5 h; (3) Treatment with 9 g/L NaOH solution at boiling temperature for 3 h; (4) Neutralization with 2 g/L H <sub>2</sub> SO <sub>4</sub> ; (5) Washing and drying at 105 °C for 24 h. Initial banana pseudostem mass: 10 g. Bath volumes: 200 mL.	[37]
	Enzymatic/chemical	(1) Delignification using trisodium citrate; (2) Treatment with protease aqueous solution with trisodium citrate as buffer; (3) Washing and centrifugation; (4) Mixing with pectinase enzyme; (5) Bleaching with H <sub>2</sub> O <sub>2</sub> in the presence of NaOH.	
	Seawater retting	(1) Retting in seawater for 1 to 5 weeks at room temperature (28 ± 5) °C; (2) Washing with sterile distilled water; (3) Cleaning with metallic hand brush.	[58]

Table 2. Cont.

Crop and Residue Raw Material	Production Process	Process Description	References
Abyssinia banana ( <i>Ensete ventricosum</i> )	Mechanical/retting/enzymatic	(1) Use of decortication machine; (2) Retting of the leaves; (3) Washing, cleaning and manual fiber separation; (4) Enzymatic treatment with 10% <i>w/w</i> amylase at 40 °C for 90 min.	[59]
Canola stem ( <i>Brassica napus</i> )	Water retting/chemical	(1) Over drying at 105 °C for 8 h; (2) Water retting at room temperature until separation from the exterior of the stem; (3) Alkaline treatment with 5% NaOH and 0.5% glycerin at 60 °C for 60 min, followed by washing and drying; (4) Acidic treatment with 4% acetic acid solution at 60 °C for 30 min, followed by washing and drying; (5) Treatment with 10% Tubingal 4748 softener at 40 °C for 30 min at pH = 4.5 in the presence of acetic acid, followed by washing and drying.	[60]
Green Coconut husk	Biological retting	(1) Soaking in 1% <i>w/v</i> ammonium dihydrogen phosphate solution; (2) Sterilization in autoclave; (3) Inoculation with <i>Aspergillus niger</i> fungus and incubation at 30 °C for 8–9 days.	[31]
Pineapple leaf	Chemical/mechanical	(1) Washing, cutting into 1–2 cm and oven drying at 70 °C (2) Treatment with 5% <i>w/w</i> sodium hydroxide solution at 90 °C for 180 min, solid-to-liquid ratio 1:20; (3) Washing until neutral pH and overnight drying at 60 °C; (4) Treatment with 5% <i>w/w</i> sodium chlorite at 70 °C for 90 min, pH 4–5; (5) Washing until neutral pH and drying at 60 °C.	[61]
<i>Prosopis juliflora</i> bark	Water retting	(1) Retting in seawater for 14 days; (2) Separation of fibers by traditional combing process and drying.	[62]
Sausage plant fruit ( <i>Kigelia Africana</i> )	Mechanical/Chemical	(1) Oven drying for 24 h; (2) Collection of the fibrous part via the use of a two-roller mechanical crusher; (3) Soaking in boiling water; (4) Treatment with 0.2 N NaOH solution for 90 min at 100 °C, 1:10 fiber-to-liquid ratio and washing until neutral pH; (5) Treatment with 10% acetic acid solution for 10 min and rinsing; (6) Drying at 110 °C for 3 h.	[63]
<i>Sida cordifolia</i> stem	Water retting	(1) Retting in seawater for 14 days; (2) Separation of fibers by traditional combing process and drying.	[64]
Soybean straw	Chemical	(1) Boiling in 8% <i>w/w</i> NaOH for 2 h; (2) Thorough washing to remove the dissolved substances; (3) Neutralization with dilute acetic acid 3% <i>w/w</i> ; (4) Washing and drying under ambient conditions.	[65]
Turmeric stem ( <i>Curcuma longa</i> L.)	Chemical	(1) Alkali treatment with NaOH solution 1M at 90 °C for 30 min, solid-to-liquid ratio 1:10; (2) Washing until neutral pH; (3) Immersion in 10% acetic acid for 10 min; (4) Washing and drying.	[57]
	Water retting	(1) Water retting at 20–28 °C for 3–4 weeks; (2) Manual separation of the fibers from the turmeric stem; (3) Washing and drying in the shade.	[66]
Wild tulsi stalk ( <i>Hyptis suaveolens</i> )	Chemical	(1) Alkali treatment with NaOH solution 1M at 85 °C for 60 min, solid-to-liquid ratio 1:10; (2) Draining and washing until neutral pH; (3) Immersion in 10% acetic acid for 10 min; (4) Washing and drying.	[67]

#### 4. 2nd Route Towards Cellulose Fibers: Step A. Extraction and Purification of CPs

Numerous methods have been developed and exploited for pulping wood. However, these are not applicable to non-wood agro-waste, where different treatment methods are used. Table 3 shows the drawbacks of the use of non-wood lignocellulosic waste in pulping. Current research explores the possible use of different agro-waste with methods that are environmentally friendly and lead to the production of high-quality end-products. In the case of textiles and compared to the paper industry, commercial MMCF production requires dissolving grade pulp (DGP) with a high alpha-cellulose content and a minimum amount



of inorganic and non-cellulosic organic content [68], a condition that adds to the intrinsic problems of pulping agricultural waste and creates new research fields for scientists [69]. Prominent methods for use with agricultural and food waste are currently being researched. Furthermore, advances in the production methods of MMCs demonstrate the possible use of paper-grade pulp [70,71] along with non-derivatizing or other innovative methods for the production of the fiber. Table 4 presents a comparative overview of major processes used to produce CP.

**Table 3.** Drawbacks of the use of non-wood lignocellulosic waste in pulping [69].

Issue	Description
Bulk density	The low bulk density of non-wood raw materials significantly impacts their logistics, leading to much smaller mill sizes compared to wood-based mills. The use of baling presses can act correctively.
Fiber length	Short fiber lengths affect drainage properties of the pulp and the degree of polymerization in dissolving pulps.
Chemical composition	Different chemical composition from wood with higher hemicellulose, silicon and nutrient content. Pre-treatment or other methodological adjustment is needed.
Dissolution of silicon	Dissolution of silicon into alkali cooking liquor has resulted in scaling problems in liquor evaporation and solvent recovery.
Metal ions presence	When the pulp undergoes chlorine-free bleaching, the transition elements generate radicals that react indiscriminately with the pulp, resulting in a decrease in both yield and strength qualities.

#### 4.1. Soda Pulping

The active chemical in soda pulping is sodium hydroxide. Invented in 1851, the method remains in use for easily pulped materials such as straws and some hardwoods [72], making it a favorable selection for lignocellulosic wastes. Soda pulping, unlike the commonly used kraft (sulfate) process for wood raw materials, produces no sulfur and no odorous sulfur-containing gases. Hydroxide ions cause hydrolytic cleavage of lignin into smaller fragments and dissolve lignin, hemicellulose, and other extractives from the raw material in a strongly alkaline cooking solution [73]. Sodium hydroxide specifically contributes to a considerable increase in biomass porosity and cellulose content by saponifying the ester linkages between lignin and hemicellulose, as well as disrupting the C–C bonds of lignin molecules. However, the higher amount of NaOH used results in the generation of a significant quantity of alkali black liquors, which in turn poses challenges for the subsequent treatment of wastewater [74]. Additionally, raising the concentration of NaOH can reduce the amount of pulp produced due to the proportional increase in the speed of the hydrolysis reaction caused by the higher concentration of hydroxide ions [75,76]. This results in the production of CP with a lower amount of amorphous material [77].

To optimize the pulping procedure, some additives may be used. Specifically, studies report that anthraquinone (AQ) reduces carbonate degradation, lowers the kappa number, boosts yield, and enhances the strength properties of the derived cellulose [72,78]. These properties are associated with a redox cycle, which transforms the aldehyde end groups of carbohydrates into aldonic acid groups, thereby halting further degradation of the carbohydrates. In addition, the generation of the reduced anthraquinone, anthrahydroquinone (AHQ), reacts with lignin and promotes the delignification process. AQ is not soluble in alkaline solutions, and mass transfer is considered the main barrier to its effectiveness as a catalyst [79]. However, the high cost of AQ often leads to its use at very low levels, often on an as-needed basis to increase pulp production.

Organic solvents like ethanol may further support lignin degradation. Ethanol and sodium hydroxide can act synergistically and improve the delignification ability of the cooking liquor [78].

#### 4.2. Pulping with Organic Solvents

Extensive evaluations of solvent pulping techniques have been conducted in laboratory and pilot experiments, using hardwood or softwood as raw material [80]. A variety of organic solvents, such as alcohols, acids, ketones, esters, and others, have been utilized. Solvent pulping can be combined with recovery techniques that lower the capital costs associated with solvent use and support by-product separation [81]. Pulping conditions should be controlled, since excessive acid-induced hydrolysis will reduce the length of cellulose chains and result in a decrease in the degree of polymerization as H<sup>+</sup> ions enter cellulose molecules and link with  $\beta$ -1,4-glycosidic bonds [82].

