

## Review Article

# A Comprehensive Review on Bast Fibre Retting Process for Optimal Performance in Fibre-Reinforced Polymer Composites

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Natural fibres are a gift from nature that we still underutilise. They can be classified into several groups, and bast natural fibre reinforcement in polymer composites has the most promising performance, among others. However, numerous factors have reported influences on mechanical properties of the fibre-reinforced composite, including natural fibre retting processes. In this review, bast fibre retting process and the effect of enzymatic retting on the fibre and fibre-reinforced polymer composites have been discussed and reviewed for the latest research studies. All retting methods except chemical and mechanical retting processes are involving secretion of enzymes by bacteria or fungi under controlled (enzymatic retting) or random conditions (water and dew retting). Besides, enzymatic retting is claimed to have more environmentally friendly wastewater products, shorter retting period, and controllable fibre biochemical components under mild incubation conditions. This review comprehensively assesses the enzymatic retting process for producing high-quality bast fibre and will become a reference for future development on bast fibre-reinforced polymer composites.

## 1. Introduction

Due to the alarming rise of global warming issues and perishment of marine living organisms caused by accidentally swallowing nondegradable plastic products, awareness of plastic disposal issues (difficulties in recycling, environmental burden, and high recycle cost) had been heightened. As a result, bioplastics have gradually substituted conventional plastics in many applications [1, 2]. However, many users are still struggling to find suitable replacements as bioplastics have inconsistency and low performance profile. Therefore, reinforcement of natural fibres on plastics was reported to strengthen products with better/or maintaining biodegradability [3, 4]. The natural fibres are renewable resources because they are produced as a part of the plant from photosynthesis, where  $O_2$  is released by absorbing  $CO_2$  gas. Therefore, they decompose naturally, consequently imposing lesser burden to our environment.

Natural fibres can be extracted from three sources (plants, minerals, and animals), as shown in Figure 1. The

main component in mineral and animal fibres is asbestos or basalt and protein, respectively. Plant fibres themselves can be recognised as a composite material since they are composed mainly by cellulose, hemicelluloses, lignin, and other components. Performance of natural fibres is often influenced by their chemical composition and physical properties [6]. However, noticeable differences in performances were found in every single natural fibre even though taken from an identical source [7]. Climatic variations, plant variations, and geographical variations were reported to have influenced chemical composition (cellulose, hemicellulose, and lignin) of the natural fibres [8, 9]. Fortunately, their properties can be enhanced by chemical surface treatment as discussed in detail in previous studies [10]. The natural fibres have posed greater characteristics than conventional fibres, such as environmental friendliness, renewability, price, and performance-per-unit-mass. Hence, natural fibres become emerging filler reinforcement in composites, where glass and carbon fibres are being used traditionally [11–14].

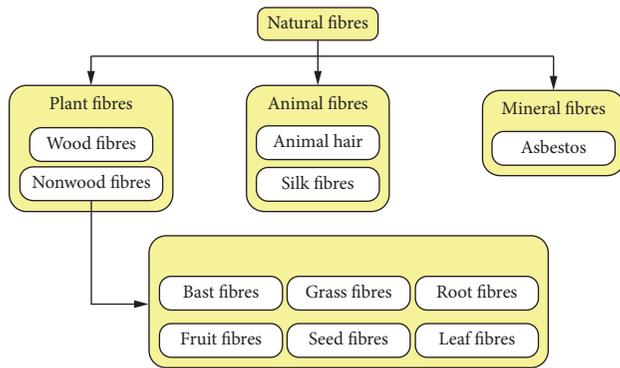


FIGURE 1: Classification of natural fibres [5].

Approximately 2,000 species of natural fibres have been used as composite's reinforcement, but only a few types of fibres are dominating by holding 90% of the natural plant fibre's market [5]. The plant fibres have been further classified into more specific groups, according to the location of the fibre obtained on the plant, as shown in Figure 1. Bast fibres are the most widely used among other groups (bast fibres, fruit fibres, grass fibres, root fibres, seed fibres, and leaf fibres) [15–17]. Retting is the first extraction process to obtain high-quality fibres. Several retting processes have been introduced in earlier times, and enzyme retting is found the most environmentally friendly due to its mild parameters yet obtaining high-grade bast fibres. In this review, the focus is putting on the retting processes for bast plant fibres, emphasising on the enzyme retting process. Numerous studies have been done on bast fibres, but there is a lack of discussion on the overview of bast fibre retting methods. Besides, the effects of enzyme retting on bast fibre and its polymer composites are also discussed in this paper.

## 2. Structural and Chemical Composition of Bast Fibres

Bast fibres are cellulosic fibres that are extracted from the phloem or outer part of the plant (Figure 2). Since bast fibre plants are annual crops, continuous supply of fibres is one of the attractive strong points for gaining interest from non-wood composite manufacturing. Table 1 shows the details of the main five bast fibre contributors.

Bast fibres are extracted from the phloem which is located at the stem of the fibrous plant. Epidermis, shives, woody core, and a combination of xylem must be removed in order to obtain the bast fibres (Figure 3). The epidermis (bark) is used to prevent the plant from moisture evaporation and resist moderate mechanical damage while xylem, woody core, and shives help to transfer water and nutrients from roots to the whole plant [31]. The fibres located in the phloem appear as fibre bundles and provide strength and stiffness to the plant [32]. A fibre bundle consists of numerous single fibres, and each fibre is connected by the middle lamella to act as glue, composed by pectin [2] and lignin components [33]. The major task of retting process is to remove these gluing components and release the fibres from bundle attachment.

Every single fibre is constructed by two layers of walls (primary and secondary wall) and a hollow lumen (Figure 4). The primary wall is built by a network of hemicellulose, pectin, and glycoproteins to protect cellulose microfibrils. On the contrary, the secondary wall, constructed by three layers (S1, S2, and S3) and the middle layer, S2, contributes to about 70–80% of fibre's mass [36]. Therefore, the S2 layer has predominantly varied the properties of the fibre by its cellulose contents, microfibril angle, and thickness [37]. On the contrary, it has been protected from excessive radial expansion and rotation prevention or sideways collapse which are avoided by S1 and S3 layers, respectively [38].

One of the biggest drawbacks is inconsistent performance from fibre to fibre due to the different biochemical profiles of every single fibre. Plant age, fibre source, and retting method are reported to influence chemical composition significantly [39]. Among the bast fibres, hemp fibre has the highest cellulose contents, and therefore, highest tensile strength was expected. Cellulose is the major component that provides stiffness, stability, and strength to the fibre. Hemicellulose is highly hydrophilic with lower molecular weight. It acts as a matrix for cellulose microfibrils. However, it is very susceptible to thermal degradation, biodegradation, and moisture absorption. On the contrary, lignin is a high molecular weight, highly branched amorphous component. It is used as cement in microfibrils to provide rigidity to the plant. In contrast, the fibre with high contents of pectin (found mostly at the middle lamella) is generally high in flexibility [1]. However, easy degradation of pectin affects the stability of fibre's performance. Therefore, retting process tends to remove pectin components as well as release the bast fibres from the fibre bundle.

*2.1. Climatic, Plant, and Geographical Variations on Bast Fibres.* In addition to bast fibre-harvesting strategies reviewed by Pari et al. [40], some important cultivation technologies in Europe and China, to obtain the best quality of hemp fibre bundles, have also been discussed in previous studies [41, 42]. Besides, Liu et al. [43] introduced a new continuous harvest technology for better quality of fibre products. This is because average fibre diameter, fibre breaking strength, and elongation rate were varied due to different harvest modes. Beyond these, plant growth parameters are also affecting the chemical composition of bast fibres naturally. One study has investigated the changes in cell wall thickness, lumen dimension, and cell breath according to the plant age for two varieties of jute plants [44]. If the plant is harvested too early, low yield with soft and immature fibres was obtained, and if they are harvested too late, though the yield is higher, the fibres are less flexible.

However, Liu et al. [45] claimed that not much difference was observed on biochemical properties on early and late harvest fibres. Bennett et al. [46] studied a few growth parameters on dew-retted hemp fibres. According to statistical analysis, hemp variety, seeding rate, and harvest time are the most significantly influencing fibre yield and its properties. Generally, dioecious hemp varieties produce higher fibre yields than monoecious hemp. Under high

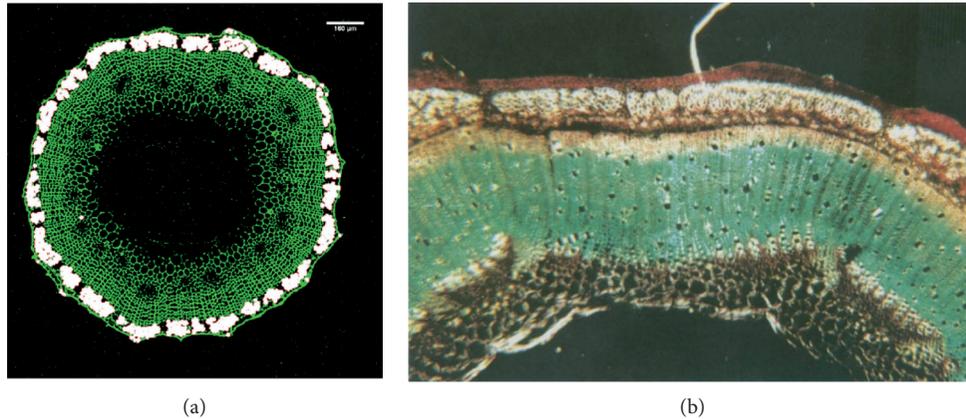


FIGURE 2: X-ray  $\mu$ CT image and optical micrograph of flax and hemp stem, respectively [18, 19].

seedling rate (seeds/m<sup>2</sup>), thinner stem and lower stem volume of the hemp plant were observed.

Besides, a huge variation of bast fibre's chemical compositions was reported due to the climate and soil condition. Jankauskienė et al. [47] studied the weather conditions of Lithuania for years 2010 and 2011. Both years are suitable for hemp plantation except higher relative humidity with richer organic matters in the soil in 2010. Hence, more fertilisers were applied in the latter year. As a result, hemp fibres with higher cellulose contents (81.7%) but with lower lignin contents (9.91%) were obtained. Haag et al. [48] also conducted a similar study for flax fibres for years 2012 and 2013. The results have confirmed that the plantation weather influences fibre's finesses and consequently its mechanical properties.

Kenaf plants are mostly produced in China and India; Bourguignon et al. [49] have migrated them to Iowa and Kentucky, US, to supply local needs. Relatively warmer and wetter conditions of Kentucky yielded higher kenaf productivity and better fibre's performance. However, a low amount of kenaf yielded in 2015 was due to infestation of the Japanese beetle pest. Besides, the plantation of "Tainung 2" kenaf variety is suggested by the authors in US southern states to compete in the local biofibre market [49]. On the contrary, another study outlined a zoning model for agroecological and agroclimatic to analyse potential growing areas for kenaf plantation in Argentina [50].

On the contrary, Liu and Labuschagne [51] studied the effects of nine kenaf variations' plantation on cultivars yields. The findings show the variety of El Salvador has high and stable dry stalk yields among other varieties due to good adaptation to the environment and irrigated condition in South Africa. Besides, warm-season species of kenaf has successfully migrated into the semiarid Mediterranean area [52]. The dry yield has been strongly affected by the amount of water applied, but nitrogen concentration in the soil observed little or no changes in the dry yield. Every 2–24 tonnes/hectares of kenaf yield, under no water limitations but reduced irrigation system, saved 42–45% of water with 23–36% crop yield reduction, showing more cost-effectiveness of the plantation method [52].

Angelini and Tavarini [53] investigated the variation of ramie fibre's chemical components caused by plant densities, harvest time, and crop stand duration. The results concluded that the harvest time has significantly affected fibre's chemical contents, while the other factors found little interaction on basal stem diameter and stem development.

### 3. Retting Process

Fibre extraction from straw is the very first step in fibre processing. At this moment, the outer layer of fibre bundles must be separated from the plant by breaking off the bonds between stem cores and fibre bundles. Réquillé et al. [54] studied the peeling effect on hemp fibres during the retting process. A detailed study on the peeling fracture mechanism helps to understand the conditions of fibre bundle cohesion under the retting process.

A schematic diagram of bast fibre processing is shown in Figure 5. There are two main methods that have been applied to extract bast fibres (mechanical extraction and retting). A comparison between retting methods is shown in Table 2. Previous research studies found that mechanical extraction provides a simple but rapid process with high quantity of fibre yield when compared to the other retting processes. Mechanical extraction uses mechanical forces to tear bonds between the fibre and its core. Plant stalks are fed into a decorticator to break into pieces via compressive, shear, and/or impact forces. Different types of decorticators have been used for fibre processing such as the crushing roller, hammer mill, ball mill, and drop weight. On the contrary, post-decortication cleaning process separates the detached fibres from the mixture of fibre/core-bounded components and fine particles. However, it is hard to control the mechanical forces applied [55]. Also, a wide range of fibre length produced and high in cost are disadvantages of the mechanical extraction [24].

Fibre retting is a complex process, and its properties are highly dependent on the type of retting and its parameters. Under-retted and over-retted fibres made inefficient fibre separation and fibre weakening, respectively [56]. During the retting process, phloem-derived fibre bundles are

TABLE 1: Details of the main bast fibre contributor (flax, hemp, jute, kenaf, and ramie).

Bast fibre	Flax	Hemp	Jute	Kenaf	Ramie
<i>General information [20–24]</i>					
Scientific name	<i>Linum usitatissimum</i>	<i>Cannabis sativa</i>	<i>Corchorus capsularis</i> and <i>Corchorus olitorius</i>	<i>Hibiscus cannabinus</i>	<i>Boehmeria nivea</i>
Plant outlook	Can grow to a height of 80–150 cm in less than 110 days Fibre flax plants are very tall, with few branches and low seed production	Plant stalks can grow to 1.5–2.5 m tall and 6–16 mm thick It has smooth and hollow stems, rough foliage at the top	Can grow to a height of 2–3.5 m with high lignin content during their lifespan of 120 days Able to absorb 15 tons of CO <sub>2</sub> and release 11 tons of O <sub>2</sub> for one hectare of jute plants	Relatively easy to grow with high yields, grow to 5 m tall in 5 months Produces about 6–10 tons of dry matter per acre in a year	Plant stalks can grow to 1–2.5 m tall and 8–16 mm thick
Climate for growth	Grow in moderately moist climates	Grow in a mild, humid atmosphere, and 625–750 mm/year of rainfall is needed Central Asia,	Hot and humid climate	Tropical and subtropical regions	—
Country	Europe and Asia	Eastern Europe, and equatorial countries	India, Bangladesh, China, and Uzbekistan	Northern Africa, India, Russia, and China	China, Philippines, Japan, Brazil, and Europe
Fibre quality	The fine, long flax fibres are usually spun into yarns for linen textiles	Hemp fibres are less flexible and coarser than flax fibres	Long jute fibres are ranging from 1–4 m with the polygonal section of various sizes with a wide lumen, resulting in a high deviation of fibre diameter, which in turn causes variations in strength The fibre has moderate moisture retention and good resistance to microorganisms, but not to photochemical and chemical attack	A potential substitute fibre for jute fibres Preferred over other fibres because of its homogeneity, uniform fibre orientation, and good carbon footprint due to kenaf's high CO <sub>2</sub> absorption	Retting is not possible due to high gum contents (xylan and araban content up to 35%); the degumming process is more preferred
<i>Chemical properties [25, 26]</i>					
Cellulose (%)	62–71	67–75	59–71	45–57	68–76
Hemicellulose (%)	16–18	16–18	12–13	21.5	13–14
Pectin (%)	1.8–2.0	0.8	0.2–4.4	3.0–5.0	1.9–2.1
Lignin (%)	3.0–4.5	3.0–5.0	11.8–12.9	12.0–13.0	0.6–2.0
Wax (%)	1.5	0.7	0.5	—	0.5
<i>Fibre properties [25, 27, 28]</i>					
Moisture content (wt%)	8–12	6.2–12	12.5–13.7	—	7.5–17
Angle microfibril	5–10	2–6.2	8.1	9–15	7.5
Average diameter ( $\mu\text{m}$ )	15–30	10–40	—	—	34
Density ( $\text{kg/m}^3$ )	1530	1520	1520	1450	1500
<i>Mechanical properties [27–29]</i>					
Tensile modulus (GPa)	58	70	60	14–38	18.3
Tensile strength (MPa)	500–1500	920	860	240–930	399
Elongation at break (%)	3.27	1.7	2	1.6	—
Moisture absorption (%)	7	8	12	—	—

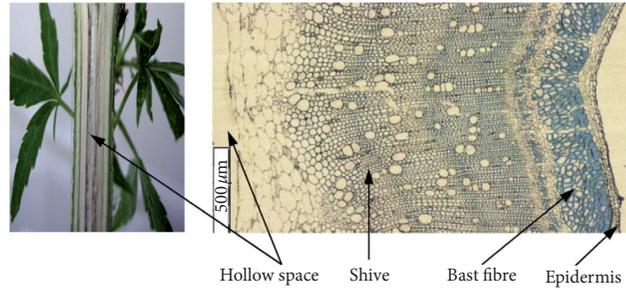


FIGURE 3: Cross section of the hemp stem and location of the bast fibre [30].

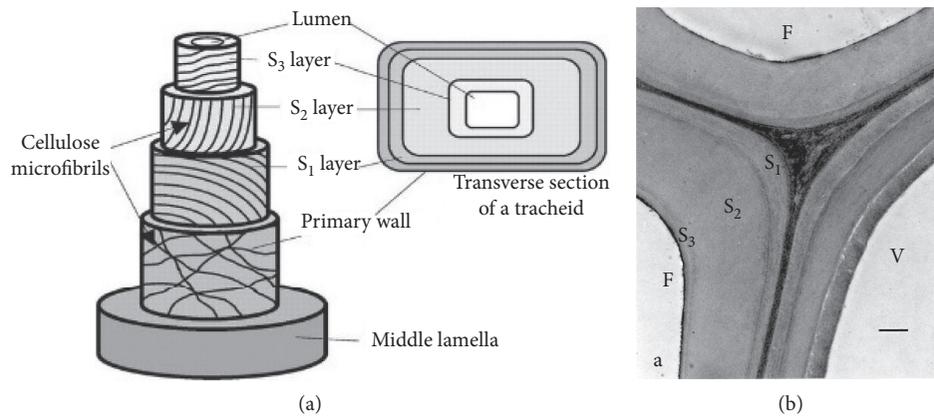


FIGURE 4: (a) Schematic of a single fibre structure [34] and (b) TEM micrograph of the cell wall layer showing the middle lamella (darkest shaded colour between fibres), S1, S2, and S3 [35].

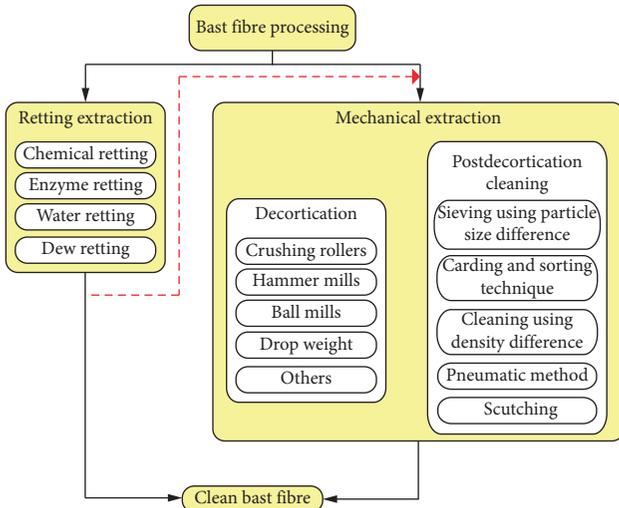


FIGURE 5: Schematic diagram of bast fibre processing [5].

loosened from hemicellulose, lignin, and pectin. Leftover fibres are rich in cellulose contents and hence performing in high-strength properties. Sisti et al. [57] justified the effectiveness of the retting process on pectin, wax, and lignin removal, thus breaking up the bonding bounded between hemp fibres, thereby improving adhesion between fibres and the matrix during composite fabrication.

Retting is a biological process, which removes noncellulosic materials attached on the fibre bundle by enzymatic activities, consequently yielding detached cellulosic fibres. All retting processes except chemical retting use enzymatic activities to extract fibres from bundles. Water retting and dew retting apply anaerobic bacteria fermentation and the fungi colonisation method, respectively, on fibre bundles, to produce enzymes that hydrolyse fibre-binding components. *Clostridium* sp. that lives in lakes, rivers, and ponds is an anaerobic bacterium. It rets bast fibres by producing pectinase enzymes during the water retting process. However, anaerobic fermentation has resulted in severe water pollution, contaminated wastewater, and putrid odour and hence resulted in introducing the dew retting process [58]. Dew retting uses colonisation of fungi presented in soil on bast stems [59]. Unfortunately, it is a time-consuming process with inconsistent outcomes, in which fibre quality depends on geographical conditions; even though this method is found cheaper, it yields higher fibre content and creates less pollution.

On the contrary, chemical retting produces a more controllable bast fibre quality within a short retting duration. High processing cost, unfavourable colour, and deteriorated tensile strength of the retted bast fibre have driven scientists to discover a better retting method. Enzyme retting has been so popular due to its mild process conditions, specificity, and high selectivity with no chemical present. The

TABLE 2: The comparison between bast fibre retting and extraction process [24].

Retting methods	Description	Advantages	Disadvantages	Duration of retting
Water retting	Plant stems need to be submerged in water and checked periodically	Produces retted fibres with great uniformity and high quality	Severe pollution issue arising from anaerobic bacterial fermentation, putrid odour, environmental problems, and high cost Requires intense treatment on wastewater	7–14 days
Dew retting	The plant stems are spread evenly on fields to receive sufficient sunlight, atmospheric air, and dew for fungal colonisation and thereby breakdown cellular stem tissues and adhesive substances to release the single fibre	Pectin materials could easily be removed	Product contaminated with soil, restriction to certain climatic change, inconsistent quality, and reduced strength	2-3 weeks
Enzymatic retting	Enzymes hydrolyse gum and pectin material in the stem. Controllable retting conditions are allowed to maximise retting efficiency	Specific properties can be achieved for different applications by varying retting period and type of enzymes used The process is cleaner and faster	Low fibre strength	12–24 hours
Chemical retting	Hydrogen peroxide, sodium benzoate, or sodium hydroxide is normally used in chemical retting	The smooth and clean surface can be obtained, inconsistency within a short period	Deterioration of fibre strength when the concentration of NaOH more than 1% is being used High processing cost and unfavourable colour	60–75 minutes
Mechanical extraction	Force applied on the fed stem to separate fibres, then postcleaning, and further filter impurities	High quantities of the short fibre shall be yielded in a short period	Lower fibre quality and high cost	—

characterisation of enzyme-retted bast fibres shows comparable quality as water-retted fibres. However, some studies found lower fibre strength because of continued activity of cellulase in the enzyme mixtures. Therefore, controlling retting duration is essential to avoid over-retting. Song and Obendorf [60] compared water-, chemical-, and enzyme retting process on kenaf fibres. The authors observed that the enzyme retting process has the highest lignin-removal activities based on the evidence of GCMS and FTIR results. Besides, it also allows greater process control under shorter retting duration, which is highly recommended for large-scale production [61].

Pandey [62] studied a variety of retting methods to analyse suitable end uses according to the retted fibre's properties (line and tow fibre percentage, line and short fibre length, fibre tenacity, fineness, and elongation at break). Figure 6 depicts the differences in fibre yield from a variety of retting methods, ranging from 8.8 to 30.0%. The methods are sorted in descending sequences based on fibre yield as follows: enzyme pectinase>gel>EDTA disodium>double retting>mixture of cellulase and/or  $\alpha$ -amylase>chemical retting with hydrogen peroxide>water retting>sodium hydroxide chemical retting. Angelini et al. [63] compared the biochemical composition of ramie fibres obtained from chemical and enzyme retting. A higher percentage of cellulose content and effective removal of hemicellulose and lignin were observed on chemical-retted ramie fibres. Liu et al. [64] studied the effects of EDTA chelator on hemp fibres' retting process. The epidermis of the hemp fibre, which is rich in  $\text{Ca}^{2+}$  ions, is bound to EDTA through proton displacement. Hence, the structure became unstable and resulted in high occurrence of pectin and low-methoxyl

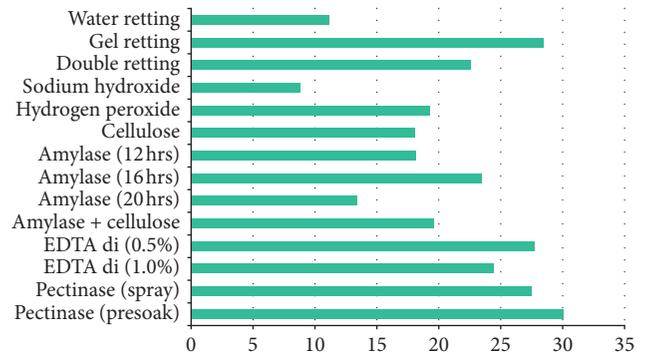


FIGURE 6: Shikha flax fibre yield from a variety of retting methods [62].

pectin removal from the hemp bast fibre. The outcomes were shown similar to another study [65]. Further details of chelators shall be discussed later, in the enzymatic retting section.

Amel et al. [66] studied the effects of retting methods on morphologies and physical and mechanical properties of kenaf fibres. Water- and NaOH-retted kenaf fibres have comparable tensile strength values, which are 426 and 393 MPa, respectively. However, both methods contribute to high strength from different perspectives. Water retting did not shorten fibre length and hence it is capable to transfer higher load. In NaOH-retted fibres a severe reduction in fibre length was observed, yet improved cellulose chain packing yields more crystalline cellulose and thereby higher tensile strength.

