Retting Process as a Pretreatment of Natural Fibers for the Development of Polymer Composites

L. Sisti, G. Totaro, M. Vannini and A. Celli

Abstract The development of high-performance materials made from natural resources is increasing worldwide. Within this framework, natural fiber reinforced polymeric composites now experience great expansion and applications in many fields, ranging from the automotive to the construction sector. The great challenge in producing composites containing natural fibers and with controlled features is connected to the great variation in properties and characteristics of fibers. The quality of the natural fibers is largely determined by the efficiency of the treatment process and can dramatically influence the properties of the final composites. The overall fiber extraction processes, applied to vegetable fibers, is called retting and consists in the separation of fiber bundles from the cuticularized epidermis and the woody core cells. Today, many efforts are being made to optimize the retting methods in terms of fiber quality production, reduction of environmental issues and production costs. This chapter aims to provide a classification and an overview of the retting procedures that have been developed during years and are applied to extract mainly bast fibers.

Keywords Plant fibers · Retting · Polymer composites

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L. Sisti (✉) · G. Totaro · M. Vannini · A. Celli
Dipartimento di Ingegneria Civile, Chimica, Ambientale e dei Materiali,
Università di Bologna, Via Terracini 28, 40131 Bologna, Italy
e-mail: laura.sisti@unibo.it

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1 Introduction

Increasing environmental concerns and depletion of petroleum resources are driving energy and chemical industries to move from petrochemical to renewable resources. As a major renewable resource, lignocellulosic fibers derived from structural plant tissues will play an important role in this transition. Fiber crops are the commodities with the longest tradition among technical and nonfood agricultural products. Actually, bio-fibers have gained popularity especially for the development of sustainable materials, thanks to their great potential for use in composite materials for applications in packaging, automotive, and other industries (Dicker et al. 2014; Frollini et al. 2013, 2015; Miller 2013), especially as reinforcing fibers.

Up to now man-made fibers, such as glass, aramid, and carbon fibers were the predominant reinforcing fibers for polymers, but they feature environmental problems both during the production and disposal processes. Natural fibers, instead, have many remarkable advantages over the synthetic ones, such as lightweight, low cost, and biodegradability. However, at present, the offer of natural fibers does not fulfill industrial demand. In fact, the intensive cultivation of some fiber crops, such as hemp, flax, kenaf, and jute has increased, while stalk processing, for the extraction of fibers, relies on the traditional methods. Natural fibers from fiber crops, as a commodity, are facing competition on two sides: synthetic fibers on the consumer's side, and more remunerative crops on the grower's side. In order to face such dual challenges, the production of fiber crops must adopt a strategy based on the agricultural and industrial research and development and market promotion of both traditional and diversified fiber crops products (van Dam and Bos 2004). The improvements in fiber processing techniques play a significant role in obtaining fibers with enhanced standardized quality, capable to match the high performances required in composite materials but also in what concerns control and a lower environmental impact in terms of energy and water waste and consumption. The present essay will provide an overview of the traditional and new emerging fiber extraction processing techniques, which are known as retting or degumming and which involve the extraction of fibers or fiber bundles from harvested stems.

Moreover, the effect of these treatments on the performance of the resulting natural fibers in the composite application is addressed.

2 Plant Fibers as Renewable Resources for Polymer Composites

The species producing fibers of commercial relevance are mainly jute, flax, ramie, hemp, and kenaf, because they are much stronger than other fibers; they are also referred to, usually, as bast fibers. Plant fibers are classified by the part of the plant from which they are obtained, such as leaf, seed, fruit, stem, and bast. As its name implies, bast fibers are obtained from the outer layer of bast surrounding the plant stem. Since the bast fiber task is to act as reinforcement within the plant and to provide stability, they supply a good reinforcement in composite materials. Leaf fibers are reported to impart improved toughness in composites and the insertion of seed or fruit fibers gives elastomeric type properties to the component under consideration. Figure 1 lists the classification of plant fibers and Table 1 reports the main bast fibers along with their cultivation area and production capacities.

Bast fibers develop as bundles of cells embedded within other tissues in the stem. The process of separation and extraction of fibers from the nonfibrous tissues and woody part of the stem through separation, dissolution, and decomposition of pectins, gums, and other mucilaginous substances is called retting (Pallesen 1996; Dasgupta et al. 1976). The quality of the fiber is largely determined by the efficiency of the retting process, which relies on the breakdown of pectic materials through which fibers are liberated. Knowing the physical and chemical structure of plants is really crucial in designing fiber processing since these factors affect the

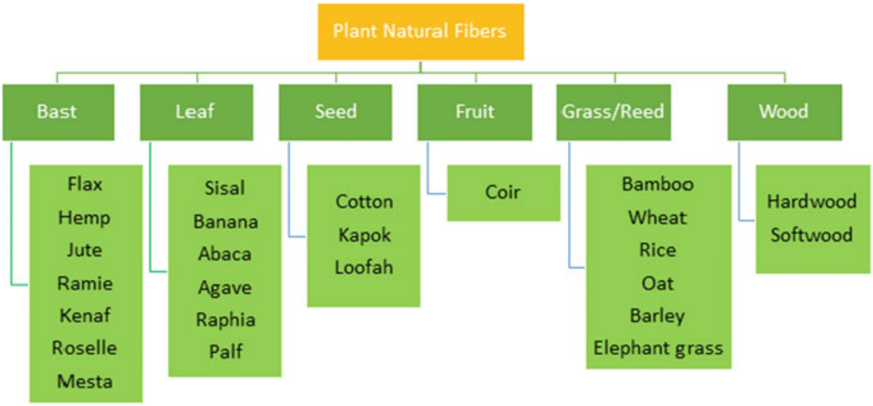


Fig. 1 Classification of plant natural fibers according to which part of the plant they are obtained from

Table 1 Cultivation area and production capacity of commercial important bast fibers and of the principal leaf, seed, fruit, and grass fibers (Yan et al. 2014; Faruk et al. 2012)

Fiber		Botanical name	Main producer countries	Production capacities per year (10^6 t)
Bast	Jute	*Corchorus capsularis, Corchorus olitorius*	India Bangladesh	2.5
	Kenaf	*Hibiscus cannabinus*	China India Thailand	0.45
	Flax[a]	*Linus usatissimum*	China Europe	0.5–1.5
	Ramie	*Boehmeria nivea*	China	0.15
	Hemp	*Cannabis sativa* L.	China Europe	0.10
Leaf	Sisal	*Agave sisalana*	Brazil China Tanzania	0.30
Seed	Cotton	*Gossypium* sp.	China USA India Pakistan	25
Fruit	Coconut	*Cocus nucifera*	India Sri Lanka	0.45
Grass	Bamboo	*Bambusa vulguaris*	China	30

[a]The real production of flax is understimated because it does not take into consideration the production of Canada

quality of the final fibers. In general, bast fibers consist of cellulose, hemicellulose, and lignin in various proportions. Other compounds, such as pectin, wax, minerals, and water-soluble compounds, are present in addition to the main components. Pectin serves as a glue to hold fibers together in bundles and bundles to nonfibrous tissues. Hemicelluloses, pectin, and lignin act like a matrix, whereas cellulose acts as reinforcement to the matrix contributing to the strength of the fiber (Thomas et al. 2011). In Fig. 2 major bast fibers and the most common leaf, seed, fruit, and grass fibers are shown while their chemical composition is reported in Table 2.

The big disadvantage of natural fibers, as compared to synthetic ones, is that they do not have the same consistency in composition and therefore in quality. This inconsistency is due to a variety of reasons, such as climate, crop variety, retting process, and processing equipment used for fibers (Thomsen et al. 2006). In natural fibers, climatic conditions play an important role in fiber production. For example, low temperature and high relative humidity during the growing season contribute to fineness and length of fibers. The role of retting in obtaining high-quality standardized fibers is crucial and research and development are heading toward the industrialization of treatment processes.

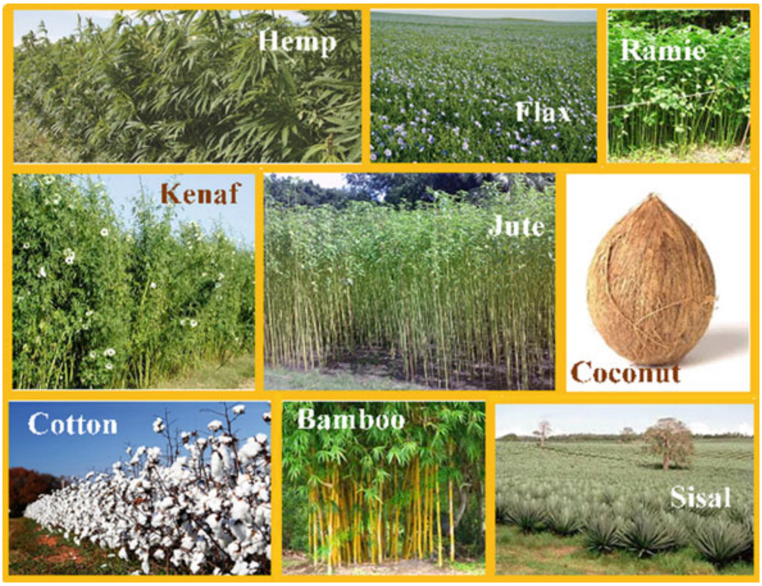


Fig. 2 Common bast fibers

Table 2 Chemical composition of major bast fibers and of the most common leaf, seed, fruit, and grass fibers (Gurunathan et al. 2015; Faruk et al. 2012; Wang et al. 2010)