The Alcell process, a highly developed organosolv technology, employs 50% ethanol. The material is steamed and then cooked by circulating the main liquor, which was previously utilized as secondary liquor in the previous cook. After an hour, the primary liquid that has been used and contains dissolved lignin, hemicellulose oligomers, and furfural is removed from the extractor and sent to the product recovery section. Degradation products of hemicellulose include acetic and formic acid that can act as catalysts for delignification. After completing this stage, the extractors are filled with the secondary liquor, which already contains trace amounts of dissolved organics because it was employed as the final wash in the previous cooking process. Once the necessary amount of time has passed, the secondary liquid that has been utilized is removed from the extractors and stored in an accumulator. This liquid will then be used as the primary liquid in the next cooking process. Ultimately, the alcohol that has been purified and contains no solid particles is mixed with the cook as the third liquid, and then it is sent to the secondary accumulator. This is performed in a sequence of extractors that are operated at a temperature of 190 °C and a pressure of 28 bar. According to Roussu et al. [69], in order to prevent the buildup of organic acids in the cooking liquor and the resulting disruption of delignification, it is important to keep their concentrations at a low level. Therefore, it is necessary to separate the breakdown products from both water and ethanol. This separation mostly entails the process of distillation but is challenging and costly due to the low concentrations of the breakdown products and the formation of azeotropes with water.

The Organocell process is another method of organosolv pulping that combines treatment with methanol and NaOH. Delignification is achieved using a two-step cooking process. The initial phase entails the combination of methanol and water, whereas the subsequent phase involves the introduction of NaOH [83]. During the initial phase, wood chips are soaked in a solution containing 50% methanol at temperatures ranging from 170 to 190 °C for a duration of 20 to 40 min. Afterwards, the wood chips that have been partially stripped of lignin are introduced into a digester. In the digester, the concentration of methanol is reduced to 35% and sodium hydroxide is included in the liquid to aid in dissolving the remaining lignin. The second phase of the Organocell process is normally conducted at a temperature of 170 °C and a residence period of 40–60 min [84].

Formic acid and acetic acid have been shown to be effective in breaking down lignin  $\alpha$ -ether bonds and dissolving the resulting lignin fragments at relatively low to moderate temperatures. Pulping can be conducted at low temperatures and atmospheric pressure. Peroxides or HCl as a catalyst are commonly added to complete the delignification [85]. The use of formic acid in the acetic acid delignification process, known as the Formacell process, can increase delignification [69]. Formic acid is a stronger acid compared to acetic acid, and as pulping is facilitated at higher hydrogen ion contents, the incorporation of formic acid in the process leads to a further decrease in cooking temperature. Furthermore, the use of organic acids has the advantage that part of the degradation products are aliphatic acids, such as formic and acetic acid, which facilitates solvent recovery [86]. Other developed acid-based organosolv pulping processes include Acetosolv [69,84,87,88], which uses acetic acid >90% and HCl as a co-solvent at 90–110 °C, and Acetocell [84,89], which uses an aqueous mixture of acetic acid >80 wt.% at 170–190 °C. Of special notice is the formic acid pulping process developed by the company Chempolis Ltd. (Oulu, Finland) [69,90] that evolved into the biorefining fractionation technique Chempolis formico<sup>®</sup> that is used to

effectively separate biomass into its individual components, such as cellulose and produce even dissolving pulp. The method is capable of using a variety of raw materials, including forestry by-products, as well as non-wood/non-food agricultural residues such as straw, grasses, and bagasse. The delignification process can be executed in one step in less than an hour and with a large fiber-to-liquor ratio. Moreover, the method separates the cooking liquor from the pulp, fully recovers the cooking chemicals, and recycles the water. In contrast to other organosolv processes that have not yet seen large-scale commercialization, the Chempolis process has moved up from pilot scale [91] to industrialization [92] by using bamboo as the primary feedstock to produce mainly bioethanol and biochemicals rather than CP.

#### 4.3. Hydrogen Peroxide Use in Pulping

Hydrogen peroxide ( $H_2O_2$ ) is commonly employed in the bleaching phase of pulping procedures to eliminate remaining lignin and other chromophores responsible for causing discoloration.  $H_2O_2$  undergoes decomposition, resulting in the formation of water and oxygen. This process does not leave behind any toxic residues [93], making  $H_2O_2$  a more environmentally friendly chemical agent compared to chlorine-based alternatives, as the latter tend to produce harmful chlorinated by-products.  $H_2O_2$  dissociates into the peroxide anion ( $HOO^-$ ) in an alkaline environment, resulting in increased reactivity. This anion has a substantially higher level of efficacy in the decomposition of chromophores, which are the molecules responsible for the coloration of materials undergoing bleaching [94]. Moreover, the alkali has the ability to open up the structure of the fibers, specifically in textiles, which enables  $H_2O_2$  to penetrate in greater depth, leading to a more uniform and efficient bleaching process [95]. Taher and Cates [96] proposed a free radical mechanism in which the bleaching activity in alkaline solutions is attributed to the activity of hydroxyl ( $OH\bullet$ ) and perhydroxyl ( $HOO\bullet$ ) free radicals that are generated from the breakdown of hydrogen peroxide. The mechanism relies on the generation of free radicals via one-electron transfer and their subsequent interaction with pigmented substances, resulting in the removal of color.

$H_2O_2$  can be employed as an oxidizing agent in chemical pulping methods to facilitate the breakdown of lignin and hemicellulose. Typically, its use is part of a multistage process, and it is coupled with sodium hydroxide as an alkaline agent to improve the delignification and brightness of the pulp [74,97]. The side chains of lignin are broken down, while quinones undergo oxidation. Nevertheless, the polymerization degree of cellulose can be reduced through the reaction with hydrogen peroxide, resulting in the conversion of cellulose into oxycellulose [98].

$H_2O_2$  has the ability to be utilized at several points in the pulping and bleaching process, either by itself or in conjunction with other substances. This allows for adaptability in the design and optimization of the process [72]. As Llano et al. [99] demonstrated, a total chlorine-free bleaching sequence that incorporates  $H_2O_2$  together with  $O_3$ , NaOH, and  $O_2$  can modify the reactivity of the cellulose in the resulting dissolving pulp and increase the brightness and purity of the dissolving pulp. Changing conditions and concentrations of the reagents can also result in differentiated final products from unbleached acid sulfite pulp, namely viscose-grade pulp, nanocrystalline cellulose, and nanofibrillated cellulose.

The efficacy of  $H_2O_2$  as a pulping agent is dependent upon various process parameters, including pH, temperature, concentration, pulp-to-solution ratio, and the type of raw material. Precise management of these factors is essential to attain the best possible outcomes and avoid excessive deterioration of the cellulose fibers [72].  $H_2O_2$  is commonly used for bleaching and has an initial pH of approximately 11.5 when sodium hydroxide is utilized as the source of alkali. Increasing the amount of alkali or raising the temperature will result in a higher concentration of perhydroxyl anions. However, adding more alkali can also encourage decomposition and side reactions that cause the alkali to darken [94]. Similar to soda pulping, the presence of alkaline conditions aids in the elimination of

natural oils, waxes, and other impurities from the surface of the fibers, hence improving the overall whiteness and purity [72].

Overall, H<sub>2</sub>O<sub>2</sub> is a valuable chemical agent, mainly because of its ability to bleach and oxidize. Due to its environmental advantages, efficacy in enhancing pulp quality, and adaptability, it is extensively employed as an agent in the pulp sector. However, it is crucial to consider cost and process optimization when applying it.

#### 4.4. Use of Ionic Liquids in Pulping

Ionic liquids (ILs) are particularly effective in the dissolution of lignocellulosic biomass, which is prevalent in agricultural detritus. ILs are a group of organic salts that remain in liquid state without the presence of other solvents (water, organic ones, etc.) in a broad set of temperatures. The hydrogen bonding network in cellulose and hemicellulose is disrupted by the distinctive solvation properties of ILs, resulting in the effective separation of these components from lignin and the extraction of high-purity cellulose [100,101]. ILs offer environmental benefits as a result of their potential for recyclability and minimal volatility [102]. Conversely, the elevated expense of ILs in comparison to conventional solvents presents an immense obstacle. To render this technology economically viable, research is concentrated on the development of cost-effective ILs and synthesis methodologies, as well as to establishing sustainable processes for the recovery, efficient separation of products and co-solvents, and recycling of ILs. In addition, large-scale applications in engineering should be supported by favorable toxicity and mutagenicity testing [103,104]. The effectiveness of ILs depends on the biomass type and the IL that is employed, and the optimization of important parameters such as temperature, IL concentration, and reaction time is essential for maximizing the cellulose extraction and yield [100]. Utilizing ILs for the pulping of agricultural and food waste shows great potential as a sustainable and promising method for biomass processing. Despite the existing limitations of cost and scalability, continuous research on more economical ILs, optimal processing conditions, and efficient recycling technologies is expected to improve the feasibility of this technology in the future.