On the contrary, water-retted flax fibres show higher strength properties than dew-retted fibres due to the conservation of cross-linking fractions [67]. These findings were agreed by van der Westhuizen [68] who reported that water retting produces the highest fibre strength for all kenaf varieties (Tainung-1, T15, Cuba-108, Everglades 71, and BG52 except Cubano) among six retting processes (enzyme, dew, water retting, NaCl, urea, and NaOH). On the contrary, Yu and Yu [69] had the opposite opinion, suggesting the water retting process produces weak, poor quality of kenaf fibres, evidence of the highest gum residual contents, and lowest tenacity of fibres.

Mazian et al. [70] studied the influences of retting period on hemp fibre biochemical contents. The results showed the development of cellulose over the period from the X-ray diffractogram and higher intensity of the crystalline peak on the specimen retted in nine weeks. Similar results were found in the previous study, increase in crystallinity on retted flax fibres with lower amorphous region [71]. However, different cellulose, lignin, and pectin contents throughout different retting periods are due to different degradation rates. The highest rate is found on pectin removal followed by cellulose and lignin [45].

Besides, colour changes on fibres were also found varying due to different retting periods [70]. The colonisation of fungi during retting causes the fibre to change its colour from light green-yellow to dark grey [72]. Superior tensile performance on 19-day-retted flax fibres ( $1036 \pm 270$  MPa) was recorded due to a low degree of impurities.

### 3.1. Steam Explosion (STEX) Pretreatment Prior to Retting.

Pretreatment prior to the bast fibre retting process is commonly applied to enhance retting efficiency. Pretreatment allows higher penetration of enzymes into fibre bundles and produces better fibre quality. Among various techniques, STEX process is one of the most widely used due to its high-efficiency-to-low cost advantage. STEX is a thermo-mechanical-chemical defibration method. It is widely used as pretreatment to enhance retting efficiency. During this process, fibres are both chemically modified and mechanically defibrated. STEX process breakdowns lignocellulosic structural components by heating, formation of organic acids, and creates shear forces to the fibres. Processes of hemicellulosic hydrolysis, chemical structure of lignin, and cellulose crystallinity index alteration are ways to destroy lignocellulosic structures and thereby obtain better retting efficiency. At the end of the process, instantaneous release of pressure stops the reaction and separation of fibres from bundles. This process is suitable for the woody bast core, which can be purified to provide chemical-grade cellulose or quality fibres for textiles and composites [73]. A study shows that this process removes amorphous materials from the inner part of the hemp fibre via depolymerisation and defibrillation [74].

Ramie provides a longest and strongest natural fibre material in the textile industry, but traditional degumming processes are costly and require a large amount of alkali,

which causes serious environmental concerns. STEX treatment is an efficient and environmentally friendly method for degumming of various natural fibres, but the treatment alone has very low retting efficiency. Hence, subsequent chemical degumming treatment is necessary. Jiang et al. [75] reduced gum contents on the ramie fibre to below 5% and 11.65% for STEX process, with and without the environmentally friendly and economically feasible reagent, sodium percarbonate (SP) bleaching agent [75]. Besides, more than 50% of chemicals were recycled, only 35% of pollution level as compared to the traditional process. Lower tenacity and fineness of ramie fibres were recorded. However, the retted products achieved refined dried ramie fibre requirements from the Chinese National Standard (GB/T 20793-2006) [75].

The STEX process is also certified as an appropriate extracting process for bast hemp fibres. STEX has found noticeable xylan content reduction [76]. Table 3 shows the ratio contents of xylan, lignin, and pectin acid to glucan in various hemp samples. On the contrary, the best condition to separate and purify woody fibres is the steam treatment of acid-impregnated process with 180 seconds duration at 200–230°C [18]. Higher temperature will induce fibre damages.

The STEX has been widely used as a pretreatment technology for lignocellulosic materials to improve enzyme-catalysed cellulose degradation [77]. Pakarinen et al. [76] studied STEX and alkalization pretreatment prior to enzyme retting to enhance hemp fibre properties. The most significant increases in enzymatic hydrolysis were observed with supplemented pectinases in the conversion of anaerobically preserved hemp fibres. The pretreatment induces swelling of microfibrils thereby increasing substrate availability to hydrolyse enzymes. Delignification has enhanced enzymatic efficiency since lignin provides structural rigidity to the fibre, preventing swelling. Besides, removing xylan components also allows swelling of the fibres to increase the surface area and eventually cleave some lignin components. After that, removal of pectin showed a strong correlation with enzymatic hydrolysis due to high accessibility of enzymes between the substrate cell wall surface.

A study by Liu et al. has applied STEX pretreatment on the dried hemp stem by using three different water vapour pressures for 30 minutes before the enzymatic retting process [78]. Reduction in arabinan, galactan, xylan, and lignin contents was recorded, while a gradual decrease in the pH value was responsible for the liberation of acetic acids and galacturonic acids at elevated temperature. Therefore, an observation of better accessibility of pectinases during enzymatic retting was enhanced by hydrothermal pretreatment. The major contribution came from higher water retention and larger macropores for improved enzyme penetration. Besides, clear synergistic action between cellulase and xylanase was observed in the hydrolysis of steam-exploded hemp [79].

Microbial contamination is one of the retting process disadvantages. Further enzymatic hydrolyzation secreted by microorganisms has caused over-retting on the fibre, thereby reducing fibre strength. The microbial quality has

TABLE 3: Ratio content of xylan to glucan, lignin to glucan, and pectin acid to glucan in various hemp samples [76].

Hemp fibre pretreatment processes	Xylan/glucan	Lignin/glucan	Pectin acid/glucan
Untreated	0.21	0.39	0.13
Steam-exploded	0.08	0.23	0.02
Chemical-treated	0.10	0.09	0.00
Acid-ensiled	0.19	0.33	0.13
Alkali-preserved	0.19	0.29	0.13

been investigated on enzyme-retted hemp fibres with and without STEX treatment [80]. The enzyme retting process promoted 300–900 folds of microorganism growth on the fibres. However, tenfold fungal contamination reduction on hemp fibres was observed after the pretreatment.

The findings show that the STEX pretreatment process is significant to increase retting efficiency due to better penetration of chemicals or enzymes into the inner part of fibres. Nonetheless, the STEX process after the enzymatic retting process helps to maintain the quality of retted fibres. Reduced numbers of enzyme activities on retted fibres avoid unintentional retting process to happen, which may deteriorate fibre quality.

**3.2. Water Retting.** Water retting is the oldest historical retting method. This process was once popular in producing quality-retted bast fibres. However, generating huge amounts of wastewater is a major issue, which cannot be ignored [81]. The discussion of water retting parameters and most importantly, wastewater management, including water recycling or nonfreshwater retting process, has been done in this review paper. Besides, some innovative modifications on bast fibre water retting process were also included at the end of this section.

Magnusson and Svennerstedt [82] investigated the effects of temperature on water-retted hemp fibres. However, the findings showed that temperature had little influence on the final yield, yet temperature higher than 45°C would not be suitable for enzymatic activities. A synergetic effect for temperature (37.5°C), pH value (4.4), and retting duration (192 hours) has observed a dramatic reduction of pectin contents [82].

Zhang et al. [83] compared the seawater retting process with the freshwater-retted hemp fibre. It demonstrated a slightly lower-quality fibre. However, the reduction of freshwater consumption would make this method in favour. On the contrary, Boukhoulda et al. [84] found better mechanical properties for seawater-retted alfa fibres. The morphology micrograph shows a smoother fibre surface, and this is because saltwater helps to remove waxes. Previous researchers observed similar findings: water retting produces finer fibres and higher mechanical properties [85, 86]. However, water retting is a time-consuming process.

To check the time factor on the retting process, Fatma and Jahan [87] retted *Kydia* bast fibres by using stagnant water at room temperature for 10–25 days. Highest fibre tenacity and elongation were observed for the 20-day-retting specimen. The results have comparable mechanical and physical properties with other bast fibres (flax and jute

fibres) and SEM micrographic for *Kydia* bast fibres (Figure 7), as an evidence of successful water retting process.

Freshwater retting has been used to produce high-quality bast fibres for many years. Contamination of freshwater during the process is always an environmental debate topic. The high concentration of organic materials and unpleasant smell were produced during the process. A case study on river water-retted kenaf fibres in Malaysia have found that most of the contamination compounds after the retting process exceeded the standard that was regulated by the Department of Environmental Malaysia, which was not suitable to release into the main water stream [88]. This finding has revealed major pollution of water retting.

Abou-Elela et al. [89] investigated the effectiveness of Fenton oxidation process with granular activated carbon on the treatment of retted wastewater (schematic flow shown in Figure 8). Effective results on removing organic and inorganic pollutants were recorded, and treated water was reused in the next retting cycle. Apart from this, in India and Bangladesh, pondwater is used for retting jute fibres, but this has caused high arsenic (toxic metalloid) contents and contaminated pondwater by 4-to-40 folds according to the WHO safe limit (0.05 mg/L) [90]. However, bacterial growth population during retting transformed arsenic-III to less toxic arsenic-V. Therefore, jute fibre cultivation after summer is a great option to stabilise the arsenic content.

On the contrary, solar photo-Fenton oxidation post-treatment was introduced on water-retted pond wastewater. Highly effective COD (97.5%) and phenol compound (98.4%) removal was recorded under optimum parameters [91]. High phenol concentration in wastewater was due to the retting process on phenolic compounds presented in husks or coir, generating dark-coloured compounds to wastewater by oxidation. Therefore, phenol is one of the target components to be removed during the wastewater treatment.

Apart from this, undeniable efforts have been made by innovation modifications on bast fibre water-retting process to increase retting efficiency and reduce freshwater usage as well as shorten retting period. Konczewicz et al. [92] applied a new water retting method with multiple water changes or continuous water flow, named osmotic retting. Water diffuses into the stem where the fibre swelled after absorbing water, which causes pectin to expand in several folds. Besides, increased hydrostatic pressure causes tensions exerted on the epidermis from longitudinal and peripheral directions. It resulted in a weaker pectin bonding strength and then it was dissolved in the flowing water. Generally, all soluble substances containing dyes, bacteria, pectin, and

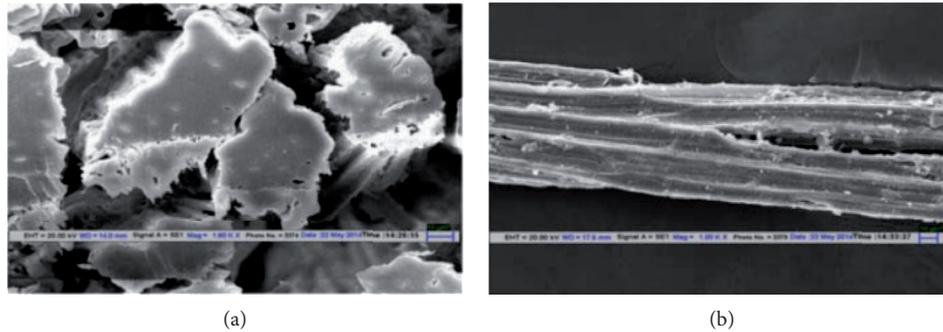


FIGURE 7: SEM images of (a) cross-sectional view and (b) longitudinal view of Kydia fibres [87].

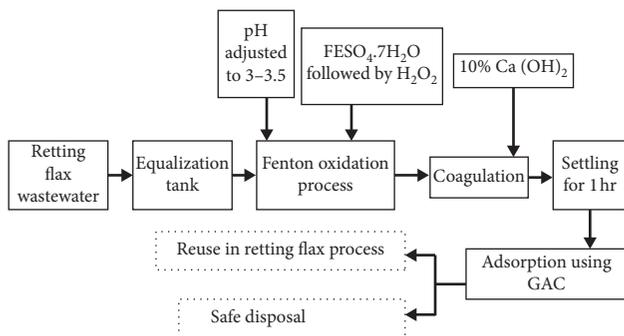


FIGURE 8: Schematic diagram of retting flax by using wastewater [89].

mineral salts are removed from the stem, where high-quality fibre is obtained. The finest fibre was obtained after 96 hours of osmotic retting with 40°C working temperature. This fibre retting method claimed to be more suitable for fibre reinforcement applications [93].

On the contrary, Ruan et al. [94] introduced radio-frequency treatment to improve retting efficiency in a variety of water-retting temperatures and durations. The absorption of the radiofrequency energy has caused cellulose molecules to vibrate violently with pectin molecules, referring to the weakened cellulose molecular chain. However, less amount of cellulose molecules observed in vigorous vibration even maintains the treatment temperature for long periods. This resulted in a higher crystallinity index.

Jahan et al. [95] introduced ribbon retting in Bangladesh for jute-retting process. In ribbon retting, barks are removed from jute in the form of ribbon. The ribbons are coiled and then allowed for retting in water. This method claimed to use a lesser amount of water in a shorter time and more environmental friendly, and the paper product shows no differences in terms of properties between conventional water and retted ribbon materials.

**3.3. Dew Retting.** During the dew retting process, stems of the plant were being cut and evenly distributed in the fields, where the presence of bacteria, sunlight, atmospheric air, and dew causes breakdown of stem cellular tissues and adhesive substances that surrounded the fibres [96]. The places that have a warm day and heavy night dew are

preferred for the dew retting process to promote colonisation of fungi.

Bleuze et al. [97] studied the changes in the flax fibre during the dew retting process. Microbial colonisation has been directly related to the cell-wall chemical compositions. Fungal hyphae and parenchyma were observed on the epidermis and around fibre bundles, respectively, after seven days. It shows partial damage and decohesion on fibre bundles. Higher enzymatic activities further destroy the primary cell wall of polysaccharides on the 14th day [97]. Microbial colonisation at stem's inner core was recorded at the end of the retting process (42 days) by the evidence of parenchyma degradation and fibre bundle decohesion.

Fila et al. [98] isolated 23 types of dew-retting agent fungi from Southern Europe. They have confirmed that all *Aspergillus* and *Penicillium* strains produce high-quality retted flax fibres. Repečkien and Jankauskiene [99] studied the effects of fungal complexes on flax dew-retting accelerating under the field conditions. High colonisation of *Cladosporium* species variants (25–29%) was reported as one of the suitable fungal for fibre separation. The largest amount of fungi persisted on flax treated with fungal complex N-3 containing six fungal strains. Jankauskiene et al. [100] optimised the dew retting process on a commercial scale. Two fungal mixtures were developed and applied on straw after pulling on swathe and/or returning of swathe. Besides, exceptional high fibre separation was observed after spraying of suspension of *Cladosporium herbarum* during fibre harvesting.

**3.3.1. Bacterial and Fungi Interaction (BFI).** Fungi colonisation is considered as the major enzymatic active method responsible for dew retting. However, recent studies have shown interest on the interaction of the bacterial and fungi community during dew retting. Liu et al. [101] studied the relationship between chemical compositions of hemp fibres and microbial community variation throughout the retting process. Fungal colonisation was found in the first seven days with very few bacteria. Gradually, increase in bacterial attachments on the fibre surface with fewer fungal hyphae was recorded after 20 days. The location for high bacterial intensity was found highly degraded. Figure 9 shows the phylogenetic tree for the bacterial and fungal community presented in dew-retting hemp fibres. Table 4 lists the

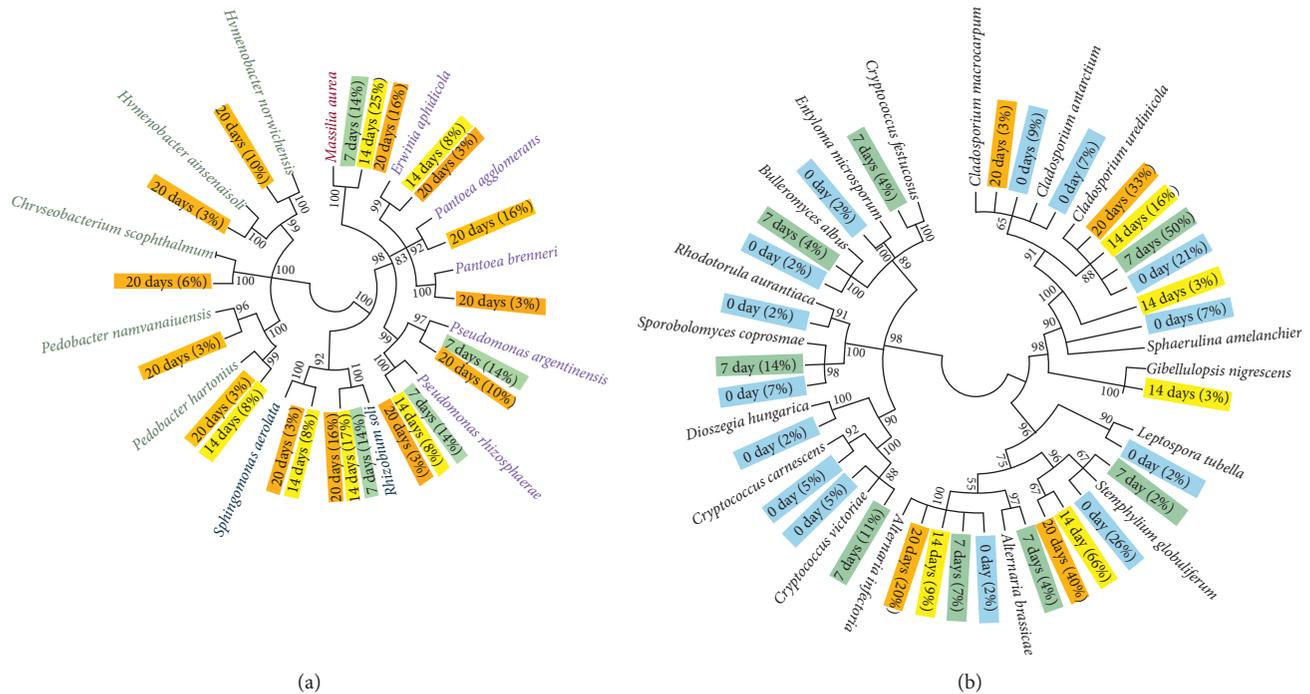


FIGURE 9: Phylogenetic tree of the (a) bacterial community and (b) fungal community present in the hemp fibre samples. Colour of the branches shows a different type of proteobacteria, while colour of the tag shows the number of bacteria/fungi in different days [101].

highlights of ultrastructural changes on the hemp stem and fibres with microbial activities along retting process.

**3.4. Chemical Retting.** Chemical retting degrades non-cellulose components, but cellulose degradation happens when over-retting. Retting duration, chemical concentration, and retting temperature have been reported that affect the quality of retted fibres [5]. Besides, uses of chemicals and production of wastewater have increased the cost as well as polluting environments. One study has reused wastewater for continued chemical retting process. An addition of 50% of chemicals is needed to obtain comparable physical and mechanical properties of the retted fibre as the first retted batch [103]. This method has essentially reduced the cost as well as wastewater generation.

Basu et al. [104] used a combination of sodium carbonate, sodium hydroxide, and sodium sulphide for chemical fibre retting. Effective chemical retting reduces retting time to two hours. The chemical-retted fibre showed positive results toward mechanical properties with reducing flexural rigidity, linear density, and diameter. Softer fibres were obtained at the end of the study by removing impurities. However, contaminated water showed exorbitant chemical oxygen demand, biological oxygen demand, total dissolved solids, sulphide content, and blackish brown colour. Therefore, a new treatment (electrocoagulation) has been introduced to reduce the contamination of wastewater, and the outcome was satisfied. It provides quicker, simpler, and economical treatment by using electrocoagulation [105]. A duration of 90 mins gives noticeable reduction in contamination compounds as well as it is cheaper in cost.

Yu and Yu [69] concluded that chemical retting is the most effective method to remove pectin and release fibres from bundles. Acidic souring and alkaline boiling were used in the study. Parikh et al. [106] studied the caustic soda retting process on kenaf fibres and the effects of additionally applied chemicals. The additional 0.1–0.2% of anthraquinone, 1% of sodium bisulphite, or higher contents of caustic soda only increase 0.77–8.00% of fibre weight loss, which does not justify any significant economic or environmental benefits. However, shorter retting period has been achieved, and the retted fibres are recommended for automotive nonwoven applications.

**3.5. Other Retting Methods.** There are some other retting methods which have been introduced by researchers hoping to have higher retting efficiency. However, such innovative retting methods may not be able to commercialise due to multiple factors like cost issues and not user friendly. One interesting retting method, microwave retting process, was applied on flax fibres by Raveendran Nair et al. [107]. The microwave energy applied breaks strong pectin bonds between flax fibres and increases relative cellulose ratio. On the contrary, short presoaking period increases moisture and decreases glass transition temperature of pectin. Therefore, higher efficiency of pectin removal can be done. 24-hour-soaked samples treated at 2 W/g for 20 min showed maximum retting efficiency. A mathematical model for compositional changes (rate of change of lignin content, hemicellulose content, and cellulose content) during the microwave-assisted retting of the flax stem [108] and method optimization [109, 110] were established in previous papers.

TABLE 4: Highlights of ultrastructural changes on the hemp stem and fibres with microbial activities along the retting process [102].

Retting period	0 days	7 days	14–20 days	After 50 days
Changes in the ultrastructure of the hemp stem and fibres	(i) Stem with the intact layered structure (ii) Uncollapsed, intact cells with native cell geometry (iii) Cytoplasm-filled living cells (iv) Clear surface with undamaged cuticle and trichomes (v) Abundant chloroplasts in the upper epidermis	(i) Overall intact structure (ii) Fungal presence on the surface and inside the stems (iii) Cellular anatomy is less stable with deformed epidermis and parenchyma	(i) Cuticle severely decayed (ii) Changes to the cellular anatomy and major destruction of the living cells (iii) Fibre bundles separated from the epidermis and each other (iv) Thick-walled cells seldom colonized-parenchyma totally degraded, but chlorenchyma has less damage (v) Bast fibres with infrequent mild attack (vi) Fibre morphology affected the characteristics of colonisation and the decay morphology	(i) Hemp structure severely affected and disintegrated (ii) Extensively colonized epidermis and cambium with dominant bacteria (iii) Complete destruction of the parenchyma cells and loss of structural integrity in the bast regions (iv) Hyphae inside the lumina of all the cell types, including the fibres (v) Intensified BFIs inside the stem (vi) Major loss to the anatomy and ultrastructure (vii) Thick-walled bast fibres with decay characteristics (viii) Effects on the fibre wall ultrastructure (a) Loosening/degradation of the CML, which led to delamination and defibration (b) Loosening and decay of the S3 layer (c) Prominent effect on the S2 layer with delamination within the S2 transwall and intrawall fractures in S2 (d) Direct removal of S2 materials (e.g., S2 thinning, broken S2, and disintegration into nanosized cellulose fibrillar structures)
				Fungi (i) Less abundant on the outside of the stem (ii) Surface mycelia in nonliving state, but active hyphae inside the stem (iii) Mycelia, an exclusive source of nutrients for the invading bacteria, reflected bacterial mycophagy (i.e., extracellular and endocellular biotrophic and extracellular necrotrophic activities) Bacteria (i) Highly abundant inside and outside the stems (ii) Highly diverse and dominant role (iii) Visible as dense overlay representing (a) Biofilms (b) Morphologically different colonies (c) Randomly scattered cells (iv) Showed strong BFIs (v) Bacterial motility occurred over and inside the hemp stem using fungal highways (vi) Showed enhanced cutinolytic and cellulolytic activities
Microbial dynamics and activities	Fungi (i) Rarely seen Bacteria (i) Not observed	Fungi (i) Sparsely growing mycelia (ii) Less diverse (iii) Colonization outside the cortical layers, primarily in living cells (iv) Dense colonisation close to the surface trichomes (v) Dependence on readily available food (vi) Less damage to the cell walls Bacteria (i) Less abundant	Fungi (i) Extensive and abundant (ii) Dense mycelia over the cuticle (iii) Diverse population (iv) A variety of abundant spores (v) Intense activities and interactions Bacteria (i) Abundant (ii) Diverse population (iii) Colonies over the cuticle (iv) Associated with hyphae and fungal spores (v) More pronounced activities after 20 days (vi) Highly degraded cuticle	

Another gel-retting method has been introduced with four hours of retting period, which resulted in high fibre yield [62]. It has high capability of absorbing and retaining liquid by hundreds of times of its own weight. On the contrary, gel retting uses three times lesser water as compared to water retting. Apart from the aforementioned retting techniques, there is also a well-known and traditional retting process called microbial retting [111]. Microbial retting could be conducted using bacteria or fungi to attain shorter retting time and better fibre quality. In this method, some microorganisms are capable to generate pectic-digesting enzymes that play an important role in breaking down pectic substances on fibres. Various species of *Clostridium*, *Pseudomonas*, and *Bacillus* have been identified as retting agents [112]. Ali [113] reported in his study that jute fibres were completely retted within 9 days by *Bacillus polymyxa*. On the contrary, Visi et al. [114] found that bacteria from the order of Clostridiales were the most dominant species during kenaf fibre retting. As for fungal retting, white-rot fungi, namely, *Dratronia* sp. and *Oligoporous* sp., were used in retting *Hibiscus sabdariffa* L. fibres [115]. Increased solubility of pectin was observed after fungal treatment, and it subsequently improved retting efficiency. Besides, enzyme retting which is considered as one of the most potential environmentally friendly retting methods as an alternative to the above-discussed retting methods, to produce high-quality fibres as reinforcements for composites, will be discussed in detail in the following section.

#### 4. Enzyme Retting

Enzyme retting process has been introduced for some years back as a potential substitution to the above-discussed retting methods. Dew retting process is often constrained by poor and inconsistent fibre quality as well as geographical region, which requires optimum temperature and moisture to promote microbial growth. Therefore, it is less efficient in countries with dry climate [58]. On the contrary, enzyme retting showed promising results in Europe when commercial pectinase-rich enzyme was used in the retting of the flax fibre. The retted fibre has higher yield and comparable quality to the water-retted fibre [116]. Heller et al. [42] concluded that substrate species, initial pH of the culture medium, cultivation temperature, retting time, and inoculum size are important parameters for enzyme retting. The parameters involved in microbe retting have been reviewed once previously [117]. Figure 10 shows customization of enzyme retting process. Source of enzymes, type of enzymes, and retting parameters are flexible to customise in order to obtain optimum fibre's properties for specific applications. Yu and Yu [118] recorded 85.54% and 91.31% removal of gum and pectin, respectively, on the microbe-retted kenaf fibre. They suggested that optimal retting conditions are held on 32°C with pH of 6.0 for 24 hours of cultivation time and 21 hours of retting period. However, its gum removal ability was dissatisfied when compared to about 3% of gum residual,

observed from chemical retting method [69]. Higher fibre tenacity from enzyme retting due to mild situations minimises the damage to cellulose.

Many of enzyme mixtures contain multiple enzyme activities against plant cell walls, including cellulases. Yilmaz [85] confirmed that enzyme retting process produces the finest fibre as compared with water retting and NaOH extraction. The concentration of xylanase has been observed as a significant reduction of linear density and breaking force. Besides, higher cellulose concentrations help to enhance breaking tenacity. High crystallinity fibre reported as more nonload bearing contents is being removed by celluloses [119]. On the contrary, kenaf fibres have the highest amount of lignin content among bast fibres, lignin decomposition is relatively slow, and therefore, over-retting is not critical as for other bast fibres [120]. Higher laccase enzyme may apply on kenaf enzymatic retting.

Zhao et al. [121] successfully employed *Bacillus licheniformis* HDYM-04 microbe for enzyme retting on flax. The composite enzymes consisted of 587.5 U/mL pectinase and 140.1 U/mL xylanase, which produced effective fibre degumming after 48 hours. Most importantly, incubation medium is maintained at pH 4.0–6.0, demonstrating a stable retting process. Significant reduction of gum components has been observed after 120 hours of retting, higher fibre strength, productivity, and long fibre yield rate compared to the water-retted fibre. On the contrary, Akin et al. [122] suggested presoaked flax fibre with distilled water before enzyme retting to produce finer fibre with better fibre yields yet sacrificing fibre strength. Besides, the authors found that enzyme retting was as efficient at 4 hours as 24 hours for fibre yield.

The right dosages of the response enzyme are crucial to obtain efficient enzyme retting process. The use of chelators into enzyme formulation has shown a magnificent effect on retting efficiency. Chelators are small molecules that bind very tightly to the calcium ion,  $\text{Ca}^{2+}$ . Removal of the epidermis and cuticle can be done easily since calcium is highly presented. Therefore, easier degradation of the plant cell wall was found with the appearance of chelators [123]. Among all types of chelators, ethylenediaminetetraacetic acid (EDTA) is considered the best agent in facilitating enzyme retting [124]. However, efficiency of chelators is highly affected by temperature and pH value. Most of the chelators worked on alkaline medium, but EDTA works effectively even under acid condition. Figure 11 shows the level concentration of free calcium dissolved by various chelators under different pH conditions. The lesser the detection of the  $\text{Ca}^{2+}$  ions, the more effective the chelator agent is.

Besides, there was a strong dependence of depectinization selectivity on the stem section, decreased from bottom to top presumably due to higher lignin content at the bottom stem. The thinner bast fibre layer at the top section has lower amount of wax substances and lignin, allowing easier entry of microbes and their secreted enzymes. Hence, easier depectinization resulted in over-retting and lower cellulose content and tensile strength. Furthermore, the fibre responded differently to *P. radiata* Cel 26 and *C. subvermispora*, causing a variation in mechanical properties

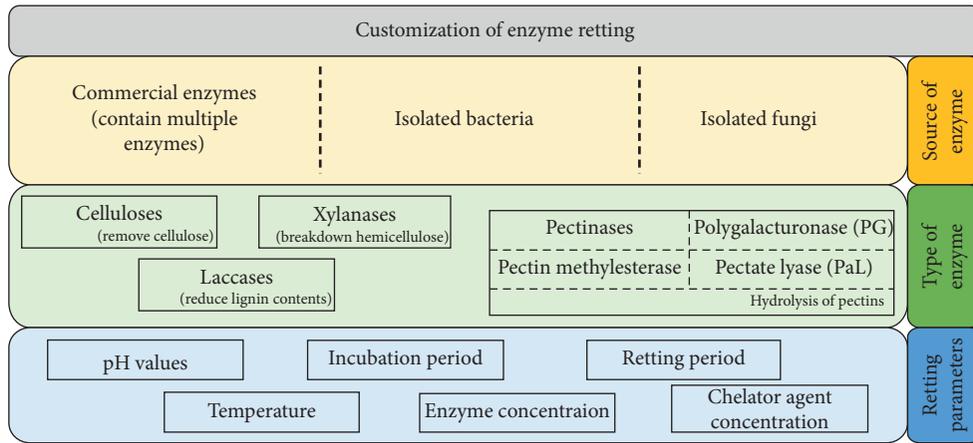


FIGURE 10: Customization of enzyme retting.

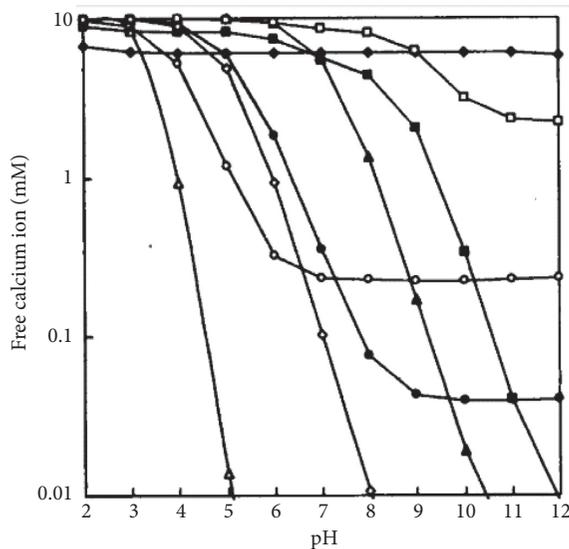


FIGURE 11: Concentration of free calcium dissolved by various chelators under different pH conditions. Symbols: sulfate (◆), nitrilotriacetic acid (◇), EDTA (△), carbonate (□), citrate (○), orthophosphate (■), diphosphate (●), and tripolyphosphate (▲) [124].

[33]. However, an observation of significant differences for properties of the untreated hemp fibre at different locations was done.

**4.1. Enzymatic Retting Mechanisms.** Enzymatic hydrolyzations can be done on every lignocellulosic fibre component by different enzymes. Cellulose, hemicellulose, lignin, and pectin components are hydrolysed by cellulases, xylanases, laccases, and pectinases, respectively. Cellulases hydrolyse  $\beta$ -1,4-glucosidic bonds in the cellulose polymer. They degrade amorphous cellulose before crystalline cellulose. Therefore, appropriate cellulase enzymes could enhance fibre crystallinity, and however, deteriorated performances were found for long retting period [125]. On the contrary, xylanases breakdown hemicellulosic

components by hydrolysing  $\beta$ -1,4-bonds in xylan chains. The most important enzymes for degrading arabinoxylan are endo-1,4- $\beta$ -xylanase and  $\beta$ -xylosidase [126]. Endo-1,4- $\beta$ -xylanase responses to the xylan backbone arbitrarily which forms xylooligosaccharides while  $\beta$ -xylosidase yields xylose by removing the terminal monosaccharide at the nonreducing end of the oligosaccharides. Another enzyme that can affect retting process of bast fibres is the laccase enzyme, which can degrade the lignin structure.

Pectinases are the most important retting enzymes to separate fibre from fibre bundles. Pectin methylesterase removes methyl groups to give access to depolymerising enzymes like polygalacturonase (PG) or pectate lyase (PaL). PG randomly hydrolyses  $\alpha$ -1,4 galactosiduronic bonds in homogalacturonans, while PaL activity resulted in eliminative cleavage to give oligosaccharides with 4-deoxy- $\alpha$ -D-galact-4-enuronosyl groups at their nonreducing ends and  $\alpha$ -D-glucuronic acid [127, 128]. Table 5 shows pectic enzyme production by numerous microorganisms and its substrate and fermentation methods (solid-state fermentation, SSF and submerged fermentation, SmF).

**4.2. Commercial Enzymes.** There are always commercialise enzymes available in the market. However, very high in cost and fixed contents of enzymatic formulations discouraged the use of commercial enzymes. Currently, not many researchers prefer to use commercial enzymes due to economical consideration. Therefore, the commercial enzyme discussion in this paper is limited to the studies that have been done for a long time.

Retting temperature is an essential parameter as enzymatic activities are sensitive to the surrounding temperature. Novozymes 249 has an optimised temperature of 60°C, while Pectinol AC and Ultrazym maximised their enzymatic activities at 45°C [130]. Two commercial enzymes Lyvelin (containing 11,000 units U/g of PG) and Peclyve (containing 375 U/g of PAL) are applied on flax-fibre retting process and compared with dew retting (6 weeks of dew retting followed by mechanical scutching) [131]. PAL-enzymatic retted flax fibre shows similar strength performance and chemical

TABLE 5: Pectic enzyme production by numerous microorganisms and its substrate and fermentation methods [129].

Microorganisms	Enzyme	Carbon sources	Fermentation methods
<i>Aspergillus spp.</i>	Pectinolytic enzymes	Wheat bran (WB) and orange-peel waste (OPW)	SSF
<i>A. niger</i>	Pectinase and cellulase	WB and OPW	SSF and SmF
<i>A. giganteus</i>	Polygalacturonase (PG)	OPW and pectin	SmF
<i>A. awamori</i>	Exo-PG and xylanase	OPW and grape pomace	SSF
<i>A. japonicus</i>	Pectinase and CMCase	OPW	SmF
<i>A. foetidus</i>	Pectinase	OPW	SmF
<i>A. fumigatus</i>	Pectinase	OPW	SSF
<i>A. sojae</i>	PG	OPW	SmF
<i>A. sojae</i>	PG	WB	SSF
<i>Penicillium oxalicum</i>	Pectinase and CMCCase	OPW	SmF
<i>P. oxalicum</i>	PAL	OPW	SSF
<i>P. viridicatum</i>	Pectin lyase and PG	OPW and WB	SSF
<i>Aspergillus</i> and <i>Penicillium</i>	PG	OPW	SSF
<i>Trichoderma sp.</i>	PG	OPW	SSF
<i>Eupenicillium javanicum</i>	Cellulase, pectinase, xylanase	Citrus processing waste	SSF
<i>Pseudozyma sp.</i>	Pectinase	OPW	SSF
<i>Bacillus licheniformis</i>	PG	Pectin	SmF
<i>Thermoascus aurantiacus</i>	PG	OPW and WB	SmF
<i>Rhizopus oryzae</i>	PAL	OPW	SSF
<i>Fusarium solani</i>	Exo-PG	OPW	SSF

compositions as the dew-retted flax fibre. This was expected since PAL specifically eliminates pectin on fibre bundles.

To maximise enzyme retting efficiency, Akin et al. [132] applied 0.05% v/v of Viscozyme L with 50 mM EDTA chelator in water at pH 5.0 under three pressure conditions (pressurized, vacuum, or atmospheric conditions) and two enzymatic application modes (enzyme applied before or after pressurized conditions being applied) [133]. The prior of enzyme application before pressurised conditions has improved enzymatic absorption in flax fibres, while no statistical differences were observed for prior pressurised and vacuum conditions before enzyme application. On the contrary, retting formulations have produced a fibre with different properties in terms of fibre fineness and strength. The effect of chelator (EDTA) and Viscozyme found influencing the fibre strength and fibre yield, respectively [132].

Evans et al. [134] used four high PG content enzymes to study the effect of PG activity for retting process. Enzymatic activities of all enzymatic solutions used have been shown in Figure 12. *A. niger* was concluded as the best retting agent among others. It produces the strongest and finest flax fibres. This finding was agreed by Zhang et al. [128], who studied the value of PG in enzymatic flax retting. The use of the chelator reported further enhanced the degree of flax retting by disrupting and weakening the middle lamella. However, the importance of the use of non-cellulolytic enzyme solution to produce strong fibres shall not be ignored [134].