	Fiber	Cellulose (wt%)	Hemicellulose (wt%)	Lignin (wt%)	Pectin (wt%)	Water soluble (wt %)	Wax (wt%)
Bast	Hemp	70.2–74.4	17.9–22.4	3.7–5.7	0.9	2.1	0.8
	Jute	61–71.5	13.6–20.4	12–13	0.2	1.2	0.5
	Flax	71–78	18.6–20.6	2.2	2.3	3.9–10.5	1.7
	Kenaf	45–57	21.5–23	8–13	3–5	–	–
	Ramie	68.6–76.2	13.1–16.7	0.6–0.7	1.9	6.1	0.3
Leaf	Sisal	67–78	10–14	8–11	10	1.3	2
Seed	Cotton	85–90	5.70	0.7–1.6	0–1	1.0	0.6
Fruit	Coconut	36–43	0.15–0.25	41–45	3–4	5.2–16.0	–
Grass	Bamboo	26–73.8	12.5–30	10.2–31	0.4	3.2	–

3 The Retting Techniques

The retting process, also known as degumming, involves the extraction of fiber bundles from the harvested stem. To date, several retting methods are applied; the most traditional, still widely used approaches, i.e., water retting and dew retting (Tamburini et al. 2004) are based on the microbiological retting. Other approaches

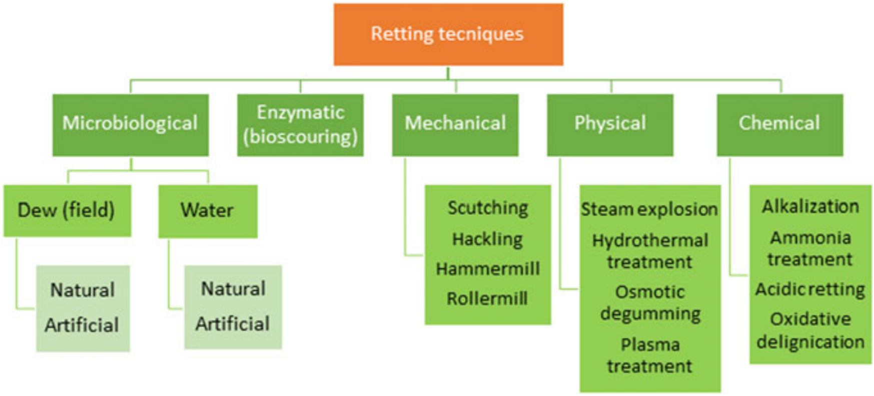


Fig. 3 Retting techniques

involve mechanical, physical, chemical, and enzymatic retting. The latter is very promising but not yet practiced on an industrial scale. Figure 3 shows a rationalization of all the retting treatments.

3.1 Microbiological Retting

Microbiological retting is a traditional and highly widespread retting method. Two different types of microbiological retting are mainly adopted: dew and water retting. Both of them are carried out by pectin enzymes secreted by indigenous microflora.

3.1.1 Dew Retting

In dew retting, also called field retting, harvested plants are thinly spread out for 2–10 weeks in fields (Fig. 4). During this period, microorganisms, mainly filamentous fungi or aerobic bacteria present in soil and on plants, attack noncellulosic cell types, removing pectins, and hemicelluloses from parenchyma cells and the middle lamellae, without attacking cellulose fibers. In this process, the colonizing fungi possess a high level of pectinase activity and the capacity to penetrate the cuticular surface of the stem: thus, fiber bundles come out separated into smaller bundles and individual fibers. Several fungal species and bacteria have been isolated from dew retted plants: *Cladosporium* sp., *Penicillium* sp., *Aspergillus* and *Rhodotorula* sp. (Ribeiro et al. 2015; Fogarty et al. 1972; Ahmed and Akhter 2001). During flax dew retting, other fungi were isolated, such as *Cladosporium herbarum*, *Epicoccum nigrum*, *Alternaria alternate*, *Fusarium* sp., *Aureobasidium pullulans*, *Phoma* sp., *Mucor* sp., *Rhizomucor pusillus*, and *Rhizopus oryzae* (Akin et al. 1998; Henriksson et al. 1997; Sharma 1986; Xiao et al. 2008;

Fig. 4 Dew retting (Flax composites 2014)

Molina et al. 2001; Booth et al. 2004). For more lignified fibers different enzymes are needed and often a mechanical step is added to separate the fibers (Ribeiro et al. 2015).

Currently, dew retting is the most used process for the industrial production of bast fibers, mainly flax and jute, because of its low cost (Bacci et al. 2010). Unfortunately, the method is limited to geographic regions, where the weather is suitable for fungi proliferation. Moreover, often low and inconsistent fiber quality is produced as compared to other methods, such as water retting. Risks of under retting and over retting are also reported: they may cause difficulties in separation or weaken the fiber (Jankauskiene et al. 2015). For example, cellulotyc enzymes secreted by the microbiota can damage the fibers if exposition lasts too long. Therefore, it is necessary to monitor the retting process to ensure the quality of the fibers. Occupation of lands for several weeks during retting and the presence of a product contaminated with soil and fungi are other drawbacks affecting this treatment.

As far as artificial dew retting is concerned, during the last few years, the employment of fungi in a more controlled environment for the extraction of natural fibers is being investigated. The controlled parameters involve the type of fungi, temperature, and period of treatment, in order to provide retting treatments with lower costs, higher efficacy, and environmentally friendly alternatives (Pickering et al. 2007a).

Among fungi, the white rot fungus (basidiomycetes) has been shown to be the only one able to degrade noncellulosic compounds from natural fibers, thus improving the mechanical properties of the resulting natural fiber reinforced composites (Pickering et al. 2007a).

Another recent study reports an enzymating enrichment of the dew retting process through the use of customized enzymatic blends (Texazym® SER). Such enzymes, developed by INOTEX, are sprayed on the field before pulling out or within the first 3 days of dew retting. It was found out that this method can increase flax long fiber yields by more than 40%. In this case, the enzymes in combination with mild mechanical treatments can replace aggressive and energy-intensive processing like Laroche cottonization (Antonov et al. 2007).

3.1.2 Water Retting

In water retting, quite widespread 50 years ago, straws are soaked in freshwater, today in large tanks, while in the past rivers or ponds were used (Fig. 5). During this treatment, that is carried out for a period of 7–14 days on most bast fiber crop straws, water penetrates into the central stalk portion, by breaking the outermost layer, and thus provoking an increased absorption of moisture and the development of a pectinolytical bacterial community (Donaphy et al. 1990). The duration of the treatment depends on the water type and temperature and on any bacterial inoculum (Bismark et al. 2005). The first stage of the process consists in the growing of aerobic microorganisms which consume most of the dissolved oxygen, ultimately creating an environment favorable for the growth of anaerobs. In the aerobic phase microorganisms belonging to the genus *Bacillus*, such as *B. subtilis*, *B. polymyxa*, *B. mesentericus,* and *B. macerans* have been found to be active (Munshii and Cathoo 2008; Ali 1958; Kapoor et al. 2001; Tamburini et al. 2003) while in the anaerobic phase different microbiota, of the genus *Clostridium*, such as *C. acetobutylicum*, *C. felsineum*, *C. aurantibutyricum,* and *C. tertium* have been isolated from retting water (Munshii and Cathoo 2008; Tamburini et al. 2003; Zheng et al. 2001; Di Candilo et al. 2000; Donaghy et al. 1990).

Water retting generally produces fibers with a higher quality than those produced by dew retting (Amaducci and Gusovious 2010; van Sumere 1992), but the water

Fig. 5 Water retting of bast fibers (**a**) in river (Frontier Culture Museum of Virginia 2012), (**b**) in tank (Retting the flax 2014)

retting process impacts the environment due to the consumption and contamination of large amounts of water (van Dam and Bos 2004) and energy (Van der Werf and Turunen 2008). With freshwater resources becoming increasingly scarce, an alternative or improvement in water retting will have to be foremost in dealing with water scarcity and pollution reduction. Enzyme retting, for example, which will be discussed in the next paragraph, has been evaluated as a replacement for current retting methods. Moreover, Zhang et al. (2008a, b) have explored the possibility of substituting freshwater with seawater, which is a natural resource considered to be inexhaustible and abundant. They demonstrated that seawater retting treatments yield good retting results and good pectinolytic strains belonging to *S. maltophilia* species and *O. antrophi* species. Artificial water retting, employing warm water and bacterial inoculum, has also been used to produce homogeneous and clean high-quality fibers in 3–5 days (Bismark et al. 2005; Sisti et al. 2016).

3.2 Enzymatic Retting

A modification of water retting is the enzymatic treatment, also called bioscouring, where degrading enzymes are directly added to tank water or in a bioreactor (Ouajai and Shanks 2005). This technique has been demonstrated to be a promising replacement for traditional retting methods in terms of time-saving, ecology friendliness, and convenient characteristics. The duration of enzymatic retting ranges from 8 to 24 h. The high energy input and nonreusability of enzymes are the main concerns, which affect the cost-effectiveness of the process (Tahir et al. 2011).

Pectinases are the main enzymes employed for retting, in order to free the fibers from other tissues. Today, with the advancement of biotechnological tools, pectinases or pectinolytic enzymes are of significant importance thanks to their all embracing applications, such as in fruit juice extraction and its clarification, scouring of cotton, degumming of plant fibers, wastewater treatment, vegetable oil extraction, tea and coffee fermentation, bleaching of paper, in poultry feed additives, and in the alcoholic beverages and food industries (Jayani et al. 2005). Pectinolytic enzymes are a heterogeneous group of related enzymes that hydrolize the pectic substances, mostly present in plants. They are widely distributed in higher plants and microorganisms (Mohnen 2008), since they help in cell wall extention and in softening some plant tissues during maturation and storage. They also help maintain ecological balance by causing decomposition and recycling of waste plant materials (Ridley et al. 2001). Plant pathogenity and spoilage of fruits and vegetables by rotting are some other major manifestations of pectinolytic enzymes. Pectinolytic enzymes, which are classified according to the main mechanism catalyzing the respective reactions, gather pectin esterase, polygalacturonase, and pectin lyase. Evans et al. and Zhang et al. reported that polygalacturonase activity plays a fundamental role during the retting process (Evans et al. 2002; Zhang et al. 2000), but also pectate lyase has been shown to be potentially important for retting bast plants (Akin et al. 2007; Bruhlmann et al. 2000).

Polygalacturonase and pectate lyase are both depolymerizing enzymes for pectin. Polygalacturonases are the pectinolytic enzymes that catalyze the hydrolytic cleavage of the polygalacturonic acid chain (Jayani et al. 2005), while pectate lyase carries out a non-hydrolitic breakdown of pectates and pectinases by a transelimination split of the pectic polymer (Sakai et al. 1993).

Pectinases can be produced from bacteria (Dosanjh and Hoondal 1996; Kapoor et al. 2001), yeast (Blanco et al. 1999), fungi (Singh et al. 1999), and actinomycetes (Beg et al. 2000). Most commercial preparations of pectinases are produced from fungal sources (Henriksson et al. 1997). *Aspergillus niger* is the most commonly used fungal species in the industrial production of pectinolytic enzymes (Jayani et al. 2005). Among bacteria sources, certain genera namely *Bacillus*, *Lactobacillus*, *Pediococcus*, and *Leuconostoc* are known to be effective during the fermentation process to produce pectinolytic enzymes (Kouhoundè et al. 2014).

Enzyme retting via the pectinases is capable of producing consistent high strength renewable fibers with variable fineness values for use in novel resins (Foulk et al. 2011). Some of the major commercialized enzymes are shown below.

Viscozyme® L, produced by Novozymes, is a multienzymatic solution containing a wide range of carbohydrases, including arabase, cellulose, β-glucanase, hemicellulose, and xylase. It has been tested by different authors in flax fiber extraction (Bacci et al. 2010; Foulk et al. 2008; Akin et al. 2003; Adamsen et al. 2002; Evans et al. 2002).

Pectinex® Ultra SP-L, produced by Novozymes, is a highly active pectolytic enzyme obtained by a selected strain of *Aspergillus aculeatus* that contains pectolytic and a range of hemicellulolytic activities that can disintegrate plant cell walls (Bacci et al. 2010).

Scourzyme® L has been developed by Novozymes for the bioscouring technique; it contains an alkaline pectate lyase, which degrades the pectin from the primary cell wall of fibers without degrading the fiber itself (Oujai and Shanks 2005).

Flaxzyme®, a commercial enzyme mixture from Novo Nordisk, consists of pectinases, hemicellulases, and cellulases and was developed specifically for enzyme retting (Akin et al. 1999).

BioPrep® 3000 L was developed by Novozymes as the first commercially available alkaline pectate lyase. It was isolated and produced for its unique ability to degrade the pectin layer between the waxy cuticle and cellulosic fabric cotton (Durden et al. 2001; Etters et al. 2001).

Texazyme® BFE was developed by Inotex for elementarization of bast fibers through the degradation of pectin layers. It consists of a multicomponent product without cellulase activity (Foulk et al. 2008).

For each enzyme, specific conditions are identified for employment in retting, since the activity can change dramatically with pH, temperature, and enzyme concentration. Moreover, chelators and surfactants are usually employed in formulations to improve activity. For example, the role of Ca(II) chelators, such as ethylenediaminetetracetic acid (EDTA), is well known (Akin et al. 2004) to improve retting, particularly, in removing the epidermal/cuticle material from the fiber and fiber bundles. In fact, EDTA acts by destabilizing bridges between Ca(II) and polygalacturonic acid, thus leading to the disruption of tissues.

Foulk et al. (2008) reported that, with a specific knowledge of the composition of the enzyme mixture, enzyme retting could be used to tailor fibers/fiber bundles with particular properties, such as strength and fineness, and for specific applications. Strength, for example, which is a major concern in many applications, is preserved by retting with relatively pure pectinase, either pectate lyase, or polygalacturonase. However, a mixed enzyme preparation containing cellulase could be used for advantageous applications where the fibers will be shortened, such as for paper/pulp or injection molding. In fact, it has been found that enzyme formulations like Viscozyme, containing cellulase as a component, can weaken the bast fibers, since the nodes of the fibers are particularly sensitive to the attack by this enzyme (Foulk et al. 2008). The final application, therefore determines the retting formulation.

3.3 Mechanical Retting

The mechanical extraction of fibers consists of various steps, as developed since ancient times, mainly to recover hemp and flax fibers (Fig. 6). Today, this treatment is a completely automated process but steps have not changed (Fig. 7): a first separation is carried out by **breaking**, that is the stalks are passed between fluted rollers to crush and break the woody core (shive) into short pieces (hurds); the remaining fibers and hurds are subjected to **scutching** (traditionally performed with boards and hammers), by which the fiber bundles are gripped between rubber belts or chains and carried past revolving drums with projecting bars that beat the fiber bundles, separating the hurds, and broken short fibers (tow) from the remaining long fibers. Finally, in the **hackling** (realized in the past by pulling the fibers through a set of pins) thick fibers are divided by passing the long fibers through a series of combs of increasing fineness to clean and align the long fibers and separate the remaining tow and debris. Interestingly, the modern mills maintain the integrity of the long fibers, by disentangling and aligning the fibers, without destroying length. Another process currently used to mechanically separate the fibers is called **decortication** and can be performed by **hammermilling** or **rollermilling** (Deyholos and Potter 2014). In the first case (Fig. 8), single or multiple concurrent drums rotating with hammers projecting transversely from the drum surface beat the straw until the separated hurd/shive and fiber particles can pass freely through some meshes placed inside the machine. In the second case, (Fig. 9), long cylindrical corrugated rollers are assembled in such orientations as to crack the straw stalks while producing minimal damage to the fiber. The two processes differ in the *pro* and *con*: if the hammermilling is characterized by higher throughput capability, the rollermilling gives much greater length control, producing even very long fibers and will better preserve the integrity of the fibers, without damages or entanglements.

The choice of the preferable mechanical retting depends on the type of the fiber, its final application, type of ensuing treatments. Therefore, with these multiple

Fig. 6 Traditional retting of hemp fibers: scutching (**a** and **b** frames) and hackling (**c** and **d** frames) (Weald and Downland Open Air Museum 2016)

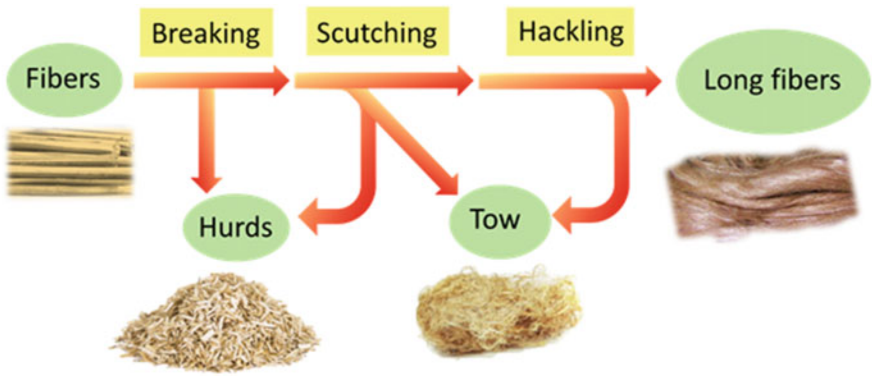


Fig. 7 Mechanical processing of fibers

Fig. 8 Automated decortication by hammermilling

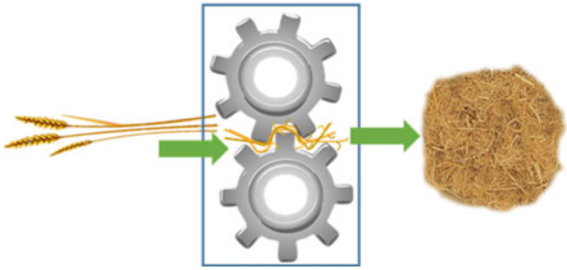
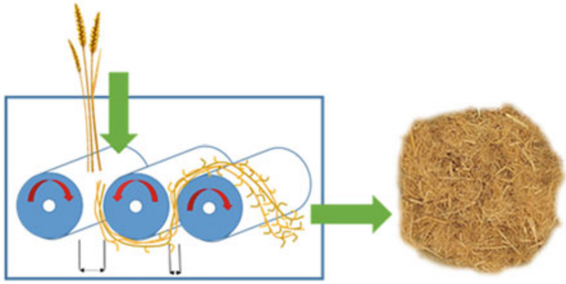


Fig. 9 Automated decortication by rollermilling



variables in mind, it is continuously under investigation. However, it is well known that mechanical retting tends to disrupt the fiber cell wall structures, leading to dislocations, kink bands, or nodes, which negatively affect the tensile mechanical properties (Davies and Bruce 1998; Baley 2002, 2004) and could even impair the composite performances (Huges et al. 2000; Hänninen et al. 2012). Although on the one hand, it was established that the defect extent of the mechanical retting is strongly related to the previous retting treatments (Van de Weyenberg et al. 2003), on the other hand, some researchers found that the fibers can be decorticated without significant damage and can be produced with high quality, even when no pretreatments are carried out (Hobson et al. 2001; Gañán et al. 2008; Bacci et al. 2010; Kengkhetkit and Amornsakchai 2012).

These findings are important and noteworthy: indeed, the elimination of pre-decortication enzymatic or microbiologic treatments should prove advantageous in terms of market costs, as it would reduce the variability of fiber quality, the uncertainties of production, and the amount of time necessary for the field to be available for the next crop. Moreover, new decortication methods can be developed in order to obtain better quality fibers and to reduce production costs by decreasing the times and increasing the treated biomass volumes.

3.4 Physical Retting

Among the physical treatments of fibers, the processes using electromagnetic radiation, high temperature, and/or pressure can be considered.