**Table 4.** Comparative overview of major processes used to produce CP.

Method	Short Description	Pros	Cons	References
Soda pulping	Treatment with NaOH.	<ul style="list-style-type: none"> <li>- High cellulose purity</li> <li>- Low cost</li> <li>- Use of a well-established process</li> <li>- Applied in existing facilities</li> <li>- Implementation on an industrial scale</li> </ul>	<ul style="list-style-type: none"> <li>- Black liquor production</li> <li>- Energy intensive</li> <li>- Wastewater treatment is required</li> </ul>	[74,105–108]
Organosolv	Treatment with organic solvents (e.g., ethanol, formic acid, acetic acid, ketones, esters).	<ul style="list-style-type: none"> <li>- Lower environmental impact</li> <li>- Closed-loop solvent recovery is possible</li> <li>- Water recycling.</li> </ul>	<ul style="list-style-type: none"> <li>- High solvent consumption</li> <li>- Not industrialized yet</li> </ul>	[69,81,83–91]
H <sub>2</sub> O <sub>2</sub>	Use of H <sub>2</sub> O <sub>2</sub> for delignification and bleaching.	<ul style="list-style-type: none"> <li>- Environmental friendliness (water and oxygen as byproducts)</li> <li>- Effectiveness</li> </ul>	<ul style="list-style-type: none"> <li>- Precise control of pH, temperature, and reagent concentration is required</li> <li>- Possible cellulose depolymerization</li> </ul>	[72,93–99]
IL treatment	<ul style="list-style-type: none"> <li>- Dissolution in ILs (e.g., imidazolium, phosphonium, triethylammonium) and phase separation</li> </ul>	<ul style="list-style-type: none"> <li>- Environmental advantages of ILs due to their recyclability and minimal volatility</li> <li>- Closed-loop solvent recovery is possible</li> </ul>	<ul style="list-style-type: none"> <li>- Cost of ILs</li> <li>- Small scale</li> <li>- Precise control of reaction, temperature, and reagent concentration is required</li> </ul>	[100–103,109,110]

#### 4.5. Production of Agro-Waste CP and DCP

In the frame of the circular economy, researchers have studied the implementation of the aforementioned processes into different agro- and food-waste materials to develop CP, DCP and cellulose nanofibrils (CNFs). Table 5 presents relevant paradigms based on the raw materials and treatments selected and tested.

**Table 5.** Agro-waste CP and DCP in the literature.

Source	Major Production Process	Process Description	Product	Final Product	References
Agave bagasse ( <i>Agave tequilana</i> )	Chemical treatment	(1) Acid hydrolysis with 0.5% H <sub>2</sub> SO <sub>4</sub> at 160 °C for 30 min, with a hydromodule of 8:1; (2) Cooking with 23.2% NaOH and 0.1% AQ 0.1% at 170 °C for 2.5 h, with a hydromodule of 5:1; (3) Bleaching with 10% w/w pulp, 1.7% w/w ClO <sub>2</sub> and 0.15% w/w HCl 1.0 N at 60 °C for 0.5, pH 2–3; (4) Extraction with 5% w/w NaOH 1.0 N at 70 °C for 1 h, pH ≥ 10.5; (5) Bleaching with 1% w/w ClO <sub>2</sub> and 0.15% w/w NaOH 1.0 N at 80 °C for 3 h; (6) Treatment with 1.5% w/w H <sub>2</sub> O <sub>2</sub> and 1.0% w/w NaOH 1.0 N at 80 °C for 3 h, pH 11.5–11.8; (7) Removal of hemicelluloses with 0.5 g of NaOH per 100 g of dry pulp at 25 °C for 0.5 h. Final alpha-cellulose content: 89% w/w, yield: 15%	CP	Thermoplastic starch/cellulose nanofibrils nanocomposites	[111]
Agave leaves	Organosolv: Acetosolv	(1) Treatment with 80–95% acetic acid at boiling temperature and addition of 0.05–0.3% w/w HCl for 30–90 min, solid-to-liquid ratio 1:10; (2) Filtration into medium-porosity glass crucibles and washed with acetic acid ×4; (3) Washing until neutral pH and drying at room temperature.	CP	Paper	[88]
Coconut mesocarp ( <i>Cocos nucifera</i> )	Chemical treatment	(1) Drying, crushing and cutting; (2) Pre-washing: ×2 washing with distilled water for 10 min; ×2 treatment with water at 50 °C for 2 h; filtering; (3) Treatment ×2 with 2% NaOH solution for 2 h at 80 °C, filtering, washing and drying at 50 °C for 24 h; (4) Bleaching with NaClO <sub>2</sub> in the presence of glacial acetic acid at 60–70 °C, cooling, filtering and washing; (5) Treatment with 0.05 N HNO <sub>3</sub> for 1 h at 70 °C; screening and washing; (6) Treatment with H <sub>2</sub> O <sub>2</sub> at different concentrations 0.5–3% v/v at 45 °C for 15 h, pH 11.5; (7) Alkaline treatment with 10% w/v NaOH and 1% w/v Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> at 28 °C for 15 h; (8) Treatment with 80% v/v CH <sub>3</sub> COOH and 70% v/v HNO <sub>3</sub> (10:1 ratio) at 120 °C for 15 min; (9) Acid hydrolysis with 64% H <sub>2</sub> SO <sub>4</sub> at 45 °C for 30 min and washing; (10) Centrifugation at 10,000 rpm for 10 min; (11) Dialysis with water for 3 days until constant pH.	CNF	Sewing thread via electrospinning	[112]
Corn stover	IL	(1) Separate dissolution with imidazolium ILs at 80 °C for 3 h, solid-to-liquid ratio 1:10 w/w: (a) 1-dodecyl-3-methyl-imidazoliumbis-(2,4,4-tri-methyl-pentyl)-phosphinate, [C <sub>12</sub> mim][( <sup>i</sup> C <sub>8</sub> ) <sub>2</sub> PO <sub>2</sub> ] and (b) 1-Decyl-3-methyl-imidazolium bis-(2,4,4-tri-methyl-pentyl)-phosphinate, [C <sub>10</sub> mim][( <sup>i</sup> C <sub>8</sub> ) <sub>2</sub> PO <sub>2</sub> ]; (2) Addition of water and hexane to form a microemulsion; (3) Centrifugation at 4400 rpm for 15 min for phase separation; (4) Washing with methanol and drying overnight at 105 °C.	CP	-	[109]
		(1) Separate dissolution with phosphonium ILs at 80 °C for 3 h, solid-to-liquid ratio 1:10 w/w: (a) Tetra-butyl-phosphonium acetate, [P <sub>4444</sub> ][CH <sub>3</sub> COO], (b) tri-butyl-methyl-phosphonium acetate, [P <sub>4441</sub> ][CH <sub>3</sub> COO], (c) tetra-butyl-phosphonium 2-ethyl-hexanoate, [P <sub>4444</sub> ][CH <sub>3</sub> (CH <sub>2</sub> ) <sub>3</sub> CH(C <sub>2</sub> H <sub>5</sub> )COO] (2) Addition of methanol; (3) Centrifugation for phase separation; (4) Washing with methanol ×4 and drying overnight at 105 °C.			



Table 5. Cont.

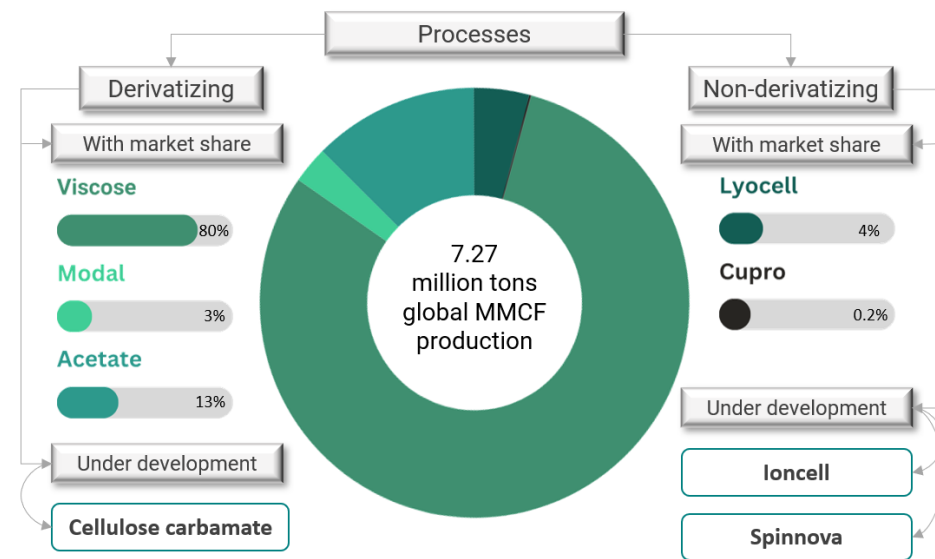
Source	Major Production Process	Process Description	Product	Final Product	References
Hemp shives/ isolated fibers	Soda pulping/ hydrogen peroxide	(1) Treatment with 3.5–4.0% <i>w/w</i> NaOH at 160–170 °C for 105–150 min; (2) Washing with 20% acetic acid (pulp density 3% <i>w/w</i> ); (3) Neutralization and bleaching (pulp density 10% <i>w/w</i> ) with 1% NaOH and max 5% H <sub>2</sub> O <sub>2</sub> optimally at 85 °C for 30 min.; (4) Neutralization and repetition of step (2) and washing until neutral pH. <i>Hemp pulp (shives based): alpha-cellulose content: 87%, DP: 559</i> <i>Hemp pulp (fiber based): alpha-cellulose content: 96%, DP: 656</i> <i>Oil flax pulp: alpha-cellulose content: 91%, DP: 542</i>	DCP	Lyocell fiber via NMMO dissolution and air gap spinning	[113]
Mango tegument	Alkali treatment	(1) Alkaline treatment of grounded tegument with 2% or 4% <i>w/v</i> NaOH at 80 °C for 60 or 120 min, solid-to-liquid ratio 1:20; (2) Washing with distilled water and vacuum filtering until neutral pH; (3) Drying at 50 °C for 24 h; (4) Bleaching with 30% <i>v/v</i> H <sub>2</sub> O <sub>2</sub> and 4% <i>w/v</i> NaOH at 50 °C for 2 h, solid-to-liquid ratio 1:20; (5) Washing with distilled water until neutral pH; (6) Drying at 50 °C for 24 h. <i>Holocellulose content: 73–75% w/w, yield: 37–42%</i>	CP	-	[77]
	Organosolv	(1) Acetic acid treatment with 1:1 or 1:2 CH <sub>3</sub> COOH:H <sub>2</sub> O <sub>2</sub> at 60 or 70 °C for 1 or 2 h, solid-to-liquid ratio 1:20; (2) Washing with distilled water and vacuum filtering until neutral pH; (3) Drying at 50 °C for 24 h. <i>Holocellulose content: 78–88% w/w, yield: 47–48%</i>			
Mango seed	Alkali treatment	(1) Grinding and sieving using a mill and an 18-mesh screen; (2) Alkali treatment with NaOH 2% <i>w/w</i> at 100 °C for 4 h × 4; (3) Washing until neutral pH and drying at 40 °C for 24 h; (4) Bleaching with 1:1 ( <i>v/v</i> ) acetate buffer: 1.7% <i>w/w</i> NaClO <sub>2</sub> at 80 °C for 6 h × 2; (5) Washing until neutral pH and drying at 40 °C for 24 h × 2. <i>Holocellulose content: 86% w/w, yield: 29%</i>	CP	Cellulose nanocrystals via acid hydrolysis	[114]
Orange	Hydrogen peroxide/ organosolv/ Soda pulping	(1) Separation of albedo from flavedo via dissolution in toluene and ethanol; (2) Cooking with 1–3% <i>v/v</i> H <sub>2</sub> O <sub>2</sub> at 65–80 °C, pH 11–12 and filtering; (3) Treatment with acetic acid and formic acid in the presence of 0.5–6% <i>w/w</i> H <sub>2</sub> O <sub>2</sub> at 65–90 °C; (4) Alkali treatment with 0.5–6% <i>w/w</i> NaOH at 90–110 °C followed by filtration; (5) Washing and drying. <i>Alpha-cellulose content ≥ 90% w/w, yield ≥ 10%</i>	DCP	-	[97]
Oil palm empty fruit bunches	Soda pulping	(1) Pre-treatment with 0.1% <i>w/v</i> H <sub>2</sub> SO <sub>4</sub> at 150 °C for 2 h and filtering; (2) Alkaline processing using 10% <i>w/v</i> NaOH at 150 °C for 2 h, solid-to-liquid ratio 1:7; (3) 1st bleaching using 0.5% NaClO <sub>2</sub> in the presence of CH <sub>3</sub> COOH at 80 °C for 1.5 h, solid-to-liquid ratio 1:12, followed by filtration; (4) 2nd bleaching using 10% H <sub>2</sub> O <sub>2</sub> at 70 °C for 5 h, solid-to-liquid ratio 1:12, followed by filtration; (5) Washing until neutral pH. <i>Oil palm pulp: cellulose: 72.38% w/w, hemicellulose: 8.11%, lignin: 12.09% w/w; rice straw pulp: 76.10% w/w, hemicellulose: 5.4%, lignin: 3.2% w/w</i>	CP	Cellulose acetate via acetylation	[108]
Paddy ( <i>Oriza sativa</i> ) rice straw					
Pineapple leaf	Alkali/acid treatment	Treatment according to steps (1)–(5) of the NCFs production process (see Table 2). (6) Hydrolysis with 3.5 M HCl at 70 °C for 12 h and washing until neutral pH; (7) Overnight hydrolysis with 7.5 M HCl and washing until neutral pH.	CP	Microcellulose by homogenization and ultrasonic action	[61]

Table 5. Cont.

Source	Major Production Process	Process Description	Product	Final Product	References
Sugarcane bagasse	Oxidizing/Alkali treatment	(1) Dewaxing with toluene-EtOH (2:1, <i>v/v</i> ) in a Soxhlet apparatus; (2) oven drying at 60 °C for 16 h; (3) Removal of water-soluble components by treatment with H <sub>2</sub> O at 55 °C; (4) Filtering and washing until neutral pH; (5) Delignification using acidified 1.3% NaClO <sub>2</sub> , pH 4; (6) Treatment with 10% KOH and 10% NaOH at 20 °C; (7) Washing with EtOH/H <sub>2</sub> O and drying at 60 °C for 16 h.	CP	- Cellulose nanocrystals with acid hydrolysis - CNF with ball milling	[115]
	Soda pulping	Treatment with 10% NaOH with 1% consistency at 90 °C for 24 h Treatment with 19.2% NaOH and 0.1% AQ at 170 °C for 1.5 h, solid-to-liquid ratio 1:10.	DCP	MMCF via wet spinning	[3]
South African sugarcane bagasse	IL	(1) Treatment with protic IL triethylammonium hydrogen sulfate [TEA][HSO <sub>4</sub> ]:H <sub>2</sub> O 4:1 <i>w/w</i> at 120 °C for 1–24 h, solid-to-liquid ratio 1:10 <i>w/w</i> ; (2) Ethanol wash ×4; (3) Soxhlet extraction and air-drying.	CP	Enzymatic hydrolysis towards glucose and xylose	[110]
Rice straw	Soda pulping	(1) Mixing with NaOH 12% (optimal concentration) at 121 °C and 1 bar for 1 h; (2) Removal of soluble lignin by centrifugation at 5000 rpm for 10 min; (3) Drying overnight at 60 °C; (4) Bleaching with a 5% <i>w/v</i> NaClO <sub>2</sub> in 1 M glacial acetic acid at 70 °C for 10 min, solid-to-liquid ratio 1:10.	CP	Ball milling to nanofibers	[116]
	Steam explosion	Treatment in a high-pressure reactor under 10–15 bar at 180 °C for 10 min.			
	Organsolv	Treatment with formic acid and ethanol 3:1 at a solid-to-liquid ratio 1:10 at 160 °C for 1 h, solid-to-liquid ratio 1:10			
Tea stem	Soda pulping	(1) Grinding and sieving (150–210 μm); (2) Washing and filtering to remove water-soluble impurities; (3) Treatment with 4% NaOH solution at 80 °C ×3; (4) Bleaching with H <sub>2</sub> O <sub>2</sub> or NaClO <sub>2</sub> 1.7% <i>w/w</i> in acetic acid buffer at 80 °C ×4; (5) Oven drying at 105 °C.	CP	Cellulose aerogel after hydrolysis, regeneration and freeze-drying	[117]
Wheat straw Elephant grass	Organosolv/Chempolis	(1) Delignification using formic acid, less than 1 h; (2) Alkaline bleaching with oxygen-based chemicals such as oxygen, ozone, H <sub>2</sub> O <sub>2</sub> and peracetic acid; (3) Recovery of formic acid through: evaporation and drying; partial distillation of water to avoid accumulation; and hydrolysis of esterified formic acid during delignification.	CP	-	[69]

## 5. 2nd Route Towards Cellulose Fibers: Step B. Production of MMCFs

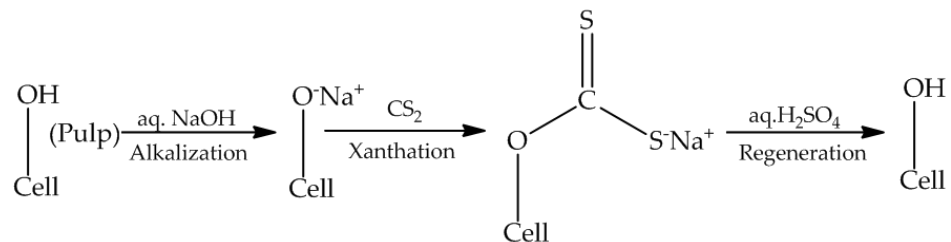
Cellulose is insoluble in water or most organic solvents because of the way its chemical structure is made up of crystalline areas and hydrogen bonds between and within molecules [118,119]. Despite this, researchers have created numerous manufacturing methods for regenerating cellulose fibers over the years and are constantly researching their optimization. MMCFs accounted for around 6% of the overall fiber production volume, with an annual production volume of around 7.3 million metric tons in 2021 [120]. The procedures are commonly classified based on the mechanism of cellulose dissolution in the solvent system. Two types are distinguished: derivatizing and non-derivatizing. In derivatization processes, cellulose is changed chemically to make an intermediate cellulose derivative that is soluble, like cellulose xanthate [121,122]. Conversely, in non-derivatization processes, the solvent system directly dissolves cellulose by breaking the hydrogen bonds [123]. These methods are summarized in Figure 4.



**Figure 4.** Different cellulosic regeneration processes. Market share data derived from Textile Exchange, 2023 [120].

### 5.1. Derivatizing Processes

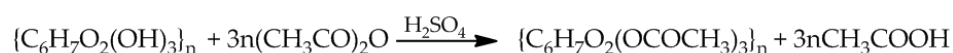
Viscose is the predominant MMCF, accounting for approximately 80% of the total market share of all MMCFs [11,120]. When additional processing is introduced to increase wet strength and softness, it leads to the production of modal fibers [124]. The viscose process (Figure 5) involves the following main stages: (1) dissolution and swelling of the wood pulp, where cellulose transforms into sodium cellulose (alkali cellulose); (2) pre-aging, where alkali cellulose undergoes storage under specific conditions (oxygen, temperature) to undergo oxidation; (3) xanthation, where alkali cellulose reacts with carbon disulfide to produce sodium cellulose xanthate; (4) dissolution of the xanthation product in caustic soda to produce the spinnable solution known as viscose; (5) filtration; (6) spinning; and (7) washing [125,126]. Nevertheless, viscose production has significant environmental impacts because it is both a water- and chemical-intensive process that generates significant liquid and solid waste, including sludge from wastewater treatment [125]. Viscose production entails the use of hazardous solvents and chemicals, such as carbon disulfide, resulting in the creation of toxic substances and gases (volatile organic compounds, hydrogen sulfide). This underscores the necessity for thorough chemical management [17]. Currently, advanced closed-loop manufacturing systems that recycle solvents and chemicals can limit, but not eliminate, the release of dangerous substances [125,127].



**Figure 5.** Cellulose reactions during the viscose process.

Cellulose acetate has a market share of approximately 13% [11,120]. However, the textile industry only uses 5% of the total produced acetate fibers, with the remaining going to non-textile applications like cigarette filters, photographic film, and as a component of adhesives [17,128]. The manufacturing process typically entails combining cellulose with acetic anhydride in the presence of sulfuric acid (acting as a catalyst) to create cellulose acetate, as can be seen in Figure 6. The acetate is subsequently subjected to partial hydrolysis to achieve the necessary level of substitution [129]. To produce fibers, cellulose acetate is

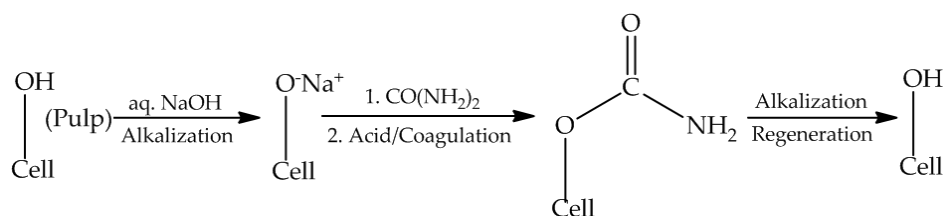
dissolved in acetone to create the spinning dope [130]. It is then subjected to dry spinning, where the polymer solution is extruded into a chamber while the solvent evaporates, leaving solid fibers [131]. In theory, cellulose acetate has the potential to decompose into cellulose and acetic acid when exposed to water in the natural environment. In reality, the rate at which the material breaks down naturally is determined by the level of acetyl substitution and the specific conditions that it is exposed to. As the acetyl substitution increases, the biodegradation rate decreases [132]. Factors such as molar mass, crystallinity, physical form, the presence of impurities, and electron acceptors also influence the degradation rate. Thus, cellulose acetates are not classified as fully biodegradable, which is seen as a significant benefit for many uses [23]. Nevertheless, these materials can be easily recycled using traditional alkaline treatment, which effectively turns them back into cellulose.



**Figure 6.** Cellulose acetylation.

Research has demonstrated that acetylation of cellulose can be conducted in ILs. Wu et al. [133] reported a one-step cellulose acetylation in 1-allyl-3-methylimidazolium chloride ([Amim][Cl]) without catalyst, yielding cellulose acetate with a controllable degree of substitution. The yield of cellulose acetate reached 86% when the reaction took place in 1-N-butyl-3-methylimidazolium chloride ([C<sub>4</sub>mim][Cl]) at 80 °C for 2 h without using any catalyst [134].

The cellulose carbamate process is based on the chemical reaction between cellulose and urea that forms cellulose carbamate [135]. The mechanism involves the decomposition of urea into isocyanic acid when the reaction temperature is higher than the melting temperature of urea (133 °C) and cellulose reacts with isocyanic acid to form cellulose carbamate [71]. Isocyanic acid is a colorless, volatile, and toxic chemical that undergoes hydrolysis to produce carbon dioxide and ammonia. Carbamation must be conducted in a sealed container to ensure safety, and any resulting gases, particularly ammonia, should be rinsed away to eliminate them. The production process (Figure 7) [71,135,136] involves an initial step of alkalizing to swell and partially degrade the cellulose. Then, it reacts with urea at a temperature range of 135 to 175 °C to form cellulose carbamate having nitrogen content of 2.5–4.5% and degree of polymerization (DP) of 250–400. Next, the carbamate groups (maximum 10% concentration) undergo hydrolysis at high temperature in an alkaline decomposition bath that contains salt. The resulting solution is filtered and dissolved air is removed before being wet spun in an acidic precipitation bath. The solidified fibers need to be regenerated by immersing them in a dilute alkali solution with a concentration of less than 1%. This process removes the carbamate group and releases NH<sub>3</sub>.

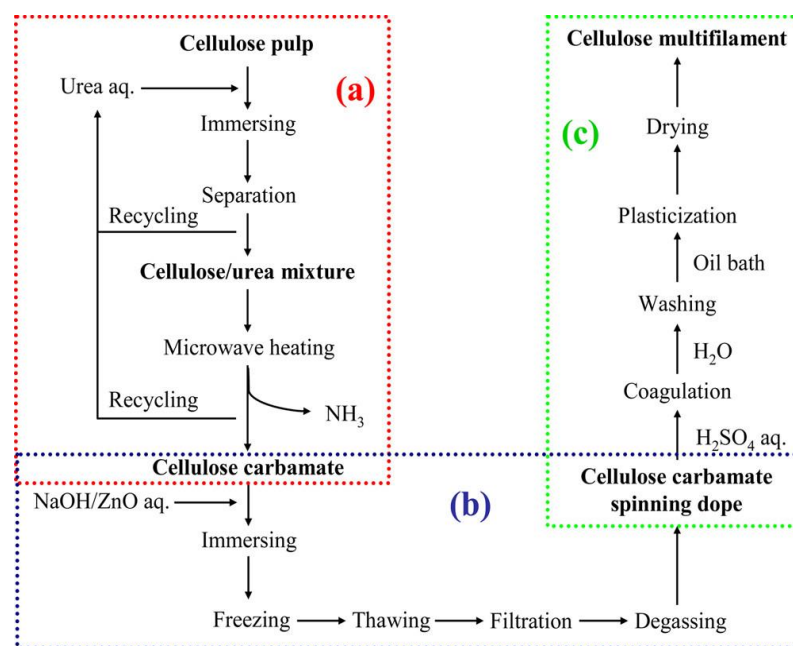


**Figure 7.** Cellulose reactions during the carbamate process.

The technological process of the cellulose carbamate technique closely resembles the viscose method and can be retrofitted in viscose factories, with the exception that it does not include the use of a hydroxide solution containing sulfur [136]. Cellulose carbamate is more stable than cellulose sulfonate sodium, making it possible for the dissolution and spinning processes to be separated [137].

The patented CarbaCell procedure utilizes a unique method of synthesizing carbamate technology by employing xylene as the transfer medium [137]. Moreover, a microwave-assisted process was successfully applied both at a laboratory [138] and on a pilot scale [139],

with Figure 8 demonstrating the flow chart of the pilot scale. With the application of microwave irradiation, the cellulose conversion is efficiently achieved within a short time period without the need for a catalyst or solvent, thanks to a very manageable heating process. The researchers discovered that the solubility of cellulose carbamate could be greatly enhanced by adding a small quantity of ZnO to the NaOH solution. The microwave reactor-assisted method was also employed to produce cellulose carbamate from kenaf core pulp, which is often treated as agro-waste [140].



**Figure 8.** Green method for production of cellulose multifilament from cellulose carbamate on a pilot scale. (a) Microwave-assisted synthesis of cellulose carbamate, (b) dissolution of cellulose carbamate in NaOH/ZnO aqueous solution, and (c) preparation of cellulose multifilament from spinning dope [138].