**4.3. Bacteria-Isolated Enzyme.** To reduce the cost of enzyme retting process, bacteria or fungi isolation is often being done to allow secretion of suitable enzyme(s) for retting. Wang et al. [135] isolated two bacterial strains (X12 and P05) that produced promising levels of xylanase and pectinase from soil of a ramie garden. A synergic retting effect was

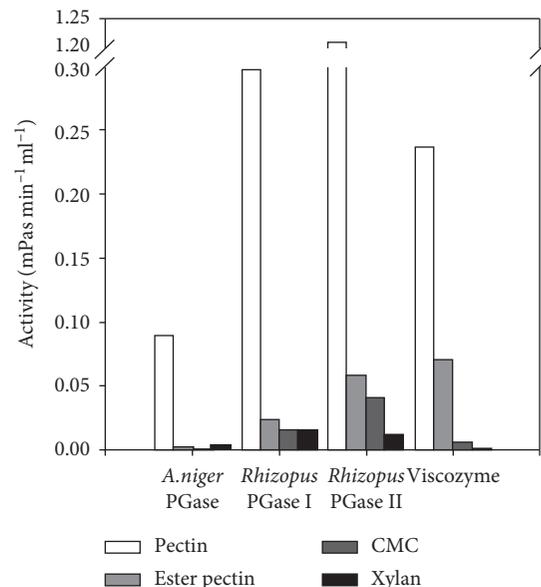


FIGURE 12: PG activities of enzymatic solutions [134].

observed on the retted bast fibre under formulation of two bacteria with equal ratio. A bioaugmentation concept was applied by the authors, showing better gum removal and breaking strength with shorter retting duration. Bioaugmentation is a practice of adding cultured microorganisms with the purpose of biodegrading specific soil and groundwater contaminants, originally. However, it works perfectly fine for bast-fibre enzyme retting process. They confirmed that the composition of two bacterial strains changes rapidly in the first 12 hours after bioaugmentation (Figure 13(a)). The amount of *Clostridium*, *Bacillus*, and Paenibacillaceae increased significantly on the 36<sup>th</sup> hour, giving the highest pectinase activities (Figure 13(b)). On the

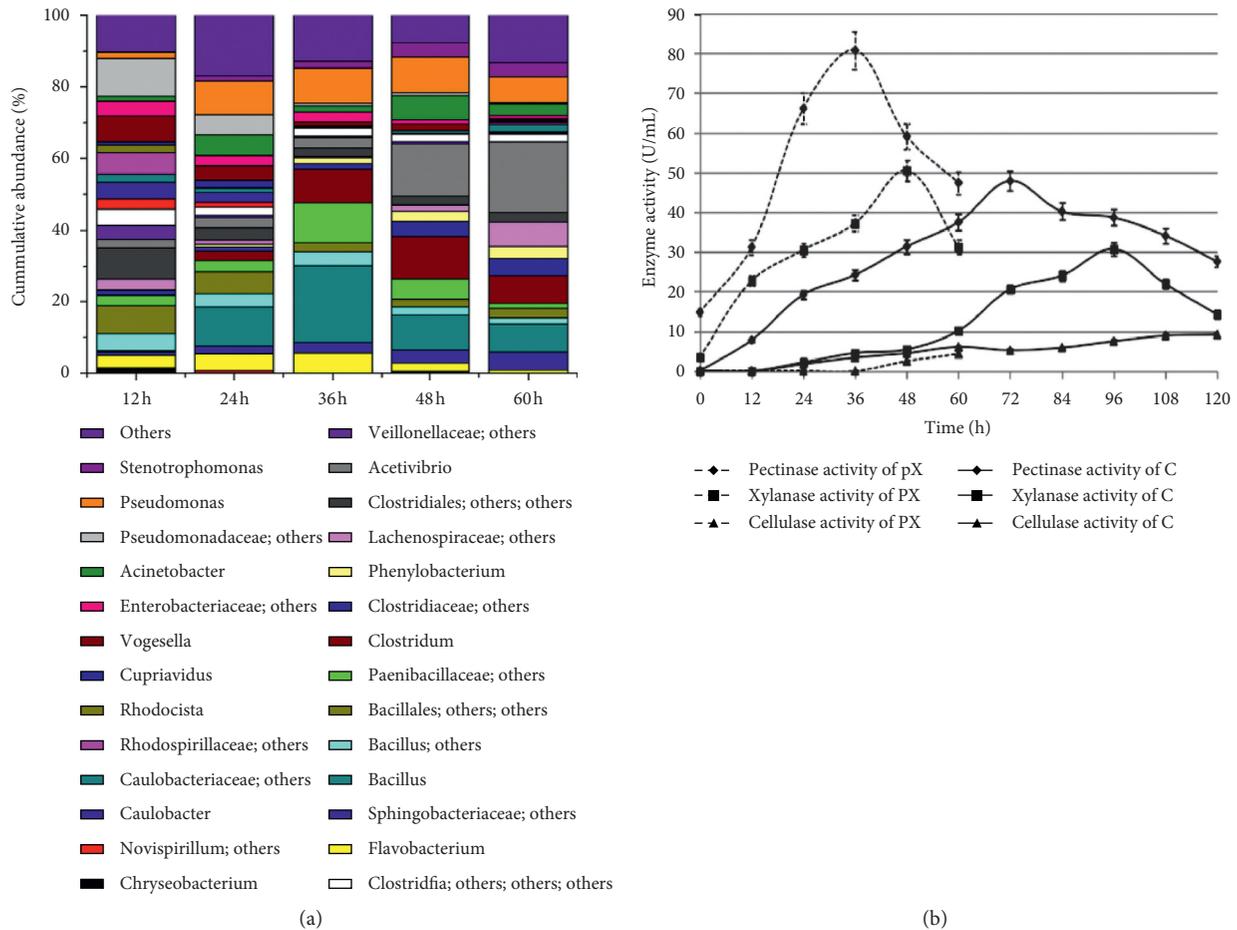


FIGURE 13: (a) The changes in bacterial composition during bioaugmentation retting throughout 60 hours and (b) enzyme activity levels (U/mL) of water retting, C and P05/X12 microbe retting, PX during bioaugmentation retting [135].

contrary, *Bacillus pumilus* strain, DKS1, was isolated from soil to study a combination of enzymatic and chemical retting process [136]. The microbial intervention followed by mild alkali treatment showed a high percentage of weight loss from retted fibres.

A thermo-alkaline PAL gene from an alkaliphilic *Bacillus clausii* strain S10 was cloned and overexpressed in *Escherichia coli* [137]. It shows the highest specific activities of 936.2 U/mg on methylated pectin at pH 10.5 and 70°C, presenting its high cleavage capability on methylated pectin. Another *Bacillus* strain, *B. cereus*, was identified in the previous study, which was able to produce the highest pectin hydrolysis activities in a selective culture medium among 153 single bacterial colonies [138]. Dramatically, reduction of sugar contents in the kenaf carbon source at 2–6 hours indicates the growth and reproduction of bacterial strains. After that, no reduction of sugar contents was recorded, giving the evidence that carbon source of reproduction for *B. cereus* bacteria are noncellulolytic components in bast fibres. The outcomes were reported close to *B. tequilensis* SV11-UV37 bacteria-retted kenaf and sunn hemp fibres, which show a good efficiency-to-cost ratio under eco-friendly manner [139].

Four bacterial strains from *Bacillus* strains with high PG, PAL, and xylanase activities with minimal cellulase tracks

were used in jute fibre retting [140]. A highlighted synergetic effect of combining microorganisms resulted in better PG (35.52–46.61 IU/g), pectin lyase (39.79–72.12 U/ml), and xylanase (0.705–0.840 μmol/ml/min) activities. As expected, they produced remarkable enhancement on fibre’s tenacity and fineness. On the contrary, contaminated postretted water increases hardness, acidic value, and chemical oxygen demand (COD).

On the contrary, comparison has been made between anaerobic strain *Clostridium* sp. L1/6 and aerobic strain *Bacillus* sp. ROO40B which are isolated from raw flax and hemp ret liquor, respectively [141]. The selection was based on the highest PG activities secreted by bacteria. One very distinct retting condition is acidity of the incubation medium. Anaerobic strains found highest PG activity (100 IU/g) at pH 4.8 medium, while aerobic strains displayed a pectinolytic activity (169 IU/g) at pH 8.0. Besides, absence of cellulose activity preserved fibre strength, but all pectinolytic anaerobic strains observed a significant amount of cellulose activities [142]. There are no significant retting differences observed between both bacteria in sugar hydrolysis process, even though aerobic bacteria were predominant in the first phase of the process, while anaerobic strains became predominant at the latter process. The reason for this is because

the growth of aerobic bacteria creates anaerobic condition that is suitable for anaerobic bacterial spore germination [141]. Another study has also confirmed that highest enzymatic activities for anaerobic strains and aerobic strains belong to *Clostridium* sp. and *Bacillus* sp., respectively, which showed a PG activity more than 100 IU/g [142]. High PG activity is well correlated with retting efficiency. Inoculation of water tanks with the highest PG activity strains reported a reduction in time required by half [143].

As the retting process via microbial isolation was significantly reducing the cost of fibre production, Fan et al. [144] designed a retting process by using *Bacillus* sp. (HG-28) for ramie fibres at commercialise scale. Detailed retting schematic for in situ microbial retting process is shown in Figure 14. An in situ microbial retting process with direct involvement of the bacterium on the ramie fibre as a carbon source, rather than treating fibre bundles with enzymes secreted by bacteria, was highlighted. This method could increase retting efficiency as well as reduce the cost. Lower gum contents on the ramie fibre and higher breaking tenacity were achieved, and additionally, consumption of chemicals, water, and energy was significantly reduced. Another rapid ramie degumming by using *Pectobacterium* sp. CXJZU-120 was conducted, and 90% of gum removal was evaluated in just 6 hours [145]. Furthermore, it could reduce production cost up to 20.5% by optimising more than 50% of resources, and this reduces 80% of pollutants as compared to traditional retting process. The study also recorded the schematic for traditional chemical and rapid enzymatic retting process to provide a repeatable methodology for further research.

Fungi always share a common substrate with bacteria, and their coexisting in many situations has given us synergistic or antagonistic interactions. In the discussion of the previous Section 3.3-Bacterial and Fungi Interaction, fungal colonisation will occur before the growth of bacteria. An inverse relationship between fungal growth and tolerance towards bacteria was observed in aquatic solutions. The fungi growth is suppressed by the presence of bacteria. However, if fungi incubations are done under a controlled environment, established growth of fungi producing higher biomass was reported [146].

**4.4. Fungi-Isolated Enzyme.** Under a controlled environment, fungi-isolated enzymes worked very well as compared to dew retting process. Maheshwari et al. [147] studied the effects of controlled colonisation environment on sunn hemp fibres. Traditional water- and dew-retting methods have been used as control sets. The results showed all fungi-retted fibres under a controlled environment have higher strength properties.

Musialak et al. [148] improved the retting process by using *Aspergillus aculeatus* (pectinases) to reduce pectin contents in tissue-cultured and field-grown plants. The fungus was contributing a significantly high retting efficiency without alteration in lignin or cellulose contents throughout the process. These have indicated that the overexpression of enzymes does not affect flax fibre

chemical compositions. The growth rate and soluble sugar and starch contents were in the range of the control levels.

Apart from this, Henriksson et al. [149] isolated seven fungi from dew-retted flax to produce enzyme filtrates that were highly effective in flax retting process. However, only *R. pusillus* enzyme with the use of chelator (oxalic acid) recorded higher retting score than the commercial enzyme (Flaxzyme). The fungus can be characterised as thermotolerant since it can be cultivated at higher temperature. Besides, the culture of *R. pusillus* did not show any significant xylanases and mannan activities, suggesting that hemicelluloses and cellulase may not be required to ret flax. Zeni et al. [150] studied PG activities from 107 microorganisms (92 newly isolated and 15 preidentified), which were collected from soil, leaves, fruits, teas, processed products, and agro-industrial wastes. Among all, there are 20 strains which are able to synthesise PG activities above 3 U/ml. Furthermore, five isolated fungi undergone 24 hours of fermentation and led to favourable PG activities. Throughout the kinetic study of PG activity, pH variation can be used to predict enzyme production as the release of galacturonic acid strongly affects acidity of the fermentation medium [151].

The enzymatic activities of *A. niger* HYA4 incubated by solid-state fermentation (SSF) in the absence or near-absence of free water have been studied previously [152]. The SSF requires lower initial capital and operating cost, producing lesser wastewater and energy needed with higher productivity. The proposed methodology has resulted in a significant reduction in retting cost. Wong et al. [153] discovered a newly isolated *A. fumigatus* R6 fungus using rice bran as a substrate in SSF condition. Optimised conditions produce 2.65-fold increments of PG enzyme activities and consequently yielding satisfying mechanical properties. They also investigated the effect between initial moisture level, temperature, and incubation time on enzyme activities (Figure 15).

## 5. Properties of Enzymatic Retted Fibres and Their Composites

One of the useful applications of bast fibres is to serve as reinforcement in composites. Bast fibres reinforced in the petroleum-based matrix could provide a certain degree of biodegradability to the composite in order to meet the current global market needs. Besides, bast fibre reinforcement could enhance strength properties of composites by regulating better load transfer mechanism. However, hydrophilic nature of bast fibres is not compatible with hydrophobic polymers, making the composite lower in performance. The main reason behind this is the poor interfacial bonding between the fibre and the matrix. Therefore, enzymatic retting process allowed complete bast fibre separation from fibre bundles and removed noncellulosic components on the fibre surface to provide higher amount of active sides for stronger interface bonding, thereby improving strength performance of bast fibre-reinforced polymer composites. Table 6

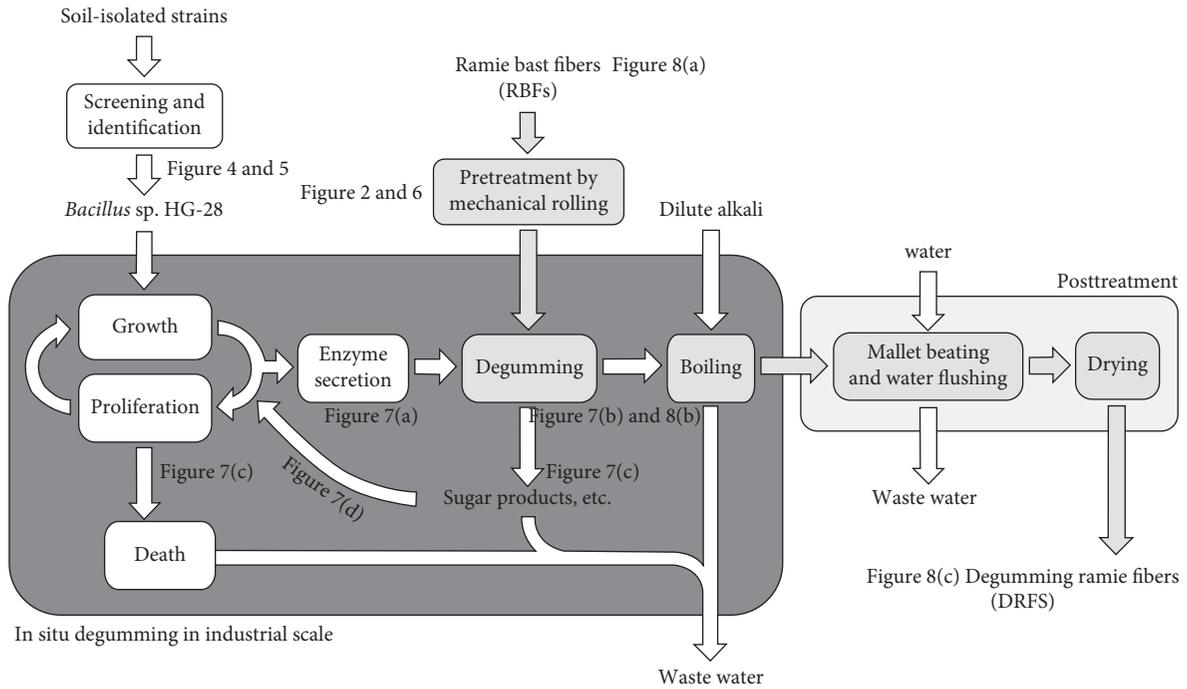


FIGURE 14: Detailed retting schematic for in situ microbial retting process [144].

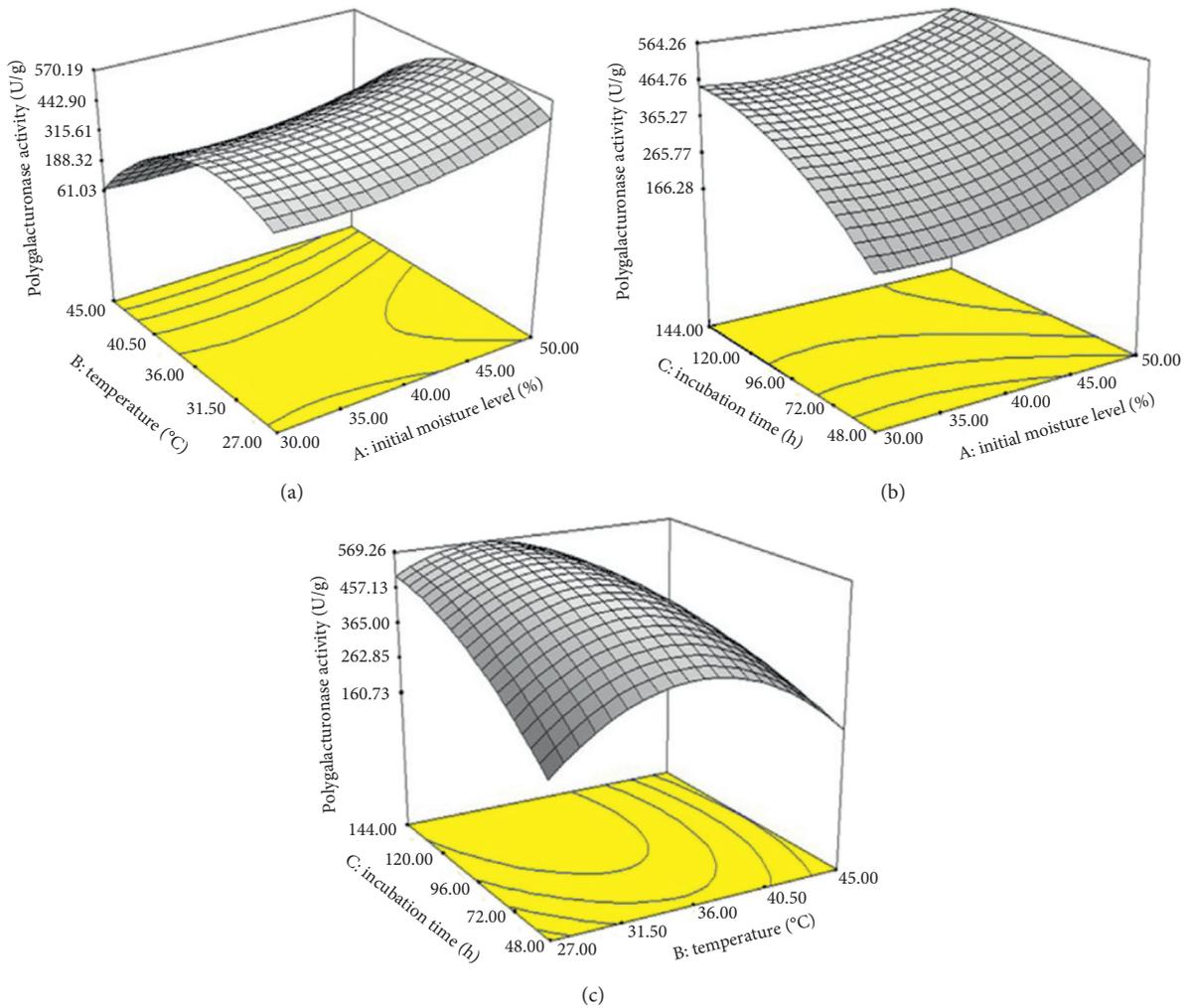


FIGURE 15: Response surface plot on the effect of (a) initial moisture level vs. temperature, (b) initial moisture level vs. incubation time, and (c) temperature vs. incubation time [153].

TABLE 6: Recent study on enzymatic-retted fibre-reinforced composites.

Year of the study	Enzymes used	Incubation conditions/retting conditions	Target fibres	Matrix	Composite strength (MPa)	Reference
2019	Ethylenediaminetetraacetic acid	Treated with aqueous solution of pectinase (1%) containing ethylenediaminetetraacetic acid at 50°C for 24 h	Kenaf	Starch	<sup>a</sup>	[154]
2019	Pectate lyase, pectin methylesterase, PG, hemicellulases, and xylanase	25 mM EDTA, pH 6.5, 40°C, 24 h	Flax	Epoxy	—	[155]
2018	—	Approximately 30 kg of hemp fibres were treated at 39°C–42°C for 24–30 h depending on the conditions. pH of the solution was 4.1–4.5 throughout the process. The treated fibres were rinsed with water until water dripping from the fibres reached a pH of at least 6.0	Hemp	Epoxy	46	[156]
2017	<i>P. radiata</i> Cel 26 and pectinase	Retting was conducted for 7, 14, and 20 days in 1 L Erlenmeyer flasks at 28 C	Hemp	Epoxy	280–300	[101]
2016	Endo-polygalacturonase (EC 3.2.1.15) and pectin lyase (EC 4.2.2.10)	Hydrothermal pretreatment with three different water vapour pressures at 40°C followed by 0, 30, 90, 150, 240, and 300 min at a pH of 6.0 using 25 mmol dm <sup>3</sup> citrate buffer, a temperature of 40°C, and an agitation of 100 rpm	Hemp	Epoxy	325	[78]
2008	SIHA-Panzym® DF (EC 3.2.1.15, Novozyme A/S, Bagsvaerd, Denmark)	1 M sodium hydrogen carbonate, 1 M citric acid, 10 mM EDTA at 35°C and pH 4.5	Hemp	Polypropylene, PP	34.0–47.6	[157]
2014	Cellobiohydrolases and endoglucanases	50 mM Na-citrate buffer, pH 5.0, with a fibre consistency of 4%. The treatment time was two hours, and the temperature was 45°C	Flax	PLA	—	[158]
2012	Pectinase, laccase, cellulase, and xylanase	Jute fabrics were treated with 2 wt% nonionic detergent solution at 70°C for 1 h before enzymatic treatment. The liquid ratio was 1 : 40 for all the treatments. Enzymatic treatments were performed with various enzyme solutions for 90 and 180 min	Jute	Polyester	35.86–50.19	[159, 160]
2012	Cellulase	A bath ratio of 20 : 1, pH 4.8, and 50°C in acetic acid and sodium acetate buffer solution	Flax	PLA	<sup>a</sup>	[161]
2011	Bacterial pectinolytic enzyme with lyase activity	HCl 50 mM with Ca buffer, pH 8.5, 42°C, and 50 rpm in lab-scale reactors for 10–46 hours	Flax	Vinyl ester	48.54–71.46	[162]
2009	White-rot fungi <i>Phanerochaete sordida</i> (D2B), <i>Pycnoporus</i> species (Pyc), and <i>Schizophyllum commune</i> (S.com)	Dried nonretted hemp fibres were sterilised using gamma radiation of 26.0 kGy (kilogray) in sealed sterilisation bags. Irradiated hemp fibres were then inoculated with white-rot fungi (D2B, Pyc, and S.com) for 2 weeks. Water was added for all fungal treatments and bag retting to give a moisture content of 60 wt.%	Hemp	PP	37.54–45.33	[163]

<sup>a</sup>Composite strength values are provided in previous papers as cited, but the values are unable to be compared here.

shows a recent study on enzyme-retted fibre-reinforced composites. Kenaf bast fibres were retted under pectinase with chelators [154]. The retted fibres were then used in

composite fibre reinforcements. All mechanical properties show that chemical retting provides better enhancement to the fibres. However, enzyme-retted fibre composites

showed higher biodegradability. This is because the enzyme acts as a catalyst to boost up microorganic activities in the soil.

Optimum enzyme retting formulation was reported to improve the quality of fibre, thereby the performance of composites. Smoother fibre surface and better separation from fibre bundles are the key points to have better strength of enzyme-retted bast fibre-reinforced polymer composites. A study of the effectiveness of controlled water retting, enzyme retting with and without EDTA chelator, was performed on flax fibre-reinforced epoxy composites [164]. The untreated flax fibres were, on average, 30–35% stronger than retted flax, while no obvious difference in the strength properties among different retting methods. However, reinforcement of untreated flax fibres in the epoxy matrix showed the lowest tensile value due to that most of the reinforcement fibres are presented in bundle forms which have the brittle manner and significant amounts of debris. Thus, affecting degree of adhesion and poor interfacial bonding between the matrix and the fibre were found. Besides, fibre-bundled reinforcements would restrict effective surface area by covering the fibre surfaces and hindering active bonding between the fibre and the matrix [165].

On the contrary, EDTA-treated flax fibre-reinforced epoxy composite shows the highest tensile value because the chelator contains  $\text{Ca}^{2+}$  ions that allow the fibres to separate from the epidermis easily, resulting in clearer and smoother fibres. Over-retting was suspected for enzyme retting process with EDTA since relatively poorer bonding between the matrix and the fibre was observed. Excellent fibre separation with ethylenediamine tetramethylene phosphonic acid (EDTMPA) has been reported [166]. Relative environmental, economic, and strength performances have been listed in Table 7 for the chelator, combined chelator, and enzyme-treated hemp-reinforced polypropylene composite.

The pectinolytic enzyme-retted hemp bast fibre strips were aligned and untangled to process the fibres unidirectionally in epoxy composites followed by vacuum degassing. A 100 kPa hydrothermal pretreatment before enzymatic retting produces the highest fibre ultimate tensile strength [78]. This is because macropores observed on cell walls have been prevented during pretreatment, hence reducing probability of over-retting which is because of penetration of enzymes. However, some researchers commented that individual pectinase retting showed lack of fibre separation. Neither laccase nor pectinase was able to breakdown the waxy layer and remove lignin and pectin to release fibres from fibre bundles [165]. On the contrary, effective removal of pectin by enzymes has been reported with evidence of superior mechanical properties of hemp fibre-reinforced epoxy composites. An increment of 31% and 41% of tensile strength and stiffness was recorded, respectively [78]. Similar results were observed on enzyme-retted flax fibre-reinforced epoxy composites [155, 167]. Besides, SEM micrographic shows the lowest porosity factor due to parenchyma cell elimination on the fibre surface, indicating good impregnation of the hemp fibres by the epoxy matrix. There was no strong correlation being observed between pectinase

retting process with composite's impact strength and elongation at break off [157].

High effectiveness of enzyme retting to remove fibre's impurities has been confirmed in previous work [57]. Surface chemical modifications have been induced by enzyme retting which was evident by ATR-FTIR spectroscopy. Different densities of spectrum peaks have been recorded in Figure 16 when comparing between raw and retted hemp fibres. There is disappearance of  $2850\text{ cm}^{-1}$  peak responsible for  $-\text{CH}$  symmetrical stretching of polysaccharides, for waxes and oils, respectively. The absence of  $1640$ ,  $1550$ – $1400$ , and  $1244\text{ cm}^{-1}$  peaks observed in retted fibres was responsible for effective removal of carboxylate ions for pectin and hemicellulose and aromatic ring in lignin by enzyme hydrolysis. Besides, a higher amount of cellulose and hemicellulose contents for retted fibres can be viewed by the sharper peak of  $3300\text{ cm}^{-1}$ . Apart from this, synchronised information from XRD spectra shows a higher crystallinity index for retted hemp fibres. When these retted fibres were inserted into the PBS matrix, better tensile and flexural strength values were observed, regardless of any fibre volume contents in the composites (Table 8).

Besides, enzyme-retted kenaf fibres and modified starch biocomposites were fabricated with additional plasticisers for the purpose of starch fluidity [154]. The authors claimed that PVA plasticiser is most compatible with enzyme-retted kenaf fibre-reinforced starch composites. At the same time, this formulation is given highest biodegradability since the enzyme acts as a catalyst for microorganic activity in the soil, accelerating biodegradation rate. Two bacterial strains of *Clostridium felsineum* L. have been selected for ramie microbe retting process in a previous study [63]. The results showed chemical retting is more effective than both microbial retting processes from the biochemical contents of retted ramie fibres. However, both microbe-retted ramie fibre-reinforced PHA composites found better mechanical properties than the chemical-retted fibre composite, suggesting higher suitability of microbial retting process for composite applications.

To enhance composite performances, interfacial bonding between the matrix and fibres is an important key factor. Three white-root fungi have been applied to investigate strength of the hemp fibre-reinforced polypropylene composite affected by the condition of interfacial bonding [163]. White-rot fungi reported to have high degradation rate of noncellulosic components and produce microholes, which can roughen the surface of hemp fibres [168]. FTIR spectra observed a reduction of the  $1736\text{ cm}^{-1}$  peak for white-rot fungi-retted hemp fibres, which can be attributed to effective pectin and wax removal, resulting in high crystallinity index of retted hemp fibres (84–88%). Although over-retted process was being blamed for the fine holes on the roughened fibre surface, it resulted in deteriorated tensile strength [163]. The previous study found similar findings, in which enzyme-treated flax fibres were pulled out from the matrix, which were more intensely coated with the PLA matrix [158]. Improved adhesion between PLA and the enzyme-retted fibre is shown in Figure 17(a) due to different surface morphologies of untreated (Figure 17(b)) and

TABLE 7: Relative environmental, economic, and strength performance for the chelator, combined chelator, and enzyme-treated hemp-reinforced polypropylene composite [165].

Treatment	Environmental impact	Treatment cost				Composite strength (MPa)
		Chemical	Energy	Equipment	Processing	
EDTMP treatment, 5 g/l	Low	Lowest	Lowest	Lowest	Lowest	41.55
EDTMP treatment, 10 g/l	Low	Low	Lowest	Lowest	Lowest	42.3
EDTMP treatment, 5 g/l followed by pectinase retting	Low	High	High	High	High	41.73
EDTMP treatment, 5 g/l followed by laccase retting	Low	High	High	High	High	41.41

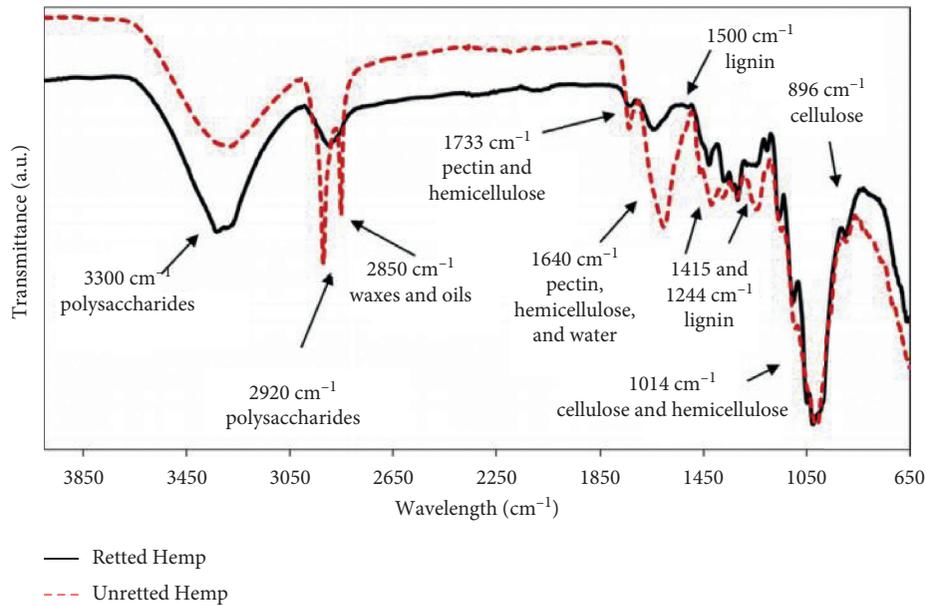


FIGURE 16: ATR-FTIR spectroscopy for enzymatic retted and unretted hemp fibres [57].

TABLE 8: Tensile and flexural properties of hemp fibre-reinforced PBS composites [57].

Sample	Tensile properties			Flexural properties		
	$\sigma_{\max}$ (MPa)	$\varepsilon_b$ (%)	$E$ (MPa)	$\sigma_{\max}$ (MPa)	$\varepsilon_b$ (%)	$E$ (MPa)
PBS	$17 \pm 1$	$3.8 \pm 0.3$	$599 \pm 25$	$11 \pm 1$	$1.4 \pm 0.2$	$854 \pm 150$
10UR	$23 \pm 3$	$2.4 \pm 0.3$	$1094 \pm 200$	$27 \pm 0$	$2.8 \pm 0.3$	$1500 \pm 132$
10R	$25 \pm 4$	$2.8 \pm 0.5$	$968 \pm 180$	$26 \pm 2$	$2.6 \pm 0.1$	$1433 \pm 196$
20UR	$29 \pm 3$	$1.9 \pm 0.2$	$2221 \pm 210$	$31 \pm 2$	$2.7 \pm 0.1$	$1884 \pm 150$
20R	$32 \pm 3$	$2.5 \pm 0.3$	$2414 \pm 241$	$40 \pm 5$	$2.8 \pm 0.8$	$2523 \pm 165$
30UR	$22 \pm 4$	$1.7 \pm 0.2$	$2232 \pm 255$	$32 \pm 5$	$2.4 \pm 0.3$	$2045 \pm 175$
30R	$28 \pm 3$	$2.0 \pm 0.4$	$2295 \pm 250$	$35 \pm 3$	$2.4 \pm 1.0$	$2259 \pm 170$

enzyme-retted fibres (Figure 17(c)). Figure 17(b) shows waxy materials covering the fibres and hence hindering effective adhesion, while Figure 17(c) reveals a smooth flax fibre surface which promoted good interfacial bonding. On the contrary, Foulk et al. [162] claimed that smoother fibre surface by long enzyme-retting periods found reduced mechanical interlocking between the flax fibre and thermoset vinyl ester resin [162]. Apart from this, strong interfacial bonding has a positive effect on shrinkage- and fire-

resistant properties of enzyme-retted bast fibre-reinforced polymer composites [161].

Liu et al. [22] confirmed that 50 wt% of laccase-retted hemp fibre-reinforced epoxy composite had 33% and 56% increment of stiffness and strength, respectively. Besides, higher thermal stability of retted hemp fibres was observed due to oxidation of lignin components by the laccase enzyme. However, flax fibres are generally having more hemicellulose than hemp fibres. Removal of

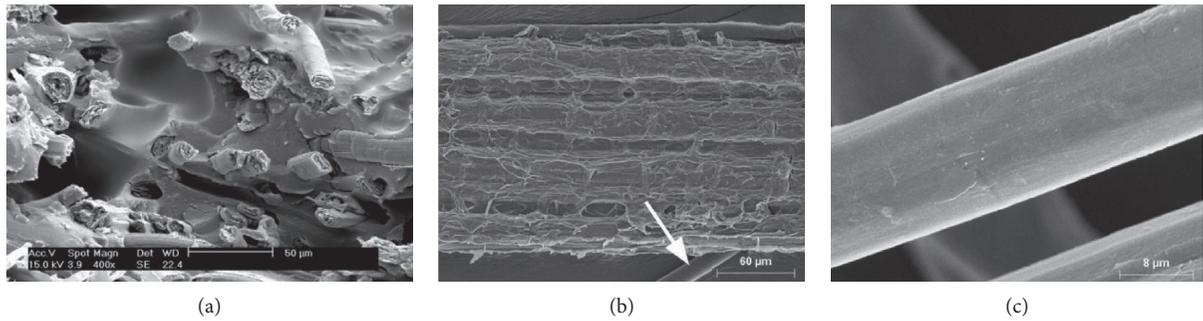


FIGURE 17: (a) Improved adhesion between PLA and the enzyme-retted fibre due to different surface morphologies of (b) untreated and (c) enzyme-retted fibres [158].

TABLE 9: Maximum decomposition temperature for different systems [171].

System	Maximum decomposition temperature (°C)	
	Enzyme	NaOH + enzyme
Control	354 ± 0.52	350 ± 2.34
Xylanase	346 ± 1.04	360 ± 1.24
Xylanase + cellulase	351 ± 0.31	364 ± 1.03
PG	365 ± 0.50	367 ± 1.39
Pectin methylesterase	354 ± 0.53	362 ± 1.03
Laccase	348 ± 1.24	364 ± 1.35

noncellulosic components resulted in a weaker structure since lignin acts as an adhesive holding the fibril network together [126]. On the contrary, ineffective retting by the laccase enzyme alone was reported in the previous study [169]. This is because most of lignin components in the hemp fibre were confined at inner regions of the primary cell wall and lumen. Hybrid treatment (alkaline treatment with laccase retting) is a solution to increase fibre adhesion force, surface polarity, and smoother surface [169, 170]. George et al. [171] proved that mercerisation before enzymatic fibre retting did enhance fibre thermal stability, regardless of the type of enzymes (Table 9). Alkaline treatment helps in better enzyme penetration to hydrolyse low thermal stability of noncellulosic components. Nevertheless, the thermal stability of the matrix plays a dominant role in thermal stability properties of biocomposites. The insertion of reinforcement bast fibres with or without retting process into highly thermal stability matrix will only observe slight differences in thermal stability [57].

## 6. Conclusion

As more and more innovative research is being conducted on natural bast fibre-reinforced composites in advanced sectors, bast fibres with high performance must be achieved. Bast fibre retting process is the first and the most important process for obtaining promising strength. Water retting process used to be the most recommended retting process for quality bast fibre production. Nevertheless, generation of

large amounts of wastewater has it prohibited by most countries. Chemical and dew retting was then applied to substitute water retting process. However, high chemical cost and low retted fibre quality, of chemical and dew retting process, respectively, have driven people to look for another suitable process.

Enzyme retting is claimed to have a more environmentally friendly process by reducing wastewater products, shorter retting period, and controllable fibre biochemical components under mild incubation conditions. The right dosages of the response enzyme are crucial to obtain an efficient retting process. On the contrary, higher enzymatic retting efficiency could be achieved by addition of chelators containing a large amount of  $\text{Ca}^{2+}$  ions that can remove the epidermis and cuticle easily to release the bast fibre from fibre bundles. EDTA has been reported as the best chelator agent.

Commercial enzymes are not preferred by academics and fibre manufacturers, due to fixed enzyme ratio and high cost. Fungi always share a common substrate with bacteria, and their coexisting in many situations has given us synergistic or antagonistic interactions. However, an inverse relationship between fungal growth and tolerance towards bacteria was observed in aquatic solutions. The fungi growth is suppressed by the presence of bacteria. Established fungi medium producing higher biomass was reported. Therefore, the presence of bacteria and/or fungi shows difference in retted bast fibre's properties. Pectinase is the main enzyme for retting process, hydrolysing pectin components in the middle lamella to release the single fibre. However, celluloses, xylanases, and laccases can be applied depending on the type of bast fibre or the applications.

To fabricate a promising material for advanced sector applications, enzyme-retted bast fibre-reinforced polymer composite was found to meet the criteria often. High effectiveness of enzymatic retting on impurity removal produces a high cellulosic fibre which has high strength and crystallinity. Besides, roughening the surface of the microbial retted bast fibre has improved interfacial bonding with the matrix, thereby increasing strength performance and positive effects on product shrinkage- and thermal-resistant properties. Retting process, especially enzyme retting, could offer a tremendous benefit to bast fibres as green composite

reinforcements and, at the same time, increases the value of nonfood crops by optimising its potential as advanced materials.

## Conflicts of Interest

The authors declare that they have no conflicts of interest.

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