

Review

Targeted Pre-Treatment of Hemp Fibers and the Effect on Mechanical Properties of Polymer Composites

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Abstract: Research on plant-fiber-reinforced composites has gained significant research interest since it generates composites with exceptional mechanical properties; however, the potential of hemp fibers can only be fully exploited if the fibers are well separated from the bundle to achieve cellulose-rich fibers. This is because well-separated bast fibers that are long and exhibit higher fiber aspect ratio enhance the mechanical properties of the composite by influencing property translations upon loading. A key feature for successful implementation of natural fibers is to selectively remove non-cellulosic components of hemp fiber to yield cellulose-rich fibers with minimal defects. Targeted pre-treatment techniques have been commonly used to address the aforementioned concerns by optimizing properties on the fiber's surface. This in turn improves interfacial bonding between the fibers and the hydrophobic polymer, enhances the robustness of hemp fibers by improving their thermal stability and increases resistance to microbial degradation. In this study, we comprehensively review the targeted pre-treatment techniques of hemp fiber and the effect of hemp fiber as a reinforcement on the mechanical properties of polymeric composites.

Keywords: hemp; fibers; pre-treatment; polymer; composites; properties



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1. Introduction

Natural fibers such as hemp fibers are highly advantageous for use as reinforcement in composite materials, owing to their low density (1.248 g cm^{-3}) and biodegradability [1–3]. Additionally, they offer high specific modulus and a strength of 20–41 GPa and 210–750 MPa, respectively, with approximately 70% lower cost than synthetic glass fiber [4,5]. Owing to the excellent properties of hemp fiber, significant research interest in the last decade was emphasized on hemp-fiber-reinforced composites [6,7]. For instance, 4676 articles on hemp fibers during the period 2013–2022 were retrieved for the syntax string of <hemp fiber composites> via Scopus search tool. In particular, publication is seen to have increased from 175 to 651 from the year 2013 to 2022, with most of the publications focused on hemp-fiber-reinforced polymer composites (Figure 1).

Generally, the primary and secondary single (bast) fibers present in the cortex is the most useful for use as reinforcement in composite materials (Figure 2). As such, the polysaccharides and lignin that are present between bast fibers should be selectively removed to attain individual fibers. The complex interaction between cellulose, lignin, hemicellulose and pectin imposes great challenges in utilizing the lignocellulosic material [8,9]. To overcome this limitation, several pre-treatment techniques have been employed, in which the main goal of such pre-treatment techniques is to disintegrate the non-cellulosic

material and separate individual fibers from the bulk (Figure 3) without losing the desired carbohydrates and without the formation of unwanted inhibitors [10,11]. The targeted pre-treatment can be generally classified by defibrillation or surface modification techniques. For instance, cellulose-rich crops, such as hemp, consist of high amounts of pectic substance (in particular, at the middle lamella which hinder accessibility of lignocellulolytic enzymes to cellulose). Pre-treatment techniques, such as enzymatic pectin removal in traditional retting processes, enables disintegration of fiber bundles, which eases further fiber processing for use as reinforcing filler in composite materials [12,13].

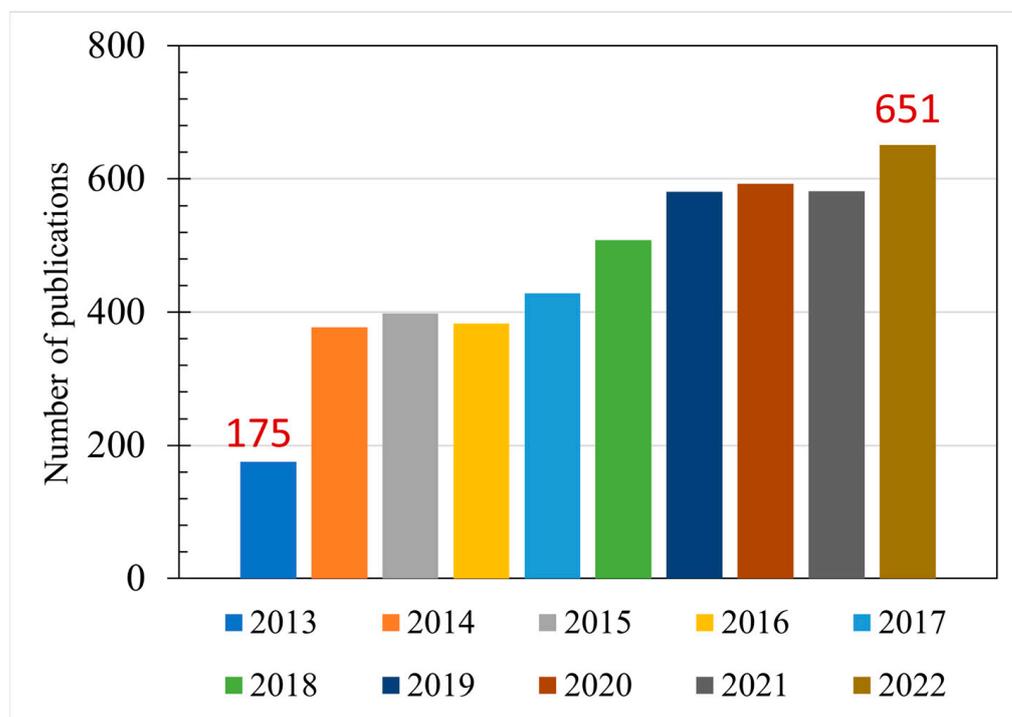


Figure 1. Number of peer-reviewed publications from the year 2013 to 2022 on hemp-fiber-reinforced composites.

Recently, targeted pre-treatment of hemp fibers has been widely investigated to increase the surface hydrophobicity and moisture resistance of hemp fibers. This is due to the ability of hemp fiber to absorb water and, consequently, deteriorate properties of the composite material while aging [14–16]. This, in turn, limits the exploitation of hemp-fiber as reinforcing filler in composite materials. To overcome this limitation, chemical treatment, such as grafting organic molecules, reduces hydroxyl groups on the surface of the fibers, thereby reducing the water absorption capability of fibers [17–19]. Similarly, recent studies have reported that targeted pre-treatment techniques are capable of enhancing the robustness of hemp fibers by improving their thermal stability and resistance to microbial degradation [20–22]. Whilst much of the emphasis has been focused on various natural-fiber-reinforced composites, comprehensive reviews on targeted pre-treatment of hemp fibers and the effect of hemp fiber as reinforcement in polymer composites are still limited. As such, the authors make an attempt to gather information from the earliest to the most recent developments regarding the pre-treatment of hemp fibers for the removal of non-cellulosic material from hemp fiber and their effect on the mechanical properties of polymer composites. To this end, Section 2 of this review discusses general characteristics, cultivation and pre-treatment of hemp fiber; Section 3 reviews the effects of pre-treatment and fiber alignment on the mechanical properties of hemp-fiber-reinforced polymer composites; while Section 4 outlines the application perspectives of hemp-fiber-reinforced polymer composites.

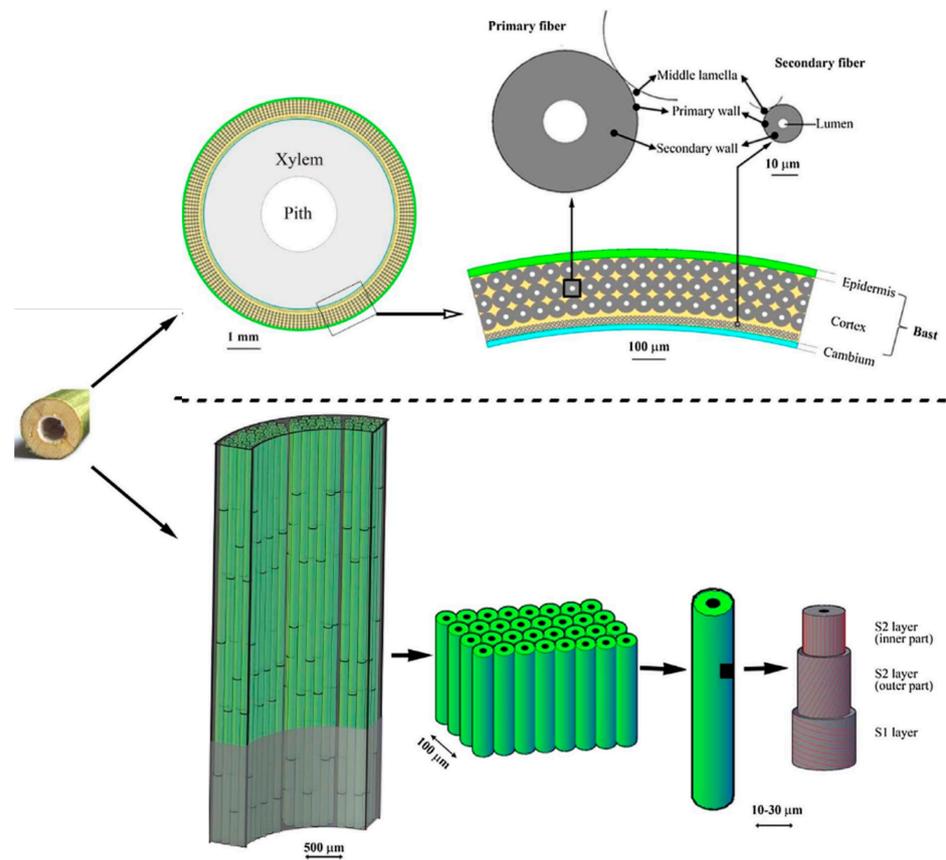


Figure 2. Schematic diagram hemp stem illustrating bast strip and single fiber at different scale levels; reproduced with permission from Elsevier 2017 [23].

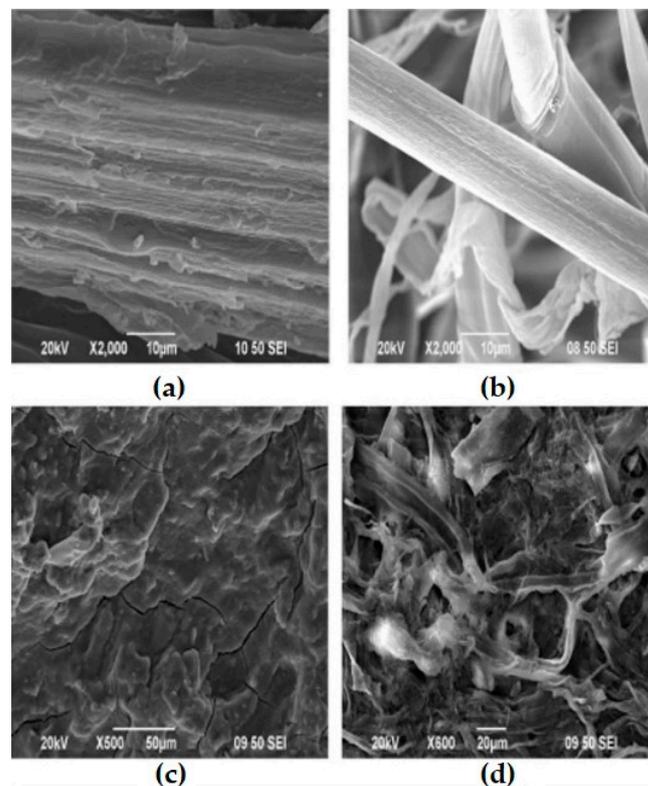


Figure 3. SEM image of: (a) untreated fiber, (b) cellulose microfibril, (c) hemicellulose, (d) lignin; reproduced with permission from Elsevier 2013 [24].

2. Processing and Targeted Pre-Treatment

2.1. General Characteristics and Cultivation Hemp Fiber

Hibiscus sativa (hemp plant), which belongs to the “Cannabaceae” family, is a tall and thin plant that exhibits a vigorous growth rate that can be utilized to produce various products. Hemp can be blended with cotton, wool and silk, and it can be spun to provide desirable characteristics, such as high temperature, heat resistance, and antistatic properties. In earlier days, hemp fiber has been explored in military applications for fabricating special uniform or civil textiles that are waterproof, windproof, insect resistant, sunscreen, antibacterial, strong wear resistance and anti-infrared flame retardant [25]. Most studies have reported that hemp fibers are composed of 60–70% cellulose, 15–20% hemicelluloses, 2–4% lignin, 2–4% pectin and 1–2% fat and wax [26]. In earlier studies, hemp fiber was blended with cotton, wool and silk to be spun. Table 1 summarizes the chemical composition of hemp fibers from various cultivars. The development of the hemp plant is highly reliant on growing conditions, such as type of soil or climate conditions. For instance, cannabis crops are most productive when grown at an average daily temperature of 14°, although they can be grown at temperatures ranging from 5.6 to 27.5° [27]. On the other hand, while hemp is able to grow in various soil types, it flourishes when grown in soil with varying texture or structure. For example, loamy soils with high level of organic matter are considered the best type of soil to maximize the cultivation of hemp [28]. At temperatures below 4.4 °C (frost) the hemp plant loses its ability to thrive. Moreover, harvesting of hemp can only occur after the staminate plants have finished flowering but before the seed has ripened, which is the stage known as “technical maturity”, where the crop is grown into long fibers. Technical maturity differs when the crop is grown for long fibers in comparison to when the crop is grown for seed. It is necessary to ret the stems after the chopping process, upon which they are laid down on the ground. This technique ensures easier recovery of long fibers, as it involves cutting of plants rather than pulling, similar to the harvesting of flax for textile production [29].

Table 1. Chemical composition of hemp fibers from various cultivars.

Cultivar	Cellulose [wt.%]	Hemicellulose [wt.%]	Pectin [wt.%]	Lignin [wt.%]	Ref.
USO 31	78.4–81.7	5.7–6.4	n/a	10.0–13.0	[30]
Fedora	55.0	16.0	8.0	4.0	[31]
Fedora 17	65.6–84.9	6.0–8.1	9.4–25.0	2.7–4.5	[32]
Fedora 19	58.6	9.3	n/a	5.0	[33]
Felina 34	57.1–61.8	8.3–14.3	2.8–8.6	1.2–7.3	[34]
Fibrimon 56	53.2	6.9	n/a	5.0	[33]
Kompolti Sargaszarú	68.2–69.2	6.7–8.5	n/a	3.5–5.5	[33]
Kompolti Hybrid TC	60.2–74.3	7.1–7.9	n/a	3.3–4.4	[33]

2.2. Pre-Treatment of Hemp Fiber

Studies have reported various techniques for the treatment of hemp fibers, which resulted in excellent adhesion and interfacial bonding with the host matrix. The pre-treatment of hemp fiber can be generally classified into three major categories as summarized below:

1. Defibrillation (traditional retting, controlled microbiological retting, mechanical treatment, steam explosion, chemical treatment and enzyme treatment);
2. Surface modification (alkali treatment, graft co-polymerization, physical treatment and esterification);
3. Antimicrobial degradation.

Most of the aforementioned pre-treatment techniques are vital to increase the cellulosic content in the fibers, which will be achieved from degrading and dissolving non-cell-wall material (pectin, waxes), lignin and, to a lesser extent, hemicelluloses. For example, the pre-treatment of fibers with retted fibers can increase the production of cellulose content up

to 90%, which is highly desirable for use as reinforcement in composite materials [35]. The following subsections will review the pre-treatment of the aforementioned hemp fibers.

2.2.1. Defibrillation

Most pre-treatment of hemp fibers results in removal of the non-cellulosic material and separation of fibers from the bundle. This leads to modified hemp fibers that are finer, with lower lignin content, higher flexibility and mechanical properties in comparison to untreated fibers. In a traditional retting process, microorganisms are used to remove and separate natural fibers from the stem in order to attain cellulose-rich fiber. Two common types of retting include field retting and water retting. Field retting involves casual attacking via microbial activities, where the growth of microbial species, such as pectinolytic microbial community, will depend on the moisture content of the plant stem, humidity, temperature and weather conditions. On the other hand, water retting involves submerging the plant stems in bodies of water, such as rivers, lakes or tanks, to induce the development of pectinolytic microbes. By submerging into bodies of water, water penetrates into plant stem structures to increase moisture absorption, thereby boosting proliferation of microorganisms. It has been reported that water-retted fibers have higher cellulose and hemicellulose contents (81.7 wt.% and 6.3 wt.%, respectively) and lower lignin content (10.2 wt.%) in comparison to field retted fibers with 78.4 wt.%, 5.9 wt.% and 13.1 wt.%, respectively [36]. In another study, it was reported that traditional water retting process modified by inoculation with pectinolytic bacteria (anaerobic strain *Clostridium* sp. L1/6 and aerobic strain *Bacillus* sp. ROO40b) reduced the duration of the water retting process while improving the quality of the fiber significantly [37]. High quality with 89 wt.% cellulose content was reported within only 3–4 days of retting. Thygesen et al. reported that water retting of hemp generally results in higher retting efficiency and stronger fibers in comparison to field retting, since water retting provides suitable microbial flora with a good combination of enzymes and low cellulolytic activity [38].

Unlike traditional field or water retting, where the process is not well controlled (depending on the spontaneous proliferation of microbes contained in specific geographic/ weather conditions), controlled microbiological retting attacks non-cellulosic components of the hemp fiber via a controlled incubation process with selected microorganisms. The microorganisms' secret enzymes, such as pectinolytic enzymes attack the non-cellulosic component of hemp fiber. For example, controlled retting with *P. radiata* Cel 26 resulted in 78 wt.% cellulose content, which was higher than water-treated hemp fiber, which resulted in 74 wt.% cellulose content [39]. In another study by Li et al., it was reported that 2 weeks of fungal retting (white rot fungi *Schizophyllum commune*) enhanced the tensile strength and stiffness of hemp fiber by 28.5% and 75%, respectively [40]. Chelators and fungal treatments have been reported to be advantageous for composites reinforced with long hemp fiber. For example, treating a polypropylene matrix with white rot fungi and the use of coupling agents, such as PP and MAPP, improves the interfacial adhesion between the polypropylene matrix and the chelator, which, in turn, increases the mechanical properties of the composite [40]. Nevertheless, it is noteworthy that the quality of fibers produced from controlled microbiological retting is highly dependent on factors such as cellulolytic enzyme activity and efficiency of depectinization, i.e., microorganisms with high pectinolytic enzyme activity and zero cellulolytic activity yields high quality fibers. A significant issue in the use of natural fibers as reinforcement in polymer matrix is their moisture absorption, which impacts the mechanical properties of the composite. This is because moisture absorption leads to delamination and defects of the interfaces. To overcome such issues, enzymatic treatment has been used to minimize moisture absorption of hemp fibers. The idea is to remove components of hemp fiber, such as pectin and hemicellulose, to increase the moisture resistance of the fibers by using enzymes, such as xylanase, polygalacturonase, laccase or a combination of these enzymes. In a study by George et al., it was reported that a combination of xylanase and cellulase enzymes significantly reduced moisture uptake of PP composites reinforced with hemp fiber to less

than 0.6 wt.% when immersed in water for 4 weeks [41]. Similarly, in another study by Li et al., it was reported that PP–hemp composites pre-treated with pectinase resulted in reduced water absorption from 31.4% without treatment to 20.5% after enzyme treatment for 90 min [42].

Mechanical treatment is a pre-treatment process after retting to separate the outer fibrous layer from the inner layer. This process of separating hemp stems is known as decortication, in which mechanical forces will be applied to detach bonds between the outer fiber and inner core. The decortication process is usually accomplished via mechanical devices such as hammer mills, crushing rollers or cutterheads, which produces shear, compressive or impact force to break the bonds between the layers of fiber. Baker et al. [43] showed that a planetary ball mill was able to produce up to 27.5% higher fiber yield compared to hammer mill. In terms of mechanical properties, it has been reported that composites reinforced with hemp fibers from pinning decortication exhibited stiffness and tensile strengths of 36.4% and 70% or higher, respectively, compared to composites reinforced with fibers produced from standard decortication processes (Figure 4) [44]. After hand combing, the pinned decortication process results in closely aligned fibers, whereas the standard decortication process results in large bundles that are well aligned and smaller bundles that are tangled into a random alignment. As such, fiber alignment increases load-transfer efficiency, thereby enhancing mechanical properties of the composite. It should be noted that mechanical decortication process may induce defects, such as kink bands or severe fiber damages, which may reduce the quality of fiber or the efficiency of the process.

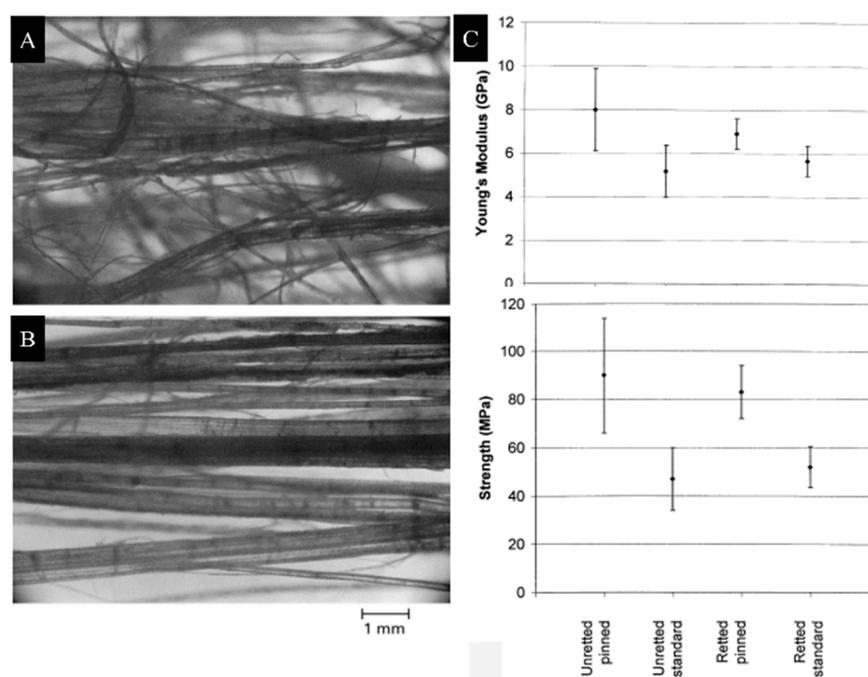


Figure 4. Images of unretted hemp fiber after (A) standard decortication and hand combing, and (B) pinning decortication and hand combing; (C) mechanical properties of retted and unretted fiber is compared with the two decortication methods; reproduced with permission from Elsevier 2000 [44].

Generally, the cortical parenchyma cells contain the pectic and hemicellulose-rich cell walls that is distinguished from the woody core, which has lignified cells. Steam explosion is a thermo-mechanical–chemical defibration method that breaks down the lignocellulosic component due to a sharp pressure change, particularly to enhance enzyme-catalyzed cellulose degradation [45,46]. The main goal is to assist in the degumming of fibers from the bundle, upon which carding mechanical treatment can be performed to separate the single fibers from the bulk [47]. In a study by Keller, it was reported that hemp fiber produced as a result of steam explosion resulted in five-times longer fiber and a

four-times higher aspect ratio compared to biologically treated hemp fiber [48]. Importantly, many studies have reported that steam explosion treatment is capable of enhancing the disintegration of hemp fibers, thereby increasing cellulose content up to 90 wt.% from 73 wt.% [34]. Steam explosion treatment has been reported to increase the crystallinity of hemp fiber as well from 70.9% (non-treated) to 79.7% (steam explosion) [49]. Figure 5 shows the SEM images of hemp fibers without pre-treatment, dew-retted fibers still fastened in bundles with amorphous matrix, and after steam explosion and NaOH pre-treatment, respectively, where hemp parts are separated into elementary fibers after the surfaces have been cleaned [49].

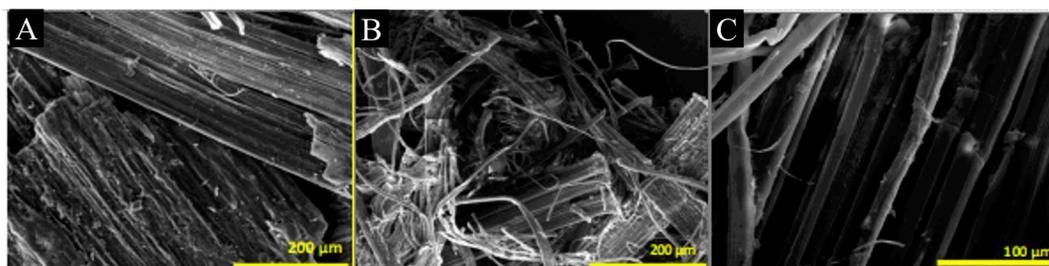


Figure 5. SEM images of (A) hemp fibers without pre-treatment, (B) dew-retted fibers still fastened in bundles with amorphous matrix, and (C) after steam explosion and NaOH pre-treatment, respectively, where hemp parts are separated into elementary fibers after the surfaces have been cleaned; reproduced with permission from IOP Publishing [49].

Chemical treatments involve the use of chemicals to remove the non-cellulosic components, such as pectin, hemicellulose and lignin, from natural fibers. For example, chemical chelators, such as ethylene diaminetetraacetic acid (EDTA) and ethylene diamine tetra (methylene phosphonic acid) (EDTMPA) [39,50], can be used to loosen the pectin structure of natural fibers, while alkali, such as sodium hydroxide or potassium hydroxide [51,52], can be used to remove the hemicellulose structure, such as xyloglucan, from natural fibers. The removal of pectin is important since it acts as the glue to bind single fibers together, and hence, the removal of pectin enhances the production of individual fibers in addition to enhancing the effective fiber stiffness and strength (Figure 6). In a study by Liu et al., it was reported that a higher EDTA concentration (2–3% EDTA) resulted in greater removal of calcium from the hemp fibers, i.e., 800 mg/100 g dry matter compared to 490 mg/100 g dry matter with 0.5% EDTA [53]. The higher removal of calcium indicates greater removal of homogalacturonan (HG), which is the abundant pectic polysaccharide in plant cell. The removal of hemicellulose increases separation of hemp fibers from the bundle and cleanliness of the fiber's surfaces to increase the stiffness of the composite. In another study using lime matrix, it was reported that chemically treated hemp fiber led to effective strengthening mechanisms at the lime–hemp-fiber interface, which resulted in enhanced strength of the composite, which is attributed to the specific rigidening process on the fiber itself [54]. Graupner et al., reported that adhesive forces as well as capillary forces and Van der Waals' forces improve the adhesive properties of hemp fibers subjected to various chemical treatments [55]. In another similar study, it was reported that chemically treated hemp–glass fibre–polyester-based hybrid composites exhibited a larger contact angle in comparison to untreated fiber composites, which is indicative of the higher thermal stability and wetting behaviour of composites [56].

Another common pre-treatment technique of hemp fiber involves enzyme treatment, in which biocatalysts selectively remove non-cellulosic components of hemp fibers under mild conditions, such as low temperature and neutral pH, e.g., pH = 5, $T = 50^{\circ}$, for the degumming of hemp fiber using acetic acid–sodium acetate buffer solution containing laccase, hemicellulase and 2,2,6,6-tetramethylpiperidine-1-oxyl radicals (TEMPO) [57]. The idea of an enzyme pre-treatment is similar to the other aforementioned pre-treatment techniques, wherein pectin is removed from the middle of the lamella of hemp fibers to

weaken the bonding between individual fibers, such that fibers can be separated from the bundle. In a study by Liu et al., it was reported that 0.2 wt.% endo-polygalacturonase decreased the polygalacturonan content by 3.5 wt.% compared to untreated hemp fibers [53]. Similar findings were reported in the study by Li and Pickering, in which pectinase assisted in decreasing the pectin content by 0.5 wt.% compared to untreated fibers. Furthermore, the treated fibers exhibited cleaner surfaces and better mechanical properties compared to untreated fibers [58].

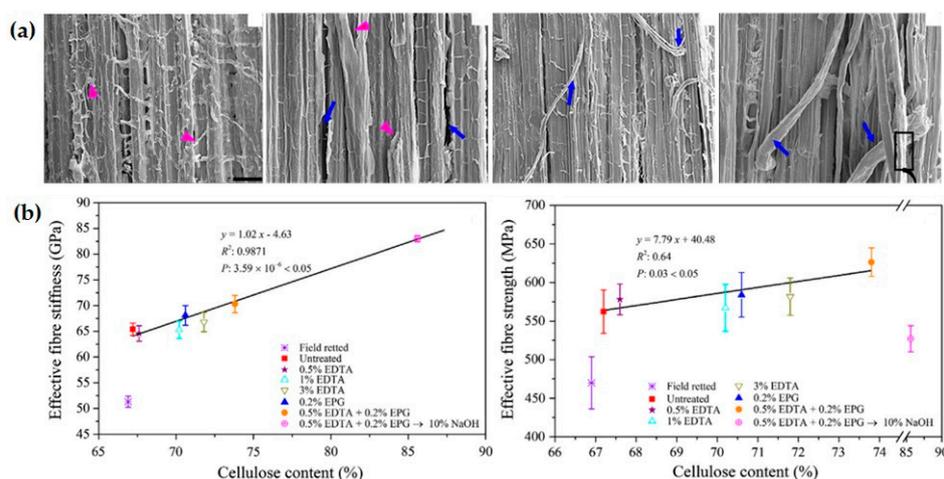


Figure 6. (a) SEM images of unretted hemp fiber, treated with 2% EDTA, treated with 0.2% EPG and treated with 0.5% EDTA + 0.2% EPG + 10% NaOH; (b) effect of various fiber pre-treatments on fiber stiffness and strength with their respective cellulose content where the pink arrowheads indicate parenchyma cell residues on fibre surface while the blue arrows indicate separated fibers and voids between fibres; reproduced with permission from Elsevier 2016 [53].

2.2.2. Surface Modification

Kabir et al. investigated the effect of surface modification on the structure of hemp fibers when the hemp fibers were treated with alkali, acetyl and silane chemicals [59]. Authors reported that cellulose was more heat-stable than lignin, while hemicellulose disintegrated rapidly compared to both cellulose and lignin when exposed to high temperatures. In another study Sunny et al., reported that the treatment of fibers at 120° using 5 wt.% NaOH improved the tensile strength and Young's modulus by 51% and 62%, respectively, in comparison to untreated fibers [60].

It was also highlighted that silane treatments were ineffective for the removal of both hemicellulose and lignin, although alkalization and acetylation were more efficient in the removal of both substances [61]. Islam et al., investigated the physio-mechanical properties of polylactic-acid (PLA)-reinforced hemp-fiber composites where the hemp fibers were subjected to alkali treatment [62]. Authors reported that the treatment of hemp fibers with alkali improved the interfacial bonding between the fibers and PLA matrix and also enhanced the percentage crystallinity of hemp fibers. Although the alkali treatment generally improved the strength of the composites, better strength for high fiber content in long-fiber composites were achieved when fibers were untreated. This was attributed to the lower fiber–fiber contact between alkali-treated fibers. Singh et al. reported that among the various pre-treatment techniques, such as wet oxidation (WO), hydrothermal treatment (HT) and steam explosion (SE), alkaline wet oxidation yielded the optimal quality of fibers, which can be utilized in textile applications [63]. In a similar vein, Oza et al. reported that the surface functionalization of fibers by alkali, silane or acetic anhydride increases the bond energy between the polymer matrix and hemp fiber, which results in higher activation energy, thereby increasing the thermal stability of the composite [64].

The graft co-polymerization of short-chain molecules and polymers on the fiber's surface can improve the wettability of fibers by matrix polymer. For instance, alkoxy

silane can be used as the coupling agent to modify the surface of hemp fiber by forming chemical bonds such as O-Si with hydroxyl groups. Studies have reported that silane treatment resulted in higher interfacial shear strength of polylactide (PLA)-reinforced hemp fibers compared to composites without silane treatment [52]. Pickering et al. reported stearic acid as an effective coupling agent to improve fiber wettability by increasing contact between the fiber and the matrix to improve strength, although it is not as effective as maleic anhydride polypropylene [65]. In other studies, the term “reactive compounding” has been commonly referred to as the technique used to increase the physical properties of a material by selective coupling chemistry. In a study by Rachini et al., it was reported that reactive compounding improved the interfacial adhesion between a polypropylene polymer and hemp fiber via the simultaneous use of two functionalized organosilanes, which creates the Si-O-Si network between the grafted organosilanes during the reactive extrusion process [66]. Authors reported that this technique was able to shift the thermal deterioration to higher temperatures while reducing water uptake at room temperature.

Physical treatment is another surface modification technique that changes the structural and surface properties of hemp fibers to improve the mechanical bonding between hemp fiber and polymer matrix. Examples of physical treatments that have been reported in the literature to date includes ultraviolet (UV), plasma, corona and gamma radiation. UV light exhibits higher energy than visible light, particularly in the presence of oxygen, and results in more hydrophilic fiber surfaces. Studies have reported up to 200% and 30% improvement in polarities on fiber surfaces and composite strength, respectively, when subjected to UV radiation treatment [67]. On the other hand, corona treatment uses low-temperature corona discharge plasma for oxidation and an etching effect on the surface of fibers to enhance the interfacial adhesion between fibers and polymer matrix. In a study by Ragoubi et al., it was reported that the tensile strength and stiffness of hemp fiber reinforced polypropylene (PP) polymer, improving them by 31% and 12.6%, respectively, after corona treatment [68]. Similarly, gamma radiation is a high-frequency electromagnetic radiation that changes the structure of hemp fiber to increase interfacial bonding between fibers and the matrix. In a study by Bilal et al., it was reported that tensile strength increased from 45.9 MPa (without irradiation) to 81.1 MPa (after irradiation of 10 KGy for 20 min), indicative of approximately 1.8 times improvement [69]. The same authors also reported that the increase in mechanical properties is related to an increase in gamma radiation doses, which enhances the cross-linking reaction and polymerization process by developing solid free-radicals and polymer chains.

Esterification is another surface modification technique used for the pre-treatment of hemp fibers to reduce the hydrophilic nature of fibers (e.g., replacing the hydroxyl groups with acetyl groups using acetic anhydride or vinyl acetate as reagents). Such acetyl treatment removes the hemicellulose and lignin components of the hemp fiber [24]. In a study by Naik et al., it was reported that hemp fibers esterified with maleic anhydride showed reduced surface and volume resistivity in comparison to untreated fibers [70]. This indicates that esterification reactions enhance the compatibility of hemp fiber with the polymer matrix, which is important to improve the interfacial adhesion between fiber and the matrix, thereby enhancing mechanical properties of the composites. In another study by the same group of authors, it was reported that maleic-anhydride-treated-hemp-fiber composites resulted in lower absorption of water and steam at ambient temperature in comparison to untreated-hemp-fiber composites [71].

2.2.3. Antimicrobial Degradation

Biological degradation represents an issue related to the use of natural fibers and can be attributed to several factors, such as the presence of microorganisms in nature or moisture uptake by fibers [72,73]. To overcome this issue, the use of germicidal agents on the surfaces of hemp fiber via chemical or enzymatic methods can protect the fibers from bacteria or fungi [74]. Several germicidal agents have been reported in the litera-

ture to date to prevent the microbial degradation of hemp fiber in composites, such as phenolic compounds (ferulic acid, p-coumaric acid, and syringaldehyde), flavonoid compounds such as catechin or metallic salts such as Ag^+ and Cu^+ . For instance, in a study by Kostic et al. [75], it was reported that silver-loaded hemp fibers resulted in enhanced antimicrobial activity, such as 100% reduction of microorganism *S. aureus* and 99% reduction of *E. coli* and *C. albicans* with a silver content of 1.3 mmol/g. Milanović et al. [76] also reported that metallic salts of AgNO_3 improved the antimicrobial activity of hemp fiber; however, there were concerns regarding the decreased mechanical properties, which might have occurred during the first stage of the process that involves oxidation using TEMPO, sodium bromide and sodium hypochlorite to create reactive sites on hemp fibers for the incorporation of metallic ions (Ag^+) [76]. To overcome these issues, milder oxidase enzymes such as laccases and peroxidases can be utilized to increase the antimicrobial activity of hemp fibers since a significant enhancement in antimicrobial degradation have been reported using milder oxidase enzymes for linen fibers, wood fibers and flax fibers [77–79]. Table 2 summarizes the various defibration and surface modification techniques of hemp fibers, their requirements, advantages and limitations.

Table 2. Defibration and surface modification techniques of hemp fibers, their requirements, advantages and limitations.

Pre-Treatment Purpose	Technique	Requirements	Advantages	Limitations
Defibration Fiber extraction	Traditional retting	Water	<ul style="list-style-type: none"> • Low environmental impact • Sustainable process • Most economical • Uniform quality of fibers 	<ul style="list-style-type: none"> • Weather conditions and soil quality influence strength and quality of fibers • Large water consumption (contamination can lead to major environmental issues) • High labor costs
	Controlled microbiological retting	Sterilization of equipment Microorganisms	<ul style="list-style-type: none"> • Low environmental impact 	<ul style="list-style-type: none"> • Some environmental impact • Microbial contamination leads to over-retting fiber which reduces fiber strength • Microbial colonization affects/disintegrates hemp structure
	Mechanical treatment	Electricity Decortication equipment	<ul style="list-style-type: none"> • Very low environmental impact • Simple process to produce high fiber quantity in short duration 	<ul style="list-style-type: none"> • Equipment can be expensive • Lower fiber quality
	Steam explosion	Electricity High-pressure equipment	<ul style="list-style-type: none"> • Low environmental impact • Economical 	<ul style="list-style-type: none"> • Formation of degradants that inhibits fermentation and enzymatic hydrolysis

Table 2. Cont.

Pre-Treatment Purpose	Technique	Requirements	Advantages	Limitations
	Chemical treatment	Alkali Chelators Sodium sulphite	<ul style="list-style-type: none"> • Process not affected by weather conditions • High quality and consistency of fibers in short time 	<ul style="list-style-type: none"> • High environmental impact • High processing cost • High consumption of water, chemicals and energy
	Enzyme treatment	Pectinolytic enzymes	<ul style="list-style-type: none"> • Very low environmental impact • Process carried out under controlled conditions • Fast and clean process • High quality and consistency of fibers 	<ul style="list-style-type: none"> • Not economical
Surface modification	Alkali treatment	Alkali	<ul style="list-style-type: none"> • Economical • Enhanced fiber–matrix adhesion 	<ul style="list-style-type: none"> • High environmental impact • Consumption of chemicals
Increased moisture resistance Antimicrobial degradation activity	Graft co-polymerization	Phenol/phenol derivatives, oxidase enzymes as catalyst	<ul style="list-style-type: none"> • Low environmental impact • Improved chemical resistance and moisture absorption behavior 	<ul style="list-style-type: none"> • Not economical
	Physical treatment	Equipment for UV, plasma or gamma radiation	<ul style="list-style-type: none"> • Low environmental impact 	<ul style="list-style-type: none"> • Degradation of structure from under/over exposure to radiation rays
	Esterification	Glacial acetic acid/vinyl acetate	<ul style="list-style-type: none"> • Moderately economical • Improved chemical resistance and moisture absorption behavior 	<ul style="list-style-type: none"> • High environmental impact

3. Mechanical Properties of Hemp-Fiber-Reinforced Composites

Hemp fiber has been commonly used as reinforcement, owing to their excellent properties, such as high elastic modulus and a strength of 20–41 GPa and 210–750 MPa, respectively, with approximately 70% lower cost than synthetic glass fiber [4,5]. Additionally, hemp fiber is classified as a natural fiber sourced from plants, which are renewable resources. As such, they can offer an environmentally friendly replacement for synthetic fibers, such as carbon fiber or glass fiber. More importantly, hemp fibers, when burned, leave minimal residue and CO₂ emissions upon combustion, all of which establishes hemp fiber as an alternative choice for reinforcement in composite materials.

3.1. Effects of Pre-Treatment

Similar to most filler-reinforced composites, the degree of reinforcement depends on the type of filler, processing technique of the composite, the bonding and the interfacial adhesion between hemp fiber and the matrix. The incompatibility between the matrix and

hydrophilic lignocellulosic molecules of hemp fiber leads to poor surface adhesion due to insufficient wetting, which causes the formation of weak interface bonding between hemp fiber and the matrix. As discussed in Section 2 of this review, the pre-treatment of hemp fiber can improve the interfacial adhesion between hemp fiber and the matrix, thereby enhancing mechanical properties of the composite. For instance, pre-treatment with alkaline NaOH increases surface roughness of hemp fiber [80]. The higher surface roughness results in increased effective wetting area as well as increased number of active OH functional groups to create better interactions on the fiber's surface, which can enhance the mechanical bonding of fiber with the matrix. Hemp fibers are bounded by lignin-rich, waxy and oily surface with weak intermolecular bonds, and an alkalization treatment provides a cleaner and rougher surface. The optimal concentration of NaOH is also capable of separating the fibers in a bundle into individual fibers. Maichin et al. [81] found that the pre-treatment of hemp fiber with a concentration of 5M NaOH increased the surface roughness of hemp fibers by washing away impurities and waxes on fiber's surface, which enhances bonding with the host matrix. This was ascertained by the contact angle measurements, wherein lower values were obtained, which indicates the "less hydrophobic" