Alternative Methods to Retting of Straw

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Bast fibers, such as flax and hemp, have been used by humanity for thousands of years. In the case of processes other than dew or field retting, they can also follow field drying. In any case, the fiber plant straw is retted first in order to be able to carry out the subsequent mechanical fiber recovery by decortication, cleaning and, if necessary, opening of the coarse fiber bundles. This can be defined as primary processing of bast fiber straw. The retting process involves the controlled decomposition of those substances that bind the fiber containing tissues to the other components of the stalk as well as the fibers to each other (to form so called bundles). It is described as one of the most important steps in the whole supply chain of fibers from bast fiber plants such as hemp and flax since it affects both the ease of performing the subsequent mechanical processing steps as well as the quality of the resulting fibers.

Keywords: bast fiber ; hemp ; flax ; retting ; degumming ; procedures ; technological aspects

1. Introduction

Bast fibers, such as flax and hemp, have been used by humanity for thousands of years [1][2][3][4]. Evidence of the use of bast fibers especially hemp and flax/linen has been found in ancient society throughout human history including the cultivation and use of flax/linen in Ancient Egypt [5] or presence of linen in Swiss dwellings [6][7]. In the recent past several Asian and European communities cultivated and processed hemp for textiles [8]. Due to a combination of reasons, the production of bast fibers drastically reduced as more attention was given to other fiber sources. A significant reason was the mechanization of the cotton spinning system and later the invention of the cotton gin in 1793, which made cotton an economically very competitive fiber compared to hemp and linen. A far more extensive influence, however, is the development and introduction of synthetic, petroleum-based fiber materials beginning from the first decades of the 20th century. The (thereafter following) social ostracizing of hemp which was a major bast crop grown in Europe and the United States dealt a major blow to the development of the processing technology for hemp and bast fibers in general. As a result, the processing technology for hemp and other bast fibers has since lagged behind, hence higher production costs. For example according to Schmitz et al. (as quoted by [9]), the production cost of hemp fibers in Germany was five to ten times compared to cotton or synthetic fibers. However, recently there has been renewed interest in finding environmentally friendly alternatives to cotton and synthetic fibers for both clothing and technical applications. Bast fibers have a great potential to provide this alternative due to their high yield per unit area as compared to cotton, limited or no need for chemicals during growth and usually no need for irrigation [10]. It is therefore important to develop an efficient and commercially viable system of extracting the bast fibers from the plant stems to fully benefit from the opportunities provided by bast fibers.

Based on increasing research in this field, a large number of papers have been published in recent years (**Figure 1**) dealing with various aspects of post-harvest treatment of bast fiber straw (stalks), the subsequent fiber extraction as well as possibilities of further fiber modifications by means of biological, chemical, or other measures. A few selected ones are Akin et al., 2004 [11]; Antonov et al., 2007 [12]; Crônier et al., 2005 [13]; Di Candillo et al., 2010 [14]; Djemiel et al., 2017 [15]; Fernando et al., 2019 [16]; Foulk et al., 2008 [17]; George et al., 2015 [18]; Jankauskiene et al., 2006 [19]; Mazian et al., 2018 [20]; Lee et al., 2020 [21]; Liu et al., 2016 [22]; Lyu et al., 2021 [23]; Pakarinen et al., 2012 [24]; Réquilé et al., 2021 [25]; Ribeiro et al., 2015 [26]; Parikh et al., 2011 [27]; Tamburini et al., 2003 [28]; Thygesen et al., 2013 [29]; Valladares Juarez 2009 [30].

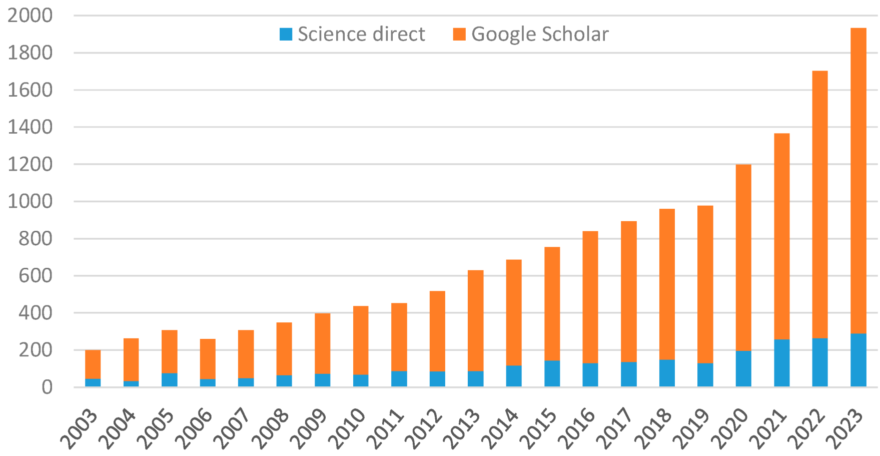

Figure 1. Number of published articles related to hemp and flax retting 2003–2023.

Nevertheless, this still shows a very diverse understanding of the metabolic processes in the post-harvest period, the classification of the individual steps in the entire process chain and their definition. In order to be able to correctly elucidate and evaluate the influence of different process factors, and in particular on the resulting properties of raw materials, intermediate and final products, a clear understanding of the entire process chain is essential.

These considerations are initially based on the choice of location with corresponding weather conditions, crop type and variety, as well as the corresponding arable and plant agronomic measures during the cultivation phase. This also results in essential framework conditions for the harvest and the subsequent post-harvest period on field. At this specific point, the retting process is defined, during which the fiber plant straw is subject to the natural weather conditions, together with the necessary drying.

In the case of processes other than dew or field retting, they can also follow field drying. In any case, the fiber plant straw is retted first in order to be able to carry out the subsequent mechanical fiber recovery by decortication, cleaning and, if necessary, opening of the coarse fiber bundles. This can be defined as primary processing of bast fiber straw. Afterwards, the resulting fibers can be further processed using biological, chemical, or even physical methods. Here, processes such as cottonization or degumming are to be considered. Otherwise valuable publications, both as reviews or as research findings do not make this fundamentally important distinction [23][31].

The retting process involves the controlled decomposition of those substances that bind the fiber containing tissues to the other components of the stalk as well as the fibers to each other (to form so called bundles). It is described as one of the most important steps in the whole supply chain of fibers from bast fiber plants such as hemp and flax since it affects both the ease of performing the subsequent mechanical processing steps as well as the quality of the resulting fibers. However, despite its long history, retting process of bast fiber straw is not yet fully developed to produce satisfactory fiber quality at a reasonable cost. Some reasons are, for example, the unpredictability of the course of field/dew retting due to random weather conditions. Furthermore, there is a lack of suitable and easy to apply methods for determining the optimal or final point of retting, which would contribute significantly to achieving uniformity and quality of the bast fibers. The other important question is the rettability of the bast stem. This may be understood by examining the significance of the different matrix substances to the process of retting. To what extent does the degradation of a given matrix substance (e.g., pectin, lignin and hemicellulose) contribute to the ease of separation of the fibers from other stem tissues (i.e., rettability). At the moment, a larger proportion of bast fibers are used in areas where certain variations in physical and mechanical quality parameters are tolerable. For example, for heat insulation and reinforcement in composites for less-demanding structures. For applications where high uniformity of quality parameters are paramount, for example in high performance load bearing composites materials, fibers for spinning and blending with cotton, bast fiber use is limited by the wide variance of its properties. These variations depend on a number of factors including; plant varieties or its inherent variability, weather during the growing and retting period and the method of retting among others. A better insight of the retting process will lead to the production of better quality fibers with more uniform distribution of mechanical and physical properties. More understanding of the retting process should also lead to an optimized setting of the operational parameters of the subsequent straw processing and thus, better predictability of the fiber properties based on given raw material properties.

Further processing (or secondary processing) of the bast fibers after straw retting and their separation from the woody parts of the stalks provides opportunity for further modification and improvement in fiber quality [32]. This can be a chemical or biological treatment or the so-called cottonization process which aims to produce cotton-like fibers from bast fibers. The methods used at the moment are however still very expensive and thus, in many scenarios economically less competitive to cotton and synthetic fibers.

2. Retting Methods and Attempts at Improvement

Very extensive research on the topic of retting—both field/dew or water-based—was carried out in the decades when cotton and synthetic fibers had not yet been introduced and bast fibers (along with wool) dominated the production and use of this class of materials. With the renaissance of traditional bast fibers in particular in Europe from the mid-80s in the last century, research also experienced a further upswing and corresponding publications appeared. Despite more recent research, the essential principles of such works are certainly still valid today, but due to the diverse origins of such works in the important cultivation countries of that era (e.g., France, Italy, Ireland, Russia or Germany), a detailed analysis is not possible here. However, due to frequent citations, e.g., Tanner 1922 [33]; Ruschmann 1923 [34]; Dujardin 1948 [35]; Turner 1954 [36]; or Sharma and van Summere 1992 [37] shall be named.

Traditionally the retting process is carried out either through water or dew retting [27].

3. Alternative Methods to Retting of Straw

Due to the shortcomings of the traditional retting methods such as high dependence on the weather conditions in dew retting, difficulty to determine the end point of retting and other factors which make the traditional methods less controllable, more efforts to produce better fibers with more uniform properties are needed. Several alternative methods and attempts at improving the traditional methods with the aim of developing processes with more controllable parameters to achieve uniformity in the physical properties of the fibers have been explored. These alternatives include inoculation with bacterial or fungal cultures, enzymatic treatment, chemical treatment, direct ensiling and mechanical decortication. Sometimes the different methods are used in combination to achieve better retting and better fiber qualities. For example chemical chelators and enzymes [38], physical and chemical processes in steam explosion technique [39], mechanical agitation and heating with caustic formulation [40]. Occasionally, it is also indicated that the efficacy of such or similar methods can be improved by mechanically stressing the stems to break its outer tissue layers (i.e., cuticle and epidermis) [16][41]. This can be achieved, for example, by turning the swath for uniform field drying and retting, which must be carried out in the postharvest period anyway.

However, a clear distinction should be made between those approaches that aim to improve the subsequent processability of the stalks (i.e., retting) and those that aim to minimize the non-cellulosic components of fiber bundles after their extraction from stem biomass (most frequently referred to as degumming). A very recent review [42] provides a very comprehensive insight into, among other things, the systematic activity of microorganisms and relevant enzymes during retting and degumming. However, without detailed knowledge of the primary sources, as well here it is difficult to distinguish between the reference level “stalk” on the one hand and “fiber bundle/fiber” on the other and thus it is hard to make a targeted classification in the process chain.

3.1. Inoculation

The idea of isolating microbes with known enzymatic activity towards the degradation of major materials that form part of the matrix substance in the stems of bast plants (such as pectin) is not new. Thus, it has been found in literature as early as 1984 (Hunter and Brown, as cited by Sharma et al. [43]) and has also been suggested by more recent scholars [26]. Early attempts involved the use of bacterial isolates *Bacillius subtilis*, which produces mainly pectin-lyase and xylanase, and *Erwinia carotovora*, which produces only pectin-lyase, on dessicated flax, where the retting of desiccated flax straw was found to be enhanced by the spraying cell suspension of *B. subtilis* [44]. More potential benefits of the use of bacterial and fungal cultures are apparent where up to nine bacterial species (including *Rhizomucor pusillus* and *Fusarium lateritium*, *Trichoderma virens*, *Alternaria alternata*, *Fusarium oxysporum*, and *Fusarium equiseti*) were successfully isolated and used for retting of flax straw, but resulting in fibers with varying qualities [45]. A potential advantage is that bacteria or fungi that are efficient in retting may be introduced to the straws even if they do not form the native colony of the soil. For example, the fungi *R. pusillus* isolated from South Carolina flax which had not been isolated anywhere before showed a high retting efficiency in the tests, and could be used to enhance retting in environments where it does not naturally occur. Thygesen et al. [29] used a laboratory-based setup to demonstrate the effects of inoculation of hemp stalk sections with the mutated white rot fungus *Phlebia radiata Cel 26* on a selective degradation of the epidermis as well as the lignified middle lamella. When this white rot fungus was applied direct to bast fibers, it showed higher selectivity in depectinisation compared to *Ceriporiopsis subvermispora* and compared to a classical water retting. A later study by [46] showed that stronger fibers with more homogeneous properties can be produced for composite applications by using the dependence of the depectinisation selectivity on the stem section. Inoculation of manual peeled fibers (which indeed is probably better to refer to as bast or bark) with selected fungi resulted in less reduction in the strength of fibers from the lower section than those from the upper stem section. When considering the whole hemp stalk independent of the origin of the fibrous tissue, fungal retting with *P. radiata Cel 26* showed better mechanical properties in contrast to *C. subvermispora*.

Inoculation with bacterial/fungal suspensions may also allow the influencing of the time of the start of colonization of the stem tissue. For example, through inoculation *A. alternate* may also be used as a primary colonizer although it has been reported to be active in latter retting process [45]. This method may also be useful for targeting fiber properties by using certain types of fungi (white rot fungi), here on green hemp fiber material sealed in bags [47]. This has produced desirable properties of cross-linked fibers known for a particular application or industry (for example, improving strength and interfacial bonding in composites).

The use of inoculum from microbial isolates has not only been used in dew retting but also in water retting. The addition of anaerobic *Clostridium* sp. and aerobic *Bacillus* sp. in retting liquor accelerated the retting process of hemp and significantly increased the fiber quality [14]. This further opens possibilities of developing a more controllable retting process for stem material by varying the dose and time of adding the inoculum. While the aerobic bacteria does not show significant activity in degrading pectin, the use of aerobic inoculum in addition to the anaerobic inoculum increases the speed of retting. This speeds up the retting process since the addition of aerobic bacteria inoculum most likely rapidly generates the anoxic environment needed by anaerobic bacteria to grow [48].

3.2. Enzymatic Treatment

Another attempt at developing an alternative retting process is the direct application of enzymes to ret fiber crop straw. In distinction to this, however, enzymatic processes are also used to degum subsequently mechanically extracted bast fiber structures. Enzyme retting appears to have a better commercial prospect as compared to chemical treatment in terms of costs [49][50]. In addition, effluents from enzyme treatments tend to be more environmentally friendly [51] by, for example, reuse [52] which increases economic success in the increasingly environmentally conscious society since the products may be marketed as ecofriendly. The enzymes may be obtained from pectinase producing fungi or bacteria by methods such as solid state fermentation and submerged fermentation [46][53][54] or from commercial producers. The degradation of pectin is a major factor that correlates to the degree of retting [55], therefore pectinase can be considered the most important enzyme for retting. Due to the specific action of enzymes, a consortium/mixture of enzymes may be necessary to target the different substances that make up the stem matrix for a more efficient retting [56]. For example, the commercial retting enzyme flaxzyme, which contains pectinases, hemicellulases and cellulases, was used for the retting of flax leading to a considerable reduction of the retting time from several days to within 24 h [57][58][59]. Further commercial Enzymes such as Bioprep 300L, Flaxzyme, Lyvelin, Texazyme BFE or Viscozyme have been used in different formulations to achieve the required fiber characteristics at different levels [12][38][42][60]. However, enzymes that contain cellulases tend to lead to reduced fiber strength [17].

Protective structures of fiber crop stems such as the epidermis may prevent the pectinase enzyme from reaching the pectin compounds and would therefore need to be degraded by other means (usually mechanical) to give access to the inner stem structures. The use of surfactants that reduce surface tension of the water, therefore facilitating better access to the inner stem tissues, have proved useful in improving retting [41][61]. The enzyme formulations are commonly used together with a chelator (mostly ethylenediaminetetraacetic acid EDTA) [62]. According to Akin et al. [11] the regions between the fibers, the cambium and the middle lamellae are susceptible to degradation by enzymes but the epidermis was only effectively degraded with addition of chelator to the enzyme formulation. In addition, the mature plant tissue, the middle lamella is often converted to calcium pectate thereby cementing the cells firmly [63] and hence the need for chelators. Chelators remove Ca++ and destabilize pectin molecules and therefore contribute to the removal of matrix compounds holding fibers within the stem and have been shown to improve retting as well as subsequent mechanical fiber extraction when used with enzyme formulations [38]. The addition of chelators help to reduce the amount of enzymes used in retting by up to 50-fold [11].

The ability to make different formulations with different enzymes and concentrations [62] makes enzyme retting process a good and versatile alternative method for achieving particular fiber characteristics as far as process control is concerned. To prevent the deterioration of the of fiber strength due to continued enzymatic activity, thorough rinsing is required or denaturing treatment should be carried out at the end of the process [11]

Enzymatic treatments are mentioned to be also very suitable for the modification of fiber properties after fiber separation in a process and are often referred to as cottonization [64], as well. However, this term should rather be used when (pre-)prepared fiber elements such as bundles are refined or shortened by mechanical processes.

3.3. Retting Enhancement by Additional Chemical Treatment

The exclusive use of chemical substances for retting bast fiber stalks has been scientifically investigated, but is essentially not practiced. Such processes are mainly used for the degumming of fibers following mechanical fiber extraction from the straw. Detailed information on this can be found in a subsequent chapter of the manuscript. Evidence for the use of chemicals in the retting of plant stems can only be found in the literature almost exclusively in connection with other processes, such as stand retting with the aid of herbicides. Sharma [63] refers to older literature [35] with research results on the effects of, for example, acids or bases. However, these led to poor yields and low fiber qualities. The additional statement about high costs nevertheless remains valid and must be supplemented by the fact that the use of chemicals in the production of sustainable materials is not appropriate and (at least) potentially harmful to the environment [65].

Where chemical retting of straw is mentioned in literature, it refers mainly to the use of chelators commonly in combination with other procedures as well. This technique appears to be more popular for flax and hemp and it uses compounds such as ethylenediaminetetraacetic acid (EDTA), cyclohexanediamintetetraacetic (CDTA), sodium tripolyphosphate ($Na_5P_3O_{10}$), oxalic acid ($C_2H_2O_4$) [61][66]. The most common chelators used show high binding capabilities with calcium ions at high pH (above 4). Chemical retting with EDTA and oxalic acid involves the formation of complex compounds referred to as chelates. The acids act as chelators to bind with the calcium in the calcium pectate to form complex compounds making it easier to separate them separate them from the fibers [63]. Since chelators have optimal metal binding capacities at high pH, NaOH is added to the formulation to neutralize the acids produced during incubation therefore keeping the pH of the liquor high [66]. By chelating metal ions from pectin complex, non-cellulosic plant materials (pectin and hemicellulose) can be extracted using alkaline solutions of chelating agents [63]. The use of chelators at high pH appear to be the most successful/optimal way of chemical retting that is less aggressive (compared to mineral acids or boiling in alkali) judging from the fine fibers that are produced [66] and since the process does not require additional energy in terms of heating and mechanical agitation. Other chelating agents include trisodium phosphate, sodium gluconate [66], diethylenetetraacetic acid, tetrasodium salt (Trilon TB), diethylenediaminepentaacetic acid, and Penta sodium salt (DTPA) [63].

Detergents have also been tested for retting and usually in combination with acids or chelating agents. The use of such surfactants is shown to improve the retting of flax probably due to their ability to solubilize proteins and the reduction of the surface tension of water to allow easier access to the matrix material by the chelator, therefore enhancing its action [61]. Other methods using chemicals have been developed and used in the context of plant breeding in order to enable effective and complete fiber extraction [67][68].

3.4. Physical Treatment

Methods that involve the physical disruption of the integrity of the plant structure through various means have also been applied to loosen the fibers from the woody core.

Steam explosion treatment is a method for breaking down biomass that is used in the processing of fibrous source materials as well. However, if one follows the relevant literature on this topic, a sometimes-incorrect interpretation of the underlying research results of the referenced primary sources becomes apparent. For example, experiments mentioned in all three sources in (Manian et al.) [31] do not deal with the processing of stems, but of fiber bast or fiber bundles [69][70][71].

Considering relevant literature in the area of other lignocellulose biomasses, it would have to be assumed that the entire stem biomass is dissolved and that it may not be possible to distinguish between the desired yield of fiber elements from the bast and those from other tissue parts of the stem (essentially the xylem or woody inner core).

Literature references on the use of steam explosion treatment of bast fiber stalks could not be found. However, in one research project, unpressurized steam treatment in a flow reactor was used for retting in order to investigate the release of certain elements or compounds from different hemp samples [72]. The treatment of hemp stalks resulted in an easier detachment of the bast layer from the xylem, but was not investigated in more detail.

Another physical effect, caused by **radio frequency**, has been investigated over a time period by a group of researchers to improve the retting of bast fiber stalks. The intention was to enhance the efficiency of water retting [73] or to assist enzymatic retting [74] of flax stem samples. Pre-soaking, adapted power levels and application duration in combination with the control of the water temperature can lead to an optimization of the process control and the resulting material properties.

Microwaves, as well part of the electromagnetic spectrum, have also been investigated to support retting processes. A good overview of the relationship between biological principles and the possibilities and effects of electromagnetic energy on the processing of flax can be found in Nair et al. [75]. In-depth investigations have shown the positive influence of already known process parameters such as pre-soaking, microwave power, temperature and process time on the structural decomposition of non-cellulosic, fiber cementing polysaccharides [76]. The positive results of the assisted treatment of flax stalks could also be transferred to hemp stalks on an experimental basis [77].

A comparison between the use of radio frequency and microwave assistance of enzyme retting has shown that the latter has a more significant effect under comparable process conditions with slightly lower mechanical properties of the resulting fibers [78].

Ultrasound as another physical effect has also been investigated and applied to assist the separation of fiber and non-fiber components. In contrast to the aforementioned methods, this is a mechanical wave and not electromagnetic radiation. Konczewicz et al. [79] have investigated osmotic effects in aqueous environment, where water is continuously passed through the biomass of the straw. The fibers, the woody core and the pectin absorb the water and swell thereby exerting considerable physical pressure on the cuticle, which causes it to break. The exposed pectin as well as other soluble substances are diluted and carried away in the circulating water. Thus, the separation of the fibers from each other is enhanced and resulting in reasonable fiber quality. The whole process was combined with ultrasound treatment to make it more effective.

3.5. Ensiling

In the search for alternative raw materials for the pulp industry, supply procedures based on wet preservation ("silage") of flax [80] and hemp [81] biomass were investigated for the first time in the mid-90s of the last century. From there, further scientific work dealt with the anaerobic storage of freshly harvested hemp and its use in fiber-reinforced plastics [82][83]. In the same period, a series of in-depth studies began, in particular on harvesting and storage variants, the use of additional preservatives and the resulting fiber properties after thermo-mechanical processing of the whole plant material [84][85][86].

In contrast to dew retting, ensiling offers the significant advantage of being completely independent of unpredictable weather conditions. This also means that the fields are cleared immediately after harvesting, so that the value of hemp as preceding crop can be utilized in the subsequent crop. The ensiling process is a well-established agricultural practice and the effects of the anaerobic storage processes on the properties of the bast part of the plant material are comparable to the results of common retting procedures. Based on a patent specification of Clarke et al. [87], a publication reports on a so-called bag retting process. It is shown as well that the enzymatic activity of mainly anaerobic bacteria removes non-cellulosic components of the hemp fibers in silage stock [47].

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