### 5.2. Non-Derivatizing Processes

The Lyocell method is an alternative method for making yarn from regenerated cellulose. The production method directly dissolves the cellulose in an aqueous solution of N-methylmorpholine N-oxide (NMMO) without using additional solvents. The Lyocell process, unlike the viscose process, involves fewer manufacturing steps and does not produce derivatives or use hazardous additives [141]. Additionally, NMMO is considered a green solvent [142] and is 99% recyclable in the Lyocell process. However, the cyclic ether structure of NMMO is prone to thermal runaway reactions, which leads to safety concerns and necessitates the use of stabilizers. The production process includes the following steps: (1) dissolution, (2) filtration, (3) spinning, (4) washing, (5) finishing, and (6) drying [143]. The manufacturing of Lyocell fibers uses spinnerets, which are durable in high-pressure conditions. Spinning is performed using the wet method, where the spinning dope is passed via an air gap (known as dry-wet spinning) into the aqueous coagulation bath where the fiber starts to form. The spinning process occurs at a rate of 75–100 mg/min, with increased drawing of the spinneret [144]. Given the quality of the final fabric and its environmental benefits, the Lyocell method has gained commercial use [14] and is currently the third most frequently used man-made cellulosic fiber after viscose and acetate cellulose, with a market share of approximately 4% [11].

In the second half of the 19th century, researchers developed the cuprammonium process, which involves dissolving purified cellulose in an ammoniacal solution of Cu(II) hydroxide, also known as Schweizer's reagent [145]. This process is presented in Figure 9. The cuprammonium complex can break the intra- and intermolecular hydrogen bonding of



cellulose [146] but does not cause chemical modification. To enhance the spinning process, several substances, including glycerin, glucose, tartaric acid, citric acid, oxalic acid, and cane sugar, are added to the cuprammonium solution [147]. Additional production steps include filtration and extrusion of the spinning dope via spinneret into a coagulation bath of dilute acid (e.g., acetic, sulfuric, etc.), recovery of the ions of copper and ammonium from the coagulation bath, and collection of the regenerated cellulose filaments after drawing, washing, finishing, and drying. Although cuprammonium is an established process for the production of regenerated cellulosic fibers, economic as well as environmental considerations have led to a production shrinkage, arriving at a 0.2% market share of the total MMCF market in 2021 [11].



**Figure 9.** Cellulose dissolution in the cuprammonium process.

As mentioned, ILs are capable of direct dissolution of cellulose, a property that has been explored by scientists in the formation of MMCFs. There are several ILs that have been studied for the production of cellulosic fibers, such as 1-Allyl-3-methylimidazolium chloride ([AMIM][Cl]), 1-Butyl-3-methylimidazolium chloride ([BMIM][Cl]), 1-ethyl-3-methylimidazole chloride ([EMIM][Cl]), 1-ethyl-3-methylimidazolium acetate ([EMIM][OAc]), 1,5-diazabicyclo non-5-enium acetate ([DBNH][OAc]), and 1-butyl-3-methylimidazolium acetate ([BMIM][OAc]) [109–113]. All have a melting point below 100 °C, low vapor pressure, and high thermal stability, which are favorable parameters in the preparation of dope and spinning procedure. Drawbacks in the commercialization of the use of ILs to produce MMCFs can be considered the high viscosity of the spinning dope that requires great mechanical pressures, the poor recycling of the solvent, the thermal instability and side reactions when recycling ILs, and the initial cost of ILs.

The Ioncell-F process produces high-quality, sustainable cellulose fibers from various raw materials by employing [DBNH][OAc] to dissolve cellulose and subsequently regenerate it into filaments [148,149]. It is an innovative method developed by researchers at Aalto University and the University of Helsinki that has entered the pilot scale. It follows the philosophy of the Lyocell process by combining it with the favorable properties of ILs. The process offers the advantage of spinning dope with higher cellulose content (15–17%), lower temperature at the dissolution (80 °C, 100 mbar) and spinning steps (70 °C dope, cold water coagulation bath), less cellulose degradation during the complete process, and great tenacity properties [148,150]. A newer patent [151] promotes the use of ILs containing cationic 1,5,7-triazabicyclo[4.4.0]dec-5-enium [TBDH]<sup>+</sup> moiety such as 1-methyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene acetate [mTBDH][OAc] that demonstrates very good stability during its recovering process when water is distilled (80 °C) and excellent viscoelastic properties when the cellulose/[TBDH] solution is subjected to dry-jet wet spinning. As a novel procedure, Ioncell technology faces scalability challenges which are linked to the establishment of infrastructures, the cost of ILs, the energy consumption at the solvent recovery phase, and the fact that the long-term stability of ILs under industrial processing conditions has not been tested yet [148,150,152,153].

Spinnova PLC patented technology mechanically refines pulp raw material and converts it into spinning-ready fiber suspension without the use of chemicals, dissolving, or regeneration processes [154,155]. Under high pressure, the suspension transforms into filament by passing through a specially designed nozzle. Extrusion mechanics cause the fibrils to rotate and align in accordance with the flow, resulting in the formation of a textile fiber with inherent properties [156]. The technology supports circularity by combining low carbon emissions, the recyclability of the fibers, and closed-loop water and energy recovery systems. Furthermore, the Spinnova process is reported to be applicable in agricultural waste such as wheat straw and cotton waste [155], although the current commercial product SPINNOVA<sup>®</sup> fiber is produced by wood pulp coming from FSC-certified tree farms [157].

Table 6 shows a comparative summary of the major regeneration routes for the production of MMCFs.

Table 6. Comparative summary of major regeneration routes for the production of MMCFs.

Process/Chemical Agent	Spinning Method	Pros	Cons	References	
Derivatizing	Viscose/Carbon disulfide	Wet spinning	<ul style="list-style-type: none"> <li>- Silk-like appearance.</li> <li>- Cost-effective.</li> </ul>	<ul style="list-style-type: none"> <li>- Energy cost: spinning process at elevated temperature.</li> <li>- Hazardous effluents (CS<sub>2</sub>, NH<sub>3</sub>): not eliminated by closed-loop process.</li> <li>- Low wet modulus.</li> </ul>	[17,125–127]
	Cellulose acetate	Dry spinning	<ul style="list-style-type: none"> <li>- Silk-like appearance.</li> <li>- Biodegradable.</li> <li>- Solvent recovery.</li> <li>- Susceptible to damage from acetone and other solvents.</li> <li>- Heat sensitivity.</li> </ul>	<ul style="list-style-type: none"> <li>- Addition of several enhancers.</li> <li>- Loss of strength when wet.</li> <li>- Environmental impact of the use of acetic acid and sulfuric acid.</li> <li>- Emission of volatile organic compounds: not eliminated by closed-loop process.</li> </ul>	[132]
	Carbamite/Urea	Wet spinning	<ul style="list-style-type: none"> <li>- Compatibility with viscose spinning machines.</li> <li>- Shorter production cycle than viscose.</li> <li>- Use of urea instead of CS<sub>2</sub>.</li> <li>- Spinning process conducted at room temperature.</li> <li>- Stability of the derivative</li> </ul>	<ul style="list-style-type: none"> <li>- High energy cost due to elevated temperatures.</li> <li>- Need for management of the byproducts formation (ammonia isocyanate, biuret).</li> <li>- Pilot-scale production.</li> </ul>	[135–138,158]
Non-derivatizing	Lyocell/NMMO	Dry-jet wet spinning	<ul style="list-style-type: none"> <li>- Biodegradability and recyclability of NMMO.</li> <li>- Very high-rate recovery of NMMO.</li> <li>- High-quality fibers.</li> <li>- Shorter production circle than viscose</li> </ul>	<ul style="list-style-type: none"> <li>- High energy cost: dissolution and spinning process performed at elevated temperatures; recovery of NMMO via evaporation.</li> <li>- High initial cost of NMMO.</li> <li>- Specialized equipment is required.</li> <li>- Need for stabilizers to limit cellulose and NMMO degradation and the chance of thermal runaway reactions.</li> <li>- Fibrillation of fibers.</li> </ul>	[141,148,149,151]
	Cuprammonium	Wet spinning	<ul style="list-style-type: none"> <li>- Lustrous and strong.</li> <li>- Produced in extremely fine deniers.</li> </ul>	<ul style="list-style-type: none"> <li>- Relatively expensive compared to viscose.</li> <li>- Degradation and weakening after exposure to sunlight in the presence of oxygen and moisture.</li> <li>- Wastewater polluted with copper.</li> <li>- Ammonia presence in gas emissions.</li> </ul>	[71,135,145,147]
	Ioncell/[DBNH][OAc] and [mTBDH][OAc]	Dry-jet wet spinning	<ul style="list-style-type: none"> <li>- Lack of runaway reactions.</li> <li>- Lower energy consumption than Lyocell at the dissolution and spinning phases.</li> <li>- Excellent viscoelastic properties of [mTBDH][OAc] upon dry-jet wet spinning.</li> <li>- Increased stability of [mTBDH][OAc] at solvent recovery conditions.</li> <li>- High-quality fibers.</li> </ul>	<ul style="list-style-type: none"> <li>- Specialized equipment required.</li> <li>- High initial cost of ILs.</li> <li>- High energy costs to recover ILs.</li> <li>- Pilot-scale production.</li> <li>- Instability of [DBNH][OAc] during recovery process.</li> </ul>	[148–153]
Spinnova	Aqueous suspension spinning	<ul style="list-style-type: none"> <li>- No use of chemicals.</li> <li>- Closed-loop water use.</li> <li>- Heat recovery from the process.</li> <li>- Zero waste technology.</li> <li>- Recyclable without loss of properties.</li> <li>- Entering commercial scale</li> </ul>	<ul style="list-style-type: none"> <li>- Specialized equipment required.</li> </ul>	[154–156]	

## 6. Agricultural and Agro-Industrial Waste Based Products in the Textile Industry

Table 7 displays existing branded cellulosic products derived from agricultural by-products and waste in the textile industry. These items can serve as replacements for other textile materials that have a significant negative influence on the environment while also creating additional sources of revenue for subsistence farmers.

In addition, Georgia-Pacific LLC operates a cotton linter pulp mill. This pulp can be regenerated into textile fibers.

At a research level, few additional studies are found in the literature that result in the production of MMCFs and are reported in Table 8.

**Table 7.** Branded/trademarked cellulosic textile products derived from agricultural and food-based waste for fashion.

Product Name	Main Source	Fiber Type	Process	Company	References
Agraloop™ BioFibre™	CBD hemp and oilseed flax	NCF	Decortication and Agraloop™ processing (wet treatment)	Circular Systems S.P.C. (Los Angeles, CA, USA)	[159]
Alt Fibres	Food crops, e.g., hemp oil seed, banana fruit, pineapple fruit	NCF	-	AltMat Pvt. Ltd. (Ahmedabad, India)	[160]
Bemberg™	Cotton linter as byproduct of cottonseed oil	MMCF	Cuprammonium	Asahi Kasei Corporation (Tokyo, Japan)	[161]
Lyohemp®	Hemp shives	MMCF	Chemical pulp/Lyocell process	TITK (Rudolstadt Germany), STFI (Chemnitz, Germany), FUDI GmbH & Co. KG (Zeulenroda-Triebes, Germany), MATRAK Service und Lohnarbeits GmbH (Auma-Weidatal, Germany) and Institute of Plant and Wood Chemistry (IPWC) (Dresden, Germany)	[162,163]
Orange fiber	Citrus juice by-products	MMCF	Chemical pulp/Acetate	Orange Fiber s.r.l. (Catania, Italy)	[11]
Orange fiber & TENCEL™ *			Chemical pulp/Lyocell process	Orange Fiber s.r.l. in collaboration with Lenzing AG (Lenzing, Austria)	[164]
Piñatex®	Waste pineapple leaf fiber	NCF	Enzymatic treatment	Ananas Anam Ltd. (London, UK)	[165]
Pineapple Leaf Fibers (PALF)	Pineapple leaf byproducts	NCF	-	Nextevo™ (Singapore, Singapore)	[166]
PYRATEx® element 2	Banana agro waste	NCF	Mechanical extraction	PYRATES Smart Fabrics SL (Madrid, Spain)	[167]
PYRATEx® citrea	Orange peel agro waste	MMCF	-	PYRATES Smart Fabrics SL	[168]

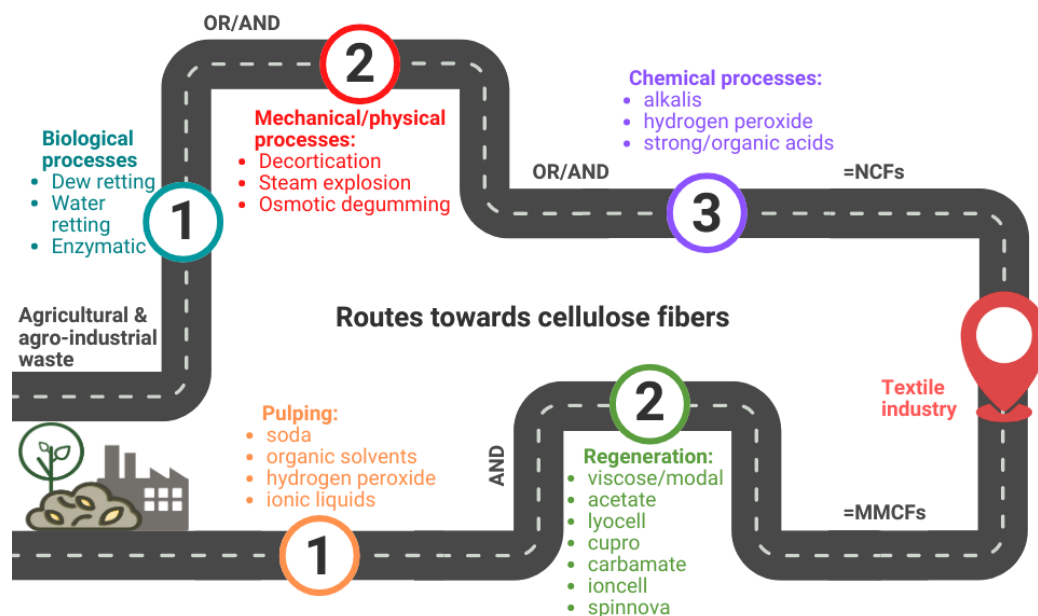
\* Limited edition initiative.

**Table 8.** Researched MMCFs derived from agricultural waste.

M	Process	References
Waste straw	Steam explosion/cellulose acetate	[129]
Coconut waste	Multistep chemical process/electrospinning	[112]
Sugarcane bagasse	Soda pulping/spinning with IL	[3]
Oil flax fibers	Soda pulping/Lyocell process	[113]

## 7. The Routes Towards Agricultural and Food Waste Cellulose Fibers

Figure 10 graphically summarizes the two major routes identified for the upcycling of agricultural residues and agro-industrial by-products intended for the textile industry. However, it is evident that there are several different sub-routes in the form of processes that researchers or manufacturers should study and choose from at each step of the way towards the valorization of cellulose fibers. Starting from agro-waste, both NCFs and MMCFs are exploitable as textiles but are commonly evaluated by researchers for different final applications. NCFs typically demonstrate very good strength properties but have a coarser appearance and lower brightness; thus, they are usually developed as reinforcement materials in composites or as technical textiles for industrial applications [65], such as thermal and acoustic insulation components. Moreover, it has been demonstrated that hybrid composites that combine NCFs with diverse matrix substances result in superior features, including strength [169], and that NCFs are able to form a uniform distribution within the matrix [170]. On the other hand, MMCFs can be produced as very fine filaments with specific properties in line with the regeneration method selected and can be easily repurposed for the fashion industry. Thus, NCFs have been used to extract cellulose in the form of pulp, as in the case of hemp, oil flax [113], and borassus fruit fibers [171]. This certainly happens at the expense of water, energy, and chemical consumption, since pulping and regeneration require intense treatments. Still, as Table 6 shows, a number of agro-waste NCFs have been branded for the fashion industry, demonstrating the appropriate properties. Table 6 also indicates the overall scarcity of agro-waste textiles, leading to the conclusion that the textile industry requires further research and piloting support, as well as standardization and traceability processes in order to include agricultural residues and food waste as raw materials in the supply chain [172]. This inclusion has been successfully performed in the case of the pulp and paper industry in India, where agricultural residues provide around 28% of the overall feedstock [173].



**Figure 10.** Routes to produce cellulose fibers from food- and agro-waste.

Nonetheless, dissolving-grade pulp for textile fiber production is highly purified compared to paper-grade pulp and has a high concentration of  $\alpha$ -cellulose (over 90%) and low levels of hemicellulose (less than 4%), lignin, and extractives [174]. This final composition makes the extraction process more advanced with different parameters to control to reach optimum values. Moreover, various regenerated cellulosic fibers have distinct raw material (pulp) specification requirements, which are reflected in their production processes and product characteristics. Typically, dissolving pulps for the viscose and Lyocell method

contain a percentage of  $\alpha$ -cellulose that is greater than or equal to 92% [99,174,175]. In addition, the mechanical strength of the regenerated fiber is directly influenced by the DP of the dissolved cellulose. The mechanical strength of the fiber is, in theory, enhanced by a higher DP. However, the spinning dope may exhibit increased viscosity and poor solubility that comes from an excessively high DP. The spinning process and the efficacy of the corresponding fiber are significantly influenced by the balance between DP and pulp solubility. The optimal DP values for the closed-loop Lyocell process are determined to be between 650 and 750 [99,142].

As a result of research and development, MMCfFs with low  $\alpha$ -cellulose content and DP have been regenerated. This could be the case when valorizing agricultural waste; making high-purity cellulose pulp may lead to lower DP because of overprocessing. Zhang et al. [175] demonstrated that a Lyocell spinning dope with a high hemicellulose content can exhibit good mechanical properties due to its ability to process at higher concentrations. The aqueous NMMO solution can also dissolve the hemicelluloses in the pulp during the spinning solution production, but an excessively high amount of hemicellulose can reduce the mechanical strength of Lyocell fibers [142]. Utilization of low-cost paper-grade pulp to produce Lyocell fiber was made possible by mixing it with dissolving-grade pulp, 25% NMMO, and 0.1% propyl gallate and imposing hydraulic or drum press at 200 bars as pretreatment method [176].

By choosing the optimal treatment method and controlling the process factors (temperature, pressure, concentration of the chemical agent, liquid-to-solid ratio, etc.) in line with the initial agricultural/agro-industrial waste composition, the extracted cellulose macromolecules may achieve a molecular weight suitable to be used as a raw material to produce fibers for the textile industry, while the percentage of  $\alpha$ -cellulose in the final product is within the desired range.

## 8. Conclusions

Cellulose is undoubtedly the most widely used biopolymer due to its excellent mechanical properties and biodegradability. Its utilization is expected to grow even more with the adoption of stronger regulations for recycling and waste handling. At the same time, agricultural residues and industrial food byproducts contain a large amount of insoluble fiber, which can provide high-value textile fibers. Their valorization demonstrates a sustainable approach to textile manufacturing—in need of raw materials—by repurposing this waste into high-quality, eco-friendly fabrics, embodying the principles of circular economy and environmental stewardship. This is possible via two distinct routes: the extraction and purification of NCFs and the extraction and purification of pulp, followed by a regeneration process to produce MMCfFs. Numerous research studies suggest different mechanical, chemical, biological, and blended approaches to navigating these pathways.

By selecting the optimal treatment route, NCFs or MMCfFs, and controlling the process factors, depending on the initial agro-industrial waste composition, we can achieve the production of fibers with a high percentage of cellulose in the final product and favorable properties for use as textiles. While mild treatments such as retting or the use of chemicals at lower concentrations and temperatures can produce NCFs, current state-of-the-art processes for producing MMCfFs are linked to the use of chemicals and highly controlled conditions. The advantage of MMCfFs is advanced mechanical properties, including textile strength and fineness. Additionally, the selection route can be based on the fiber content and quality of the raw material, as wastes containing small insoluble fibers, such as orange peel and mango seeds, can only be converted into MMCfFs.

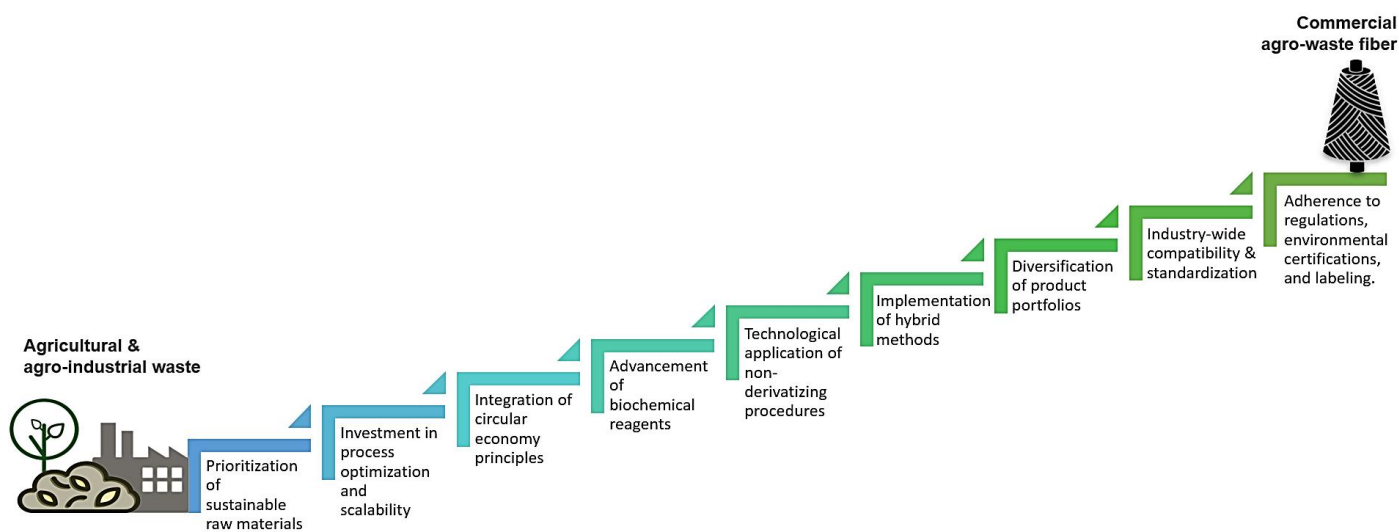
Despite the tremendous amount of research focused on their utilization in the development of different textile products, the commercialization of such products is quite scarce and still struggling its way towards the market. Most studies are either performed in research labs or on a pilot scale. But for waste textiles to enter the market, optimized processes must be developed that take into consideration all the engineering and economic constraints, leverage existing technologies, and establish quality control and traceability



protocols so that they may be exploitable by the textile supply chain. Therefore, it is critically important to conduct thorough research on the recovery of high-value products as well as environmental impact assessments, such as life cycle assessments and techno-economic analyses, in order to facilitate large-scale implementation.

#### *Recommendations and Steps Towards Industrialization*

A critical look at the processes described and the state of the art in the evaluation of agricultural and industrial cellulose wastes for the production of cellulose fibers, both NCFs and MMCFs, can lead to a number of proposals consistent with the circular economy framework, sustainability goals, and process enhancement. Figure 11 presents graphically these recommendations towards industrialization and successful marketing of the products.



**Figure 11.** Recommendations and steps towards industrialization and market presence.

- **Prioritization of sustainable raw materials**

This strategy corresponds with the worldwide initiative for more sustainable raw materials. It can be boosted by utilizing agricultural and agro-industrial byproducts, including bagasse, straw, stems, and peels, a practice that mitigates rivalry with food crops for cultivable land. Recommendation: An ecosystem between the textile industries and the agro-processing sectors should be created to provide an uninterrupted supply of lignocellulosic waste, thus a sustainable feedstock stream.

- **Investment in process optimization and scalability**

With the exception of caustic soda treatments, many novel techniques show potential but require more optimization for industrial viability. Recommendation: Solvent recovery, energy efficiency, and limited process duration should be placed under the microscope of industrial players who ought to push forward the optimization of promising, novel technologies and research results for large-scale use.

- **Integration of circular economy principles**

In addition to using waste as raw materials, the incorporation of processing subparts, such as closed-loop systems for solvent recycling, can diminish waste and lessen the ecological impact of fiber manufacturing. Recommendation: Industries ought to emphasize the implementation of closed-loop processes, including solvent recovery methods, and the use of recycled (either open-loop or closed-loop) and recyclable raw materials.

- **Advancement of biochemical reagents**

Biochemical processes can greatly diminish the use of chemicals that can impact negatively the environment and health. Nevertheless, enzymatic procedures continue to be

costly and difficult to scale. Recommendation: Industries need to investigate methods to diminish enzyme costs or enhance their reusability in industrial applications.

- Technological application of non-derivatizing procedures

Non-derivatizing methods in the production of MMCFs, such as Lyocell and Ioncell, present a more environmentally friendly alternative to derivatizing processes like viscose and acetate manufacturing. These technologies employ solvents such as NMMO (for Lyocell) or ILs (for Ioncell), which may be recycled and recovered with minimum losses. Recommendation: The industry should prioritize expanding non-derivatizing processes due to their enhanced environmental profile while also taking specific steps to reduce the final cost of the produced fibers. In particular, the high energy cost linked to the recovery of the solvent via evaporation should be tackled through research and development of alternative methods such as membrane technologies. In addition, research towards cost-effective methods for producing ILs should intensify.

- Implementation of hybrid methodologies

Methods such as steam explosion, enzymatic hydrolysis, and treatment with H<sub>2</sub>O<sub>2</sub> may be integrated or employed as pre-treatment techniques to diminish the necessity for aggressive chemicals in cellulose extraction. Besides the degradation of lignin and hemicellulose prior to further processing or the bleaching ability of some enzymatic treatments, these hybrid techniques can facilitate the further development and take-up of these prominent technologies by the industry. Recommendation: The development of hybrid methodologies that blend mechanical, enzymatic, and chemical treatments may be exploited by the industry to comply with environmental regulations while maintaining cost efficiency and leading to a stepwise integration of these technologies into the production line.

- Diversification of product portfolios

The market for cellulose-derived goods is advancing, including applications from textiles to bioplastics and composite materials. Cellulose fibers obtained from agro-waste can be utilized in specialized applications such as technical textiles, composite reinforcements, or biodegradable packaging materials. Recommendation: Companies could vary their product portfolios by exploring fresh applications for cellulose fibers and new marketing strategies. NCFs are optimal for technical textiles and biodegradable materials across many sectors, whereas MMCFs can serve as far as luxury fashion.

- Industry-wide compatibility and standardization

Establishing and applying consistent standards, guidelines, and criteria for the properties, performance, testing, and classification of textile fibers ensures that textile products meet specific quality, safety, and performance requirements and allows for uniformity in production, testing, and labeling across the industry. The marketing of agro-waste fibers can only be established by adhering to the standards, enabling thus different parts of the textile supply chain—from raw fiber producers to fabric manufacturers—to communicate and collaborate effectively. Recommendation: Novel agro-waste fibers need to conform with textile standards for quality control and classification.

- Adherence to regulations, environmental certifications, and labeling

Given the heightened examination of environmental performance, it is imperative for enterprises to adhere to standards concerning emissions, wastewater management, and sustainable sourcing. Recommendation: Industries must adhere to environmental standards and seek sustainability certifications. This practice not only improves brand reputation but also creates opportunities in new markets that emphasize eco-friendly products away from greenwashing practices. Agro-waste fibers must take advantage of the recycled content standard, while stakeholders may consider the application of explicit labeling that will positively differentiate these fibers from recycled polyester or nylon and improve consumer acceptance.

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