Steam explosion is an autohydrolysis process involving the use of saturated steam at high pressure followed by a sudden decompression, which causes the substantial breakdown of the lignocellulosic structure, the hydrolysis of the hemicellulose fraction, the depolymerization of the lignin components, and the defibrillation. High decompression rates lead to improved fiber freeness but shorter fiber length. During the process, high temperature softens the material and mechanical action during the high-pressure discharge results in fiber separation: the partially depolymerized lignin becomes more or less soluble in various organic solvents, such as alcohols, acetone, and in alkaline solutions, whereas the cellulose, much more resistant to hydrolysis than pectinic and hemicellulosic polysaccharides, retains its structure. The steam explosion treatment is a fast and well-controlled process, with a low cost and very flexible treatment parameters and is well adapted for the processing of various fibers, including those not previously retted (Zhang et al. 2008a, b). It is successfully applied on banana fibers (Sheng et al. 2014), semi-retted hemp fibers (Thomsen et al. 2006; Garcia-Jaldon et al. 1998), coir fibers (Abraham et al. 2013), flax (Kessler et al. 1998), and wheat straw (Zhang et al. 2008a, b). The steam explosion can be considered as a pretreatment that facilitates the ensuing retting processes (Zhang et al. 2008a, b). It can be carried out downstream, after alkali treatments, bleaching, and sometimes acid hydrolysis (Abraham et al. 2013; Sheng et al. 2014) in order to completely degrade the hemicellulose and lignin fractions. Usually, steam explosion is combined with an alkaline pre-soaking to favor the cleavage of lignin-hemicelluloses bonds. The reaction results in the increased solubility of the lignin alkaline solvent and in an enhanced water solubilization of hemicellulose (Sheng et al. 2014; Thomsen et al. 2006; Garcia-Jaldon et al. 1998; Abraham et al. 2013).

Another interesting physical treatment to extract the fibers is based on the **hydrothermal method**: the lignins and hemicellulose are degraded by using water at elevated pressure and temperature. This approach has been applied on hemp fibers by Thomsen (Thomsen et al. 2006) and on flax fibers by Stamboulis (Stamboulis et al. 2000), who carried out the so-called Duralin treatment, a sort of curing process at different times and temperatures, that leads to an easy separation of the fibers from the stem by a simple breaking and scutching operation. The fibers obtained via such procedures are fiber bundles rather than individual fibers and they exhibit improved moisture resistance and a somewhat higher and more uniform strength.

Recently, a Polish group (Konczewicz and Wojtysiak 2015; Kozlowski et al. 2013) has developed a new **osmotic degumming** of flax fibers. The degumming mechanism is based on the diffusive penetration of water inside the stem, where the long bundles of cellulosic fibers are clustered in slivers with polysaccharides, mostly pectins. The pectins, which are highly absorbent, increase their volume several times, which results in a considerable increase of hydrostatic pressure inside the stem and leads to pressing the epidermis. As the peripheral tension is stronger than the longitudinal one, cracks of the epidermis occur lengthwise, without breaking and shortening the fibers. Since the pectins become diluted and solved (together with other bast substances) in water, the technological liquid is subjected to proper filtration, which also serves to recover pectins for further use in the cosmetic industry. This osmotic method produces fibers characterized by good tenacity, divisibility, and soft touch. It is equally as efficient as the warm water retting method and could be applied to other bast fibers by simply changing the degumming parameters (temperature, flow velocity, and process duration).

To specifically modify the surface of fibers in order to improve their compatibility with polymeric matrix, the **plasma treatment** is an effective physical method, which can be performed at both atmospheric and high pressure under the flow of different types of gas (usually oxygen or argon).

Depending on the material to be treated, plasma flow can cause ablation, cross-linking or surface activation. Ablation consists in the removal of organic residues as well as surface layers at a molecular level. Cross-linking occurs as a result of the interaction between two or more radicals leading to the formation of covalent links while surface activation increases the surface energy as a result of the generation of polar groups on the reinforcement surface (Kafi et al. 2011). Exposure times, pressures, and discharge power are the variables that must be carefully considered to achieve the best results in terms of the surface modification. This kind of treatment is widely used for common natural fibers as flax, cane, coir, and bamboo (Bozaci et al. 2013; Scalici et al. 2016; Praveen et al. 2016; Xu et al. 2006) because, unlike chemical treatments, it is a simple nonpolluting process that can be considered as dry and clean.

The physical treatments are surely not yet completely developed and only few papers describe their use. However, they are characterized by high quickness, easy scalability, and process flexibility, that make this kind of retting noteworthy of further investigation.

3.5 Chemical Retting

With respect to water retting or dew retting, chemical processes are sometimes preferable since they produce fibers characterized by high-constant quality, regardless of weather conditions, usually in shorter times. Numerous chemical treatments can be performed on the fibers depending on their type, the ensuing retting process to be applied, their final applications. The most used chemical process is alkalization, a treatment aimed at removing hemicelluloses: it is usually carried out with sodium hydroxide, added as an aqueous solution at a variable concentration in the range 1–25% by weight. Considering the coir fibers, the NaOH effect is sometimes ambiguous and the reported results are controversial: indeed, if the alkali treatment seems to increase the elongation at break and the surface roughness (Silva et al. 2000) while improving the ultimate tensile strength, the initial modulus, the electrical properties, and the thermal stability (Mahato et al. 1993, 1995), at the same time it decreases the fiber tensile strength with increasing NaOH concentration, demonstrating that the alkalization could induce damages on fiber (Gu 2009). In order to minimize the fiber deterioration, a combination of sodium hydroxide, sodium carbonate, and sodium sulfide and short soaking treatments (only 2 h) have been successfully used on raw coconut fibers (Basu et al. 2015), achieving a reduction in diameter, linear density, and flexural rigidity of fibers.

Similar considerations also apply to kenaf: the alkali retting produces stronger, more flexible and less brittle fibers (Parikh et al. 2002a; Amel et al. 2013) but the base concentration influences the fiber morphology (Ramesh et al. 2015) because the high alkaline medium increases the microvoid volume fractions, leading to the reduction of the tensile strength of the fibers (Kawahara et al. 2005). Milder conditions prevent the degradation of kenaf fibers: the use of a weak base as sodium sulfite leads to cleaner and brighter fibers, smoother, and softer materials, having the higher tensile strength (Umoru et al. 2014). The same reagents have been tested on hemp fibers (Hurren et al. 2002) and it turns out that the combination of NaOH and Na_2SO_3 produces fibers with the whitest color and the finer diameter but not the best spinnability (obtained after acidic treatment with HCl). The need to select weak alkali treatments even on flax fibers has been highlighted by Van de Weyenberg who demonstrates that soaking in a diluted NaOH solution for only 45 s enables better adhesion between flax fibers and epoxy matrix, improving the mechanical properties of the resulting materials (Van de Weyenberg et al. 2006).

To completely remove the lignins and most hemicelluloses, aqueous ammonia treatments are an interesting alternative to alkali retting. Ammonia pretreatment techniques include the ammonia recycle percolation (ARP) and the soaking in aqueous ammonia (SAA). With ARP the biomass is pretreated with aqueous ammonia in a flow-through column reactor; after the reaction, the solid fraction of the biomass is separated whereas the liquid fraction is sent into a steam-heated evaporator in order to separate lignin, sugars, and ammonia, that will be further recycled. This pretreatment was first proposed by Iyer (Iyer et al. 1996) who tried the alkali hydrolysis on herbaceous biomass, as corn cobs and stover mixture and switchgrass, obtaining high efficiency of delignification but preserving the cellulose chains, after only 1 h or less of reaction at 170 °C (Kim et al. 2003). On the other hand, even at low temperatures, the SAA efficiently removes the lignin in the raw material by minimizing the interaction with hemicellulose and leading to an increment of surface area and pore size. The SAA has been applied on barley hulls (Kim et al. 2008) and the scientists obtained 66% of lignin solubilization when treating biomass with 15% aqueous ammonia at 75 °C during 48 h.

A further method to remove the lignin is by oxidative degradation that is commonly used when the target is the complete degradation and consumption of the lignocellulosic mass in order to recover the residues for the chemicals industry or for fuel production. In these cases, degradation is realized using peroxides and the raw biomasses are really varied: wheat straw (Klinke et al. 2002; Bjerre et al. 1996), mulberry (Cong and Dong 2007), poplar wood (Chang and Holtzapple

2000). Interestingly, the pretreatment with hydrogen peroxide of cane bagasse greatly enhances the fiber susceptibility to further enzymatic hydrolysis (Azzam 1989): the reported results show that about 50% of lignin and most of the hemicellulose content of cane bagasse have been solubilized by 2% alkaline hydrogen peroxide within 8 h.

A similar bleaching treatment on kenaf fibers (Shi et al. 2011) completely removes the hemicellulose (the lignin was already eliminated by alkali retting) within only 1 h, retaining the fiber diameter and increasing its crystallinity. Recently, a thorough investigation on the better oxidizing agent, among H_2O_2, NaOCl, NaClO/NaOH, for delignification of green coconut fibers has been described (Brígida et al. 2010). The latter study reports that hydrogen peroxide is more effective to eliminate waxes and fatty acids from the fiber surface but the treatment with NaOCl/NaOH is the most efficient in hemicellulose removal and, consequently, in cellulose exposition (which determines the inherent hydrophilicity and is useful for further functionalization).

A different method to prepare the fibers for ensuing treatments is based on the acidic retting: in such case, the risk to degrade the cellulosic fraction is quite high and the management of the processing waste is likewise complex. Therefore, such methodology is not popular and only a few papers describe this treatment. The use of concentrated strong acids, such as H_2SO_4 and HCl to treat lignocellulosic materials is widely reported when the main target is the complete cellulose hydrolysis (Sun and Cheng 2002). More frequently, strong acids were used in diluted solutions in processes exploiting less severe conditions (Shi et al. 2011a) but usually leading to sugar recovery (i.e., cellulose degradation) (Chen et al. 2007). A fascinating alternative to inorganic acids consists in the organic acids, like maleic and fumaric acids, which can be used for a diluted acid pretreatment (Kootstra et al. 2009). All acids, tested on the retting of wheat straw, drive to sugar formation.

Certainly, chemical treatments are an effective alternative to microbial dew retting which suffers from climatic risks leading to substantial harvest losses. Indeed, chemical retting is not affected by weather variability and can retain the fiber quality. However, difficulties in waste-management and the moderate risk to degrade the fibers currently make such treatments less attractive than in the past.

4 Retted Fibers in Polymer Composites Application

The use of natural fibers for composites is an attractive field from an environmental and sustainable perspective. In particular bast fibers, given their high cellulose and low lignin content, are particularly suited to composite applications and are the most promising replacement for glass fibers in composites (Deyholos and Potter 2014).

Some European countries, where a great environmental consciousness exists, have already introduced the use of lignocellulosic fibers in polymer composites for automotive applications and a great amount of products have been exported to the

US and other countries. Such materials are mostly characterized by nondegradable polymeric matrices, such as polyester, poly(propylene) (PP), epoxy, and phenol formaldehyde (PF). However, recent EU directives establish that all new vehicles must use 95% of recyclable materials in order to mitigate environmental impacts, and this boosted the development of commercially viable biodegradable composites. Therefore, bio-based lignocellulosic composites have a steadily growing market, with a growth projection in North America from US$ 155 million in 2000 to US$ 1.38 billion by 2025 (Satyanarayana et al. 2009).

Natural vegetable fibers can be used to reinforce thermosetting resins (e.g., unsaturated polyesters, epoxy, etc.), thermoplastics (e.g., polypropylene, polyethylene, etc.), and natural polymers (e.g., polylactic acid, polyhydroxybutirric acid, etc.). The most commonly used polymers in natural fibers composites matrices, and an overview of their properties are schematically shown in Tables 3 and 4.

It is well known that lignocellulosic fibers have been used in the field of composites since historical times, and one of their specific applications in aircraft took place as early as the 1940s. In the last decades, reasons for their use have increased,

Table 3 Most commonly used polymeric matrices for composites with natural fibers

Matrix material		
Thermosetting plastics	Thermoplastics	Rubber and natural polymers
Phenolic	Polypropylene	India-rubber
Epoxy	Polyamide	Modifies starch
Polyester	Polyethylene	Polylactic acid
Polyimide	Polystyrene	Cellulose esters
Polyurethane	Polyvinyl chloride	Polyhydroxybutirric acid

Table 4 Overview of the properties of some polymers commonly used in composite applications (Dicker et al. 2014)

Polymer type		Density (kg/m^3)	Glass transition T (°C)	Melt T (°C)	Young's modulus (GPa)	Tensile strength (MPa)	Elongation (%)
Bio	PLA	1210–1250	45–58	150–162	0.35–3.83	21–60	2.5–6
	PHB	1180–1260	4–15	168–182	3.5–4	24–40	5–8
	Starch	1000–1390	–	110–115	0.125–0.85	5–6	31–44
Synthetic	Epoxies	1110–1400	66.9–167	–	2.35–3.08	45–89.6	2–10
	Polyester	1040–1400	147–207	–	2.07–4.41	41.4–89.6	2–2.6
	PP	890–910	0.9–1.55	–	0.9–1.55	28–41	100–600

because of (i) the abundance of sources throughout the world, which represent value-added opportunities for agricultural industries and the possibility to create rural jobs, (ii) the possibility of reducing the dependence on petroleum products, (iii) the availability of complete data concerning fiber structure and properties. Other reasons for their increased use are related to the reduced cost, weight, and energy content in comparison with synthetic fibers (such as glass, kevlar, and carbon) along with comparable tensile strength values, as well as a lower abrasiveness for tooling and the absence of airborne particles, thus reducing respiratory problems for workers (Satyanarayana et al. 2009; Frollini et al. 2013). More in detail, lignocellulosic fibers present unique attributes such as: non-brittle fracture on impact, high flex and tensile modulus, high notched impact, low-thermal expansion coefficient, good sound abatement capability, natural appearance, and the fact that they can be easily colored (useful for textiles applications), full recyclability (Satyanarayana et al. 2009). In Table 5, some properties of the most employed lignocellulosic fibers are reported and compared with those of some man-made fibers. Such properties result from their structure and chemical composition.

Several factors limit the use of bast fibers in composites. The first problem is the lack of homogeneity. As with many natural products, there is large variation in fiber characteristics and composition according to sources (even in fibers grown in the same field and season). Quality production efficiency depends on the environmental conditions, on the cultivar and on growing, harvesting, and primary processing conditions. This lack of homogeneity greatly complicates optimization and standardization of manufacturing processes, where high reliability and stability are required (Deyholos and Potter 2014).

Table 5 Typical properties of some fibers commonly used in composite applications (Dicker et al. 2014; Callister 2007)

Fiber		Density (kg/m^3)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)	Moisture absorption (%)
Synthetic	Carbon HS	1800–1840	4400–4800	225–260	0	–
	E-glass	2550–2600	1900–2050	72–85	1.8–4.8	–
	Kevlar 49™	1444	3600–4100	131	2.8	–
Bast	Flax	1420–1520	750–940	75–90	1.2–1.8	8–12
	Hemp	1470–1520	550–920	55–70	1.4–1.7	6.2–12
	Jute	1440–1520	400–860	35–60	1.7–2	12.5–13.7
	Ramie	1450–1550	500–680	38–44	2–2.2	7.5–17
	Kenaf	1435–1500	195–666	60–66	1.3–5.5	–
Fruit	Coconut	1150–1220	135–240	4–6	15–35	8
Seed	Cotton	1520–1560	350–800	7–12	5–12	7.8–8.5
Leaf	Sisal	1400–1450	550–790	10–25	4–6	10–22
Grass	Bamboo	600–1100	140–800	11–32	2.5–3.7	–

The retting process, which represents the focus of this chapter, is a successful pretreatment for the improvement of matrix/fiber adhesion and the development of high-grade composites. An optimal retting process allows an optimum retting degree, which means that the elementary fibers are released well from the technical fibers and separated from each other by less non-fiber tissue attached on the surface. The performances of the materials increase accordingly, due to the increase of cellulose portions left on the fiber surface, and also by the higher fiber aspect ratio (length/diameter) due to the extent of the separation of elementary fibers. When the retting degree is low, impurities (pectin, lignin, hemicelluloses, etc.) between the fibers can cause stress concentrations in composites and lead to an early fracture (Hu et al. 2012a, b). The development of lignocellulosic fibers-based biocomposites requires the selection of an appropriate biopolymer matrix, suitable surface treatments of the fibers, along with low-cost but high-speed fabrication techniques. Many attempts were made to apply and adapt conventional polymer processing methods, such as compression molding and extrusion followed by injection molding, to the vegetal fiber composite preparation. However, some important aspects of composite manufacturing should be taken into account: preservation of mechanical properties of the lignocellulosic fibers by minimizing attrition and thermal degradation, attaining a high degree of fiber dispersion with control of fiber orientation and ensuring good wettability, despite of incorporation of a high volume fraction of fibers. In general, natural fiber reinforced composites can be manufactured via most production techniques, without using high temperatures (Fig. 10).

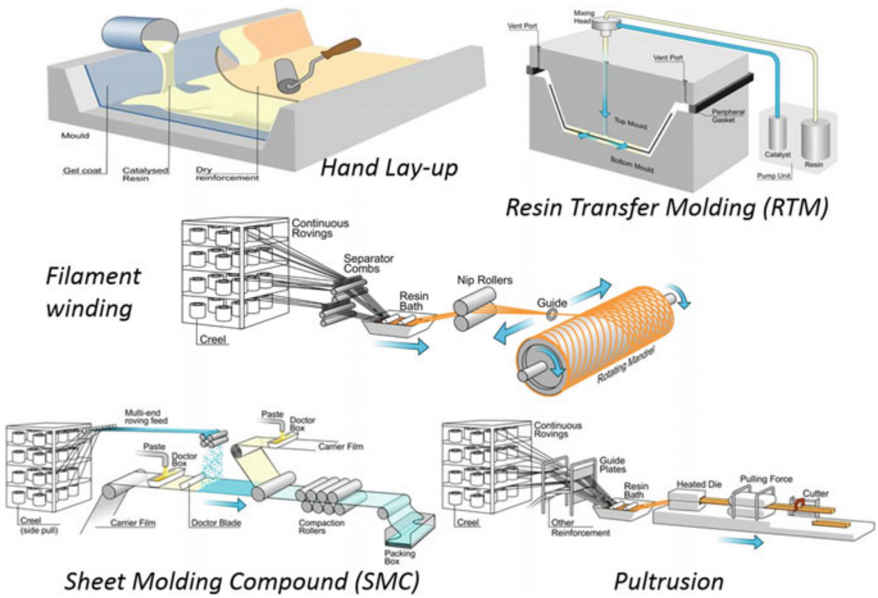


Fig. 10 Most common manufacturing techniques for natural fiber reinforced composites (Nuplex 2014)

Press molding seems to be the best preferred technique (Satyanarayana et al. 2009) since extrusion or melt mixing do not allow to take advantage of fiber length.

A wide range of biodegradable products has been produced using biopolymers containing lignocellulosic fibers for different applications, ranging from automotive vehicles including trucks, construction, and insulation panels, to special textiles (geotextiles and nonwoven textiles). Other identified uses for these materials include bathtubs, archery bows, golf clubs, boat hulls, maintenance-free roofing panels, and longer lasting and better-looking lightweight components, such as cosmetic packaging, tableware, and furniture. Furthermore, thanks to their load-bearing potential, the use of natural fiber-based composites has spread to various sectors, including aircraft, grain and fruit storage, and footwear. Thanks to the identification of all such new applications, a remarkable growth of the market for these new biocomposites is expected in a next future (Satyanarayana et al. 2009).

4.1 Flax

Flax (*Linum usitatissimum* L.) is an important agricultural crop. It is one of the first natural fibers to be extracted, spun, and woven into textiles and now widely used. Flax fibers are produced from the stems of flax bast plants. Like cotton, flax fiber is rich in cellulose, but its cellulose structure is more crystalline, making it stronger, crisper, and stiffer to handle, and more easily wrinkled.

The mechanical properties of flax fibers are reported to be highly dependent on the stem section used to obtain them. Fibers with a different chemical composition (e.g., in terms of cellulose, pectin, and lignin) resulting in different characteristics (e.g., depectinization efficiency and selectivity) are obtained from different stem sections. Fibers from the middle section exhibit higher strength than the fibers from the bottom section (Liu et al. 2015a). Lower fiber grades are used as reinforcement and filler in composites for automotive interior substrates and furniture (Yan et al. 2014).

Concerning the flax retting, Sharma et al. (1999) compare dew-, water-, and enzymatic retted fibers in terms of fiber fineness, strength, ash, and weight-losses, that are the key parameters for determining fiber quality. They are strictly connected to the proportion of residual pectins, hemicelluloses, lignin, and lipids present in the retted fiber, coming from differences in the activities of the polysaccharide-degrading enzymes involved.

In particular, in dew retting, pectinases, and hemicellulases released by the fungal colonists ret the flax slowly in 5–8 weeks. In contrast, water or enzymatic retting can be performed rapidly in 3–7 days, and seem to result in a superior retted material. Most of the pectin from the fiber can be hydrolyzed to improve fiber fineness, but only a limited proportion (nearly 10%) of hemicellulose can be removed without lowering the fiber strength. Removal of residual hemicelluloses bound with lignin will reduce fiber strength. This study, although based on a limited sample size, revealed the superiority of water retted fibers compared to the others.

As far as the enzymatic treatment of flax is concerned, Foulk compared some pectinase-rich commercial enzyme products and demonstrated that Texazym® BFE and Bioprep® 3000 L, as well as Viscozyme®, confer higher tenacity to the fibers, but indicate a potential advantage for Bioprep® 3000 L, in view of its monocomponent nature, commercial availability, price, and ability to ret flax, in combination with ethylenediaminetetraacetic acid (EDTA) at high pH (Foulk et al. 2008).

Good flax fiber quality with a minimum loss in fiber yield was also obtained by steam explosion treatment of flax following dew retting (Kessler et al. 1998).

On the other hand, Van Der Velde et al. test the tensile properties of scutched and hackled long flax fibers on individual fibers and on bundles and notice no significant influence of the retting degree on tensile strength (Van Der Velde and Baetens 2001). Pillin et al. also validate such conclusion in a study about the mechanical properties of oleaginous flax fibers as a function of variety, culture year, dew retting degree and agronomic factors. Despite the interesting mechanical properties obtained, they are not affected by the retting degree. The same conclusion is obtained with agronomic factors, such as seeding rate and plant height (Pillin et al. 2011).

In any case, numerous Authors discuss about the great importance of the retting to achieve high-performance materials.

Some representative examples of retted flax composites are discussed below.

Two kinds of retted Canadian linseed flax fibers, dew, and enzymatic retted fibers, are used in PP composites. The aspect ratio of the enzymatic retted fibers is much higher than that of dew retted as the elementary fibers are better separated. Indeed, the elementary fibers in dew retting remain tightly bundled into technical fiber wrappings with more noncellulose portions. This result induces a lower retting degree and is responsible for a lower thermal stability of dew retted fiber. Moreover, a better retting degree and fewer damages on enzymatic retted flax fibers endowed better tensile properties, thus resulting in a better reinforcement in composites (Hu et al. 2012a, b).

Enzymatic and dew retted short flax fiber composites with recycled high-density polyethylene (HDPE) were prepared and compared with similar products made with wood pulp, glass, and carbon fibers. The composites, with a 30 wt% of fiber loading, were easily blended with a Brabender mixer, because the flax fibers length was 0.21–0.40 mm. A commercial multienzyme product, Viscozyme® L, was combined in varying amounts with a commercial chelator product (Mayoquest® 200). Enzyme and chelator concentrations were denoted: low (0.05% and 5 mm), middle (0.1% and 10 mm), and high (0.3% and 25 mm). Dew retted fiber composites resulted in both lower strength and percent elongation, confirming that enzymatic retting is more efficient in producing fibers suitable for composite preparation. Moreover, enzyme and chelator concentrations did not much affect the final composites properties. Flax fibers improve the elastic modulus with respect to wood pulp/HDPE composites and HDPE alone (Foulk et al. 2004).

Enzymatic retted flax fibers and polylactic acid (PLA) were blended with a twin-screw extruder. The flax fiber content was 30 and 40 wt%. Triacetin was added as a plasticizer, in order to improve the impact properties, because of the brittle nature of PLA. The material properties have been studied and compared to those of PLA and

PP/flax fiber composite. Preliminary results reveal that the thermal properties of PLA improve with the addition of flax fibers. Moreover, the composite strength is enhanced with respect to that of PP/flax fiber composite, even if a poor interfacial adhesion is observed by morphological characterization (Oksman et al. 2003).

Short flax fibers/PP composites have been prepared by Martin et al., who assess the influence of the degree of dew retting of flax on the properties of composites. The length of the fibers was 3 mm, therefore, it was possible to prepare the composites by extrusion, with a 20% of volume fraction. The matrix was grafted with 4 wt% of maleic anhydride, in order to improve the compatibility. Water sorption studies indicate a higher water uptake for fibers with a low retting degree. The tensile properties of single fibers increased with the degree of retting in terms of both Young's modulus and strength at break. A high degree of retting enables easier splitting of the fiber bundles during fiber/matrix extrusion and injection molding, resulting in smaller fiber bundle diameters and a higher aspect ratio. Moreover, a skin/core effect is highlighted: defects are mostly located in the core of samples because the fibers are oriented parallel to the flow mostly in skin areas, resulting in better tensile properties (Martin et al. 2013).

Bodros et al. prepared natural fiber reinforced thermoplastic composites using a film-stacking technique. The materials were manufactured with a 20–30% of volume fraction of dew retted flax fibers 10 mm long, and various thermoplastics, such as PLA, poly(L-lactide) (PLLA), poly(3-hydroxybutyrate) (PHB), poly-ε-caprolactone (PCL), thermoplastic starch (MaterBi® Z), poly(butylene succinate) (PBS), and poly(butylene adipate-*co*-terephtalate) (PBAT). The tensile properties are compared to those of PP/flax composites. Preliminary results show that the tensile properties are improved by increasing the fiber volume fraction. The tensile strength and Young's modulus of PLLA and PLA flax composites prove to be higher than those of similar PP/flax fiber composites. The tensile strength and modulus of flax fiber/PLLA composite are very close to those of glass fiber polyester composites (Bodros et al. 2007).

Chemical treatments are often associated with other types of retting. Baley et al. prepared flax/unsaturated polyester resin composites using dew retted fibers (33 mm long), further subjected to sodium hydroxyde/acetic anhydride or formic acid-based treatments. This classical treatment induces a general increase of the flax fiber/polyester adhesion. The microbond test of a unique microdrop of the matrix on flax fibers allows the assessment of the interfacial adhesion, showing a good mechanical anchorage (Baley et al. 2006).

The chemical treatment with sodium hydroxide, sodium carbonate, and hydrogen peroxide, in addition to a microbiological retting was explored by Abdel-Halim et al. They prepared flax-reinforced epoxy composites by hot pressing from raw, scoured ($NaOH + Na_2CO_3$) and bleached ($NaOH + H_2O_2$) retted, and semi rotted flax fibers in order to investigate the effect of the retting degree as well as subsequent chemical treatments on the mechanical properties of the composites. The fibers were 1 cm long and were loaded in a high amount (almost 67 wt%). The chemical treatment improved the fiber fineness and the surface energy of the flax fibers, thus enhancing the mechanical properties of the composite. Such improvement follows the order: bleached fibers > scoured fibers > raw fibers. Among raw fibers, retted fiber composites had better mechanical properties than semi-retted ones. No improvement is obtained by the use of a titanate on modifying the fiber surface (Abdel-Halim et al. 2008).

4.2 Hemp

Hemp (*Cannabis sativa* L.) fiber plant is one of the world's oldest cultivated annual crops, traditionally grown for its long and strong bast fibers and seeds. In most western countries the cultivation of hemp was interrupted for decades (after 1980) as a result of competition with other feedstock, such as cotton and synthetic fibers, high labor costs, and the prohibition of cultivation due to the use of cannabis (*C. indica*) as a narcotic. The crop can grow in a wide range of geographic and climatic zones, and adjusts well to most regions of the world.

Hemp is used for a wide range of products, and has integrated many agro-industrial fields such as agriculture, textile, biocomposites, paper-making, automotive, constructions (as materials for building and insulation), bio-fuel, food, oil, cosmetics, personal care, household electrical appliances, packaging, and pharmaceutical industry (Salentijn et al. 2015).

Hemp hurds (also termed "shives"), i.e., the woody and lignified core tissues of the stems, are used as horse-bedding, pulping, and concreting. In addition to the traditional uses, novel applications for fiber hemp are being developed. The high cellulose content of hemp cell walls together with the [its??? Riferito a biomass] relatively high productivity make hemp biomass an interesting renewable feedstock for energy production, for the production of second generation bio-ethanol and as a reinforcement in "green composite" materials and concrete. It is estimated that the global market for hemp consists of more than 25,000 products (Salentijn et al. 2015). Hemp is more lignified than flax.

As reported for flax fibers, the effect of the stem section on the mechanical properties of hemp fibers is notable: fibers from the middle section of stems exhibit higher tensile strength and elongation than fibers from the bottom and the top. Compared to flax fibers, the most disadvantageous feature in hemp fibers is the presence of inferior secondary fibers (Liu et al. 2015a). In general, hemp bast fiber is organized in layers from the innermost xylem toward the surface consisting of cambium, secondary, and primary fibers, epidermis, and cuticle, as described by Liu et al. (2015b). Secondary fibers in hemp are primarily located at the bottom of the plant stem and are much shorter (approx. 2 mm long) and thinner (approx. 6 μm in diameter) than the primary fibers (approx. 20 mm long and 10–40 μm in diameter). The presence of secondary fibers may contribute to make the fibers in the bottom stem section poorer. Moreover, as already expressed, fibers from different sections show a different chemical composition, which may affect fiber extraction with biological methods, resulting in different responses (e.g., depectinization efficiency and selectivity) at different stem sections (Liu et al. 2015a).

The optimal growth stage for harvesting hemp fibers for use in composites is reported to be at the beginning of flowering when blooming begins (Liu et al. 2015b). Authors noticed that fibers harvested at an early blooming stage exhibit high tensile strength and strain, which decrease with plant maturity. Reduction in strength was related to the increase in the proportion of secondary fibers and decrease in cellulose deposition leading to inferior properties of fibers. The same Authors also noticed that extended retting dew (i.e., 70 days) had a detrimental effect on the mechanical performance of the fibers, presumably due to the accelerated degradation of cellulose by the action of microorganisms (Liu et al. 2015b).

Some representative examples of various types of retted hemp composites are discussed below.

Composites of PBS with retted and unretted hemp fibers were prepared by compression molding. The fiber content was 10, 20, and 30 wt%, while the fiber length was 3 cm. The efficiency of the retting process, carried out with warm inoculated water, was evaluated by analyzing the final performances of the materials. All the composites showed good mechanical properties, in particular, the composition of 20 wt% retted hemp fibers provided the best performances thanks to a stronger interface between fibers and matrix (Sisti et al. 2016).

Two low-cost retting methods were exploited for the preparation of short hemp fibers/PP composites. The fibers were retted in a bag in the first case, and in the presence of white rot fungi in a second experiment. The composites were prepared by extrusion, with 40 wt% of hemp fibers content (10 mm long). It was found that all the treatments increase the tensile strength of composites. In particular, composites prepared with white rot fungi *Schizophyllum commune* (*S. com*) have the highest tensile strength (Li et al. 2009).

Physical retting treatments have also been reported. Keller et al., for example, used hemp fibers with different length for the extrusion of poly(3-hydroxybutyrate-*co*-hydroxyvalerate) (PHBV) and copolyester amide (PEA) composites. The fibers, 8 mm long, were separated by a steam explosion process, while other fibers, 15 mm long, were degummed by biological processes. Composites with a fiber volume fraction of up to 42% could be achieved. The tensile strength and the Young modulus of PEA were improved by the reinforcement with 27% of fibers. In case of the brittle matrix PHBV instead, no improvement of the tensile strength was achieved, whereas its Young modulus was increased by short-fiber reinforcement, though this was at the expense of impact strength (Keller 2003).

PP/hemp fibers composite films with various amount of fibers (0–30 wt%) were prepared by melt blending and hot pressing. The fibers were chemically treated and then steam exploded. The mechanical properties of the materials resulted to be improved, in particular after a surface fiber treatment with polypropylene-maleic anhydride copolymer (Vignon et al. 1996). Similar materials have also been prepared by Pickering et al., who obtained the best mechanical results with the composite containing PP, 40 wt% of chemical retted hemp fibers and 3 wt% of a maleated polypropylene coupling agent (Pickering et al. 2007b).

Moreover, Bledzki et al. used alkali retted hemp fibers for PP and epoxy composites processed by the film-stacking technique in combination with the filament winding system (30–35 vol.% fiber content). Retted fibers and/or fibers treated with maleic anhydride-polypropylene copolymer were employed, obtaining good tensile strength (Bledzki et al. 2004).

Mwaikambo and Ansell observed an increase in the tensile properties of hemp fiber reinforced cashew nut shell liquid composites following alkalization of fibers. Nonwoven fiber mats and unidirectional fiber composites were manufactured by hand layup compression molding and the increase was observed for both types of composites (Mwaikambo and Ansell 2003).

The alkali treatment was successfully used by Mehta et al., who prepared hemp fiber mats/polyester resin composites by compression molding and the materials showed an increase in mechanical properties. The only exception was their impact strength which was found to decrease following alkalization treatment (Mehta et al. 2006).

Aziz and Ansell studied the mechanical properties of hemp and kenaf-fiber reinforced polyester composites, untreated and with alkali treatment. The composites have been prepared with short, long, and random mat fibers by hot pressing. The alkali treated fibers of both types of composites showed superior flexural strength and flexural modulus values compared to untreated fibers (Aziz and Ansell 2004).

4.3 Kenaf

Kenaf (*Hibiscus cannabinus* L.) belongs to the Malvaceae family, and is a fast growing, multipurpose crop with several harvested components: leaves and tender shoots are suitable for forage; seeds have an oil and protein composition similar to cotton seeds; the woody core can be a substitute for a number of different forest products; and the long bast fibers, traditionally used for cordage, are expanding into the field of composite materials (Ramesh et al. 2015). Kenaf grows in tropical and subtropical areas, 4–5 months after planting, with heights of 4–5 m and 25–35 mm in diameter (Umoru et al. 2014).

This plant features the highest carbon dioxide absorption among other plants (1 t of kenaf absorbs 1.5 t of atmospheric carbon dioxide). Kenaf fiber has a pale color because it contains less noncellulosic compounds than jute. Its fibers are coarse and quite brittle. It exhibits breaking strength similar to jute (Bledzki et al. 2015).

Kenaf bast fiber has been used for the production of fiber board and particle board, textiles, a fuel, and as a reinforcement material for composites (Amel et al. 2013). Applications of kenaf include newsprint, textiles, chemical sorbents, insulating, and noise-absorbing nonwoven materials for automobiles and structural applications and composite consumer products, such as laptops and cell phone cases (Ramesh et al. 2015).

Some representative examples of various types of retted kenaf composites are discussed below. The most common treatment seems to be the chemical retting.

Xia et al., for example, used kenaf fibers, 50.8 mm long, impregnated with aluminum hydroxide and alkali retted, to reinforce unsaturated polyester composites by the vacuum-assisted resin transfer molding process. The results show that the elastic modulus, tensile modulus, and tensile strength of the composites, as well as their water resistance, increase compared to those of composites made with untreated fibers. The presence of $Al(OH)_3$ proves to be advantageous in improving the interfacial compatibility between fibers and resin matrix (Xia et al. 2016).

Shi et al. prepared PP/kenaf composites by sheet molding. The fibers, 50.8 mm long, were alkali retted, impregnated with calcium carbonate and then compounded with PP films in the weight ratio of 50:50. First of all, the tensile strength of the individual fibers increases significantly (more than 20%) after the treatments and the compatibility fibers/matrix is improved by the inorganic particles. The tensile modulus and tensile strength of the composites, compared to those reinforced with untreated kenaf fibers, increase by 25.9 and 10.4%, respectively (Shi et al. 2011b).

Biocomposites based on the alkali retted kenaf fibers and natural polymer starch have been prepared by Song and Kim, by hot press. 20 wt% of fibers, 50 or 1 mm long, were used in composites. Various plasticizers, such as polyvinyl alcohol (PVA), polyethylene glycol (PEG), and glycerol (G), were added and the results show that the interfacial adhesion in the case of G and PVA is better, whereas in the case of PEG, a detachment of the fiber from starch was observed. Hence, the mechanical properties follow the general trend G > PVA > PEG (Song and Kim 2013).

Shi et al. applied progressive chemical treatments to kenaf fibers in order to prepare nano-scale to macro-scale cellulosic fibers from kenaf bast fibers. The chemical treatments included alkaline retting, bleaching, and acidic hydrolysis to obtain both pure-cellulose microfiber and cellulose nanowhisker (CNW). The chemical components of the different scale fibers were analyzed and CNWs were used for polyvinyl alcohol (PVA) composite reinforcement, prepared by film casting. The incorporation of 9 wt% of CNWs fiber (lengths of 100–1400 nm) in PVA composites increases the tensile strength by 46% with respect to the homopolymer (Shi et al. 2011a).

The successful use of chemically retted kenaf fibers in thermoformable composites with recycled polyester and off-quality polypropylene for automotive interiors has been reported by Parikh et al. (2002b).

Yang et al. prepared by compression molding a series of composites based on poly (hydroxybutyrate-*co*-valerate)/poly(butylene adipate-*co*-terephthalate) (80/20) and retted kenaf fibers. Two fiber lengths, 5 and 10 mm, and a hybrid 1:1 fiber mixture was utilized in composites. Both alkali and pectinase rettings were conducted on fibers. The alkali retted fibers showed synergistic benefits of the use of hybrid fiber lengths on modulus, that is to say, that the Young modulus of the composite containing the two length fiber mixture (5 + 10 mm) was higher respect to the samples with 5 mm and 10 mm alkali treated fibers. Such specific effect was not noticed with pectinase retted fiber samples, but in general, these composites outperformed the corresponding composites with alkali retted fibers (Yang et al. 2014).

Good mechanical properties have been obtained also by Bledzki et al. They prepared by extrusion a series of PP biocomposites with different types of fibers, obtaining the best performances with the sample containing 40 wt% of water retted kenaf fibers, 2 mm long, in comparison with abaca, jute, and wood microfibers composites (Bledzki et al. 2015).

Du et al. used mechanically retted short kenaf bast fibers, with different lengths (1.72–2.75–3.30 mm), to reinforce unsaturated polyester composites fabricated by compression molding. The effects of fiber loadings and aspect ratios on composite tensile properties were predicted using classical models in micromechanics. The results show that both composite tensile moduli and strengths increase consistently when increasing fiber loadings up to 75 vol.% (Du et al. 2010).

4.4 Jute

Jute is a long, soft, shiny fiber that can be spun into coarse, strong threads. Like cotton, it is cultivated just for its fiber. Jute fibers are extracted from the bark of the white jute plant (*Corchorus capsularis*) and from tossa jute (*C. olitorius*) by either biological or chemical retting process. It is also named "the Golden Fiber," because of its golden and silky shine. The length of a single fiber can range from 1 to 4 m. The structure of a jute fiber, consisting mostly of cellulose and lignin, has a polygonal section of various sizes, which results in an uneven thickness of fiber cell walls, and this, in turn causes variations in strength. Similarly to other lignocellulosic fibers, jute bast fiber is separated from the pith thanks to retting. In the case of water retting, cut jute stalks are placed in ponds for several weeks. Microbial action in the pond softens the jute fiber and weakens the bonds between the individual fibers and the pith. The fiber strands are then manually stripped from the jute stick and hung on racks to dry (Bledzki et al. 2015).

In hot and humid climates jute plants can be harvested in 4–6 months, therefore the suitable climate for growing jute is the monsoon season. Temperatures ranging from 20 to 40 °C and a relative humidity of 70–80% are favorable for successful cultivation. Jute requires a weekly rainfall of 5–8 cm with an extra amount during the sowing period. It is cultivated in the world and extensively grown in Bangladesh, China, India, Indonesia, and Brazil but the best quality comes from Bangladesh (Ahmed and Nizam 2008).

Jute fiber is a good insulator, has antistatic properties and moderate moisture retention. Due to a high lignin content (up to 20%), jute fibers are brittle, but strong and have a low extension to break (about 1.5%) (Bledzki et al. 2015). Due to its good spinning quality, it is a good textile fiber. It is suited for making jeans and other heavy-duty types of fabrics (Tahir et al. 2011). Jute fibers are used in many sectors of industry, like fashion, travel, luggage, furnishing, and in the production of carpets and other floor coverings, and, last but not least, as a reinforcement in biocomposites (Bledzki et al. 2015).

Some representative examples of various types of retted jute composites are discussed below.

Prasad et al. reported an example of composite made by chemical retted jute fibers and PP. More in detail, the materials have been prepared by injection molding with different fiber loading (0, 5, 10, 15, 20, 25 wt%) and fiber condition (untreated, NaOH treated at different concentrations followed by bleaching with H_2O_2).

The fiber length was 3 mm. The results showed that tensile strength increases with increase in the fiber loading up to 20%, and then there is a reduction. Regarding the alkali treatment, it seems that concentrations higher than 10% of NaOH, may cause strength deterioration. The tensile modulus of the composite with treated fibers has increased considerably when compared to plain PP and untreated fiber reinforced PP composite (Prasad et al. 2014).

Another example of chemical retted jute fibers has been reported by Graceraj et al. The Authors prepared composites based on a mixture of cashew nut shell liquid (CNSL) resin and polyester resin with alkali treated fibers, by hand layup technique. CNSL was used for the improvement of biocompatibility. The fibers were knitted in the mold, therefore, they were 250 mm long. Different amounts of CNSL (5–10–15%) and fiber volume fraction (5–10–15%) have been used. The fatigue strength could be controlled by the optimization of the composition parameters, that is almost 5–7% of CNLS, 8–24 h of NaOH treatment and 7.5–15% of volume fraction (Graceraj et al. 2016).

The water retting process was instead exploited by Gowda et al. The Authors evaluated the mechanical properties of jute-reinforced polyester composites made by hand layup. Microorganisms (mainly *bacillus bacteria*) decomposed the gums and softened the tissues in 5–30 days, depending on the temperatures (27 °C was the optimum) and the type of water used. The jute fibers contained a large numbers of short fibers and few long ones. The Authors tested the mechanical properties and concluded that the composites have better strengths than wood composites. Therefore, they could be suitable for indoor applications, such as shelves, partitions, wash basins, and table tops, and for outdoor uses, such as roofing, drainage pipes, automobile components, electrical fittings as well as larger items, such as light-weight fishing boats (Gowda et al. 1999).

Hassan studied the mechanical properties of phenol formaldehyde resin/jute composites. The fibers have been acetylated or steamed in a rotating autoclave and then sprayed with different levels of PF resin; 12 sheets were overlaid and pressed. The effect of the amount of resin (12–30%), on the flexural strength, tensile strength, water absorption, and thickness swelling of the composites was studied, as well as the effect of steaming and acetylation on the structure and thermal stability of jute fibers. Steaming resulted to be superior to acetylation in improving the dimensional stability. However, the Authors concluded that, due to the lower thermal stability of the steamed and acetylated jute as compared to untreated jute, the use of a resin having curing temperature <160 °C, for example, urea formaldehyde, is recommended, if high-mechanical properties along with high-dimensional stability are required (Hassan 2003).

Unfortunately, various authors do not specify exactly the type of retting in case of jute fibers. For example, Hu et al. used the film-stacking hot pressed method to prepare composites with polylactide film (0.3 mm thickness) and short jute retted fibers (10–15 mm length) in a volume fraction of 30, 40, and 50%. The Authors concluded that, when the volume fraction of fibers in the composite was 40%, the composite had better mechanical properties (Hu et al. 2007). Moreover, Hu et al. employed a more uniform fiber blending method to fabricate composites with retted jute fiber (100 mm) and short PLA fibers, reaching the 70% of fiber volume fraction. The fabrication process includes two steps: felt making and hot pressed molding. The composites showed very good formability and processability (Hu et al. 2012a, b).

Roe et al. used retted jute fibers and a polyester resin matrix to form uniaxially reinforced composites containing up to 60 vol.% of fibers. The composites were fabricated in a lossy mold comprising demountable steel plates forming interleaved troughs 12.7 mm wide and 40 mm deep and the fibers were cut exactly to the length of the mold. The tensile strength and Young's modulus, work of fracture determined by Charpy impact and interlaminar shear strength have been measured as a function of fiber volume fraction. The Authors conclude that the resin forms an intimate bond with jute fibers up to a volume fraction of 0.6, above which the quantity of resin was insufficient to wet fibers completely. Therefore, good quality composites with very acceptable specific properties were assessed (Roe and Ansell 1985).

Good mechanical performances have been achieved also in some mixed systems as reported, for example, by Ramnath et al., who prepared composites by hand layup technique with layers of abaca and retted jute fibers with different orientation. The materials were laminated on top and bottom with glass fibers, in order to improve the surface finish and strength (Ramnath et al. 2013). Sature et al. also reported about the good mechanical reinforcement given by jute/hemp epoxy composites laminated with woven glass mat on top and bottom by hand layup technique. Water retting was applied to jute fibers (Sature and Mache 2015).

Finally, examples of retted jute/epoxy composites made with the vacuum-assisted resin infiltration have been reported by Hossain et al. (2013), while a mixed system based on the hemp and jute/polyester composites has been reported by Hughes et al. (2002).

4.5 Other

In addition, the literature also reports examples of composites based on the other types of fibers, deriving from leaves (such as palm, abaca, sisal, etc.) or grasses (such as bamboo, elephant grass, etc.).

For example, sugar palm fibers, treated by a seawater retting process, were successfully used to reinforce epoxy composites. The results showed that the tensile and flexural strengths of the composites increased proportionally to the fiber treatment duration thanks to a good fiber-matrix bond (Leman et al. 2010).

Epoxy composites have been also reinforced with different fiber contents of royal palm fibers. The fibers were collected from a royal palm tree through the process of water retting and mechanical extraction, and further subjected to alkali treatment which resulted to be effective in improving the tensile and flexural properties while the impact strength decreased, with respect to the untreated fiber composite (Goud and Rao 2011).

Moreover, retted royal palm and bamboo fibers were used to reinforce a polyester resin matrix, tested for tensile, flexural, and dielectric properties and compared with similar sisal and banana fiber composites. It has been observed that the tensile and flexural properties increase in composites containing palm and bamboo fibers, as well as the dielectric strength in palm fiber composites, useful for electrical insulation applications (Rao et al. 2010).

Bamboo fibers, retted by the action of bacteria and moisture and then mechanically extracted, have been used as reinforcements in polyester resins for insulating materials. The results showed indeed that the thermal conductivity of composites decreased with the increase in fiber content (volume fraction 0.1–0.3). Therefore, this composite can be used in the building and automotive industries to save energy by reducing the rate of heat transfer (Mounika et al. 2012).

Polyester resin composites were also prepared with jowar. Experiments of tensile and flexural tests were carried out, and the samples were compared with sisal and bamboo composites. The fibers were extracted by water retting and manual process. The composites present high strength and rigidity, suitable for lightweight applications compared to conventional sisal and bamboo composites (Prasad and Rao 2011).

Sisal fibers extracted by field retting process were subjected to a chemical mercerization treatment and used for the production of PP composites, leading to better flexural and tensile properties, when compared to untreated and unreinforced PP matrix (Oladele and Agbabiaka 2015). Moreover, sisal fibers were used to reinforce unsaturated polyester resin composites. The fibers were subjected to both field and chemical retting (NaOH, KOH, H_2O_2, and ethanol). The tensile and hardness properties of the polyester composites turned out to be enhanced with KOH treatment (Oladele et al. 2014).

Other types of fibers investigated for polyester resin composites are wildcane grass fibers, that were water retted, in order to produce inexpensive materials with high toughness (Prasad et al. 2011). Elephant grass stalk fibers were incorporated in a polyester matrix in order to obtain materials with enhanced mechanical properties. The fibers were extracted using water retting or chemical treatment and the latter proved to be more promising (Rao et al. 2007). Chemically retted elephant grass fibers were also used to reinforce PLA composites, resulting in materials with higher tensile and impact strength when compared to similar lignocellulosic fibers/PLA composites (Gunti et al. 2016).

Cornhusk fibers instead were used in PP composites. The fibers were alkali and enzymatic (pulpzyme and cellulase) retted. The composites resulted in increased mechanical and sound absorption properties, therefore a possible application of these composites may be in structural components for automotive interiors such as headliner substrate (Huda and Yang 2008).

5 Conclusions and Future Prospectives

The main disadvantages of natural fibers in the reinforcement of composites are the poor compatibility between fiber and matrix and their relatively high moisture absorption. Therefore, natural fiber modifications trough extraction processes are necessary to improve their adhesion with different matrices. In recent years, the various retting techniques have experienced a sizable evolution and improvements that tend to minimize the lack of consistency in fiber qualities and the high levels of variability in fiber properties. As described above, the technology based on enzymes seems to be the most promising retting technique, due to its environmental friendliness, shorter processing times, and acceptable fiber quality. However, at present, it is not yet feasible and developed on a large scale, due to the high cost of the process.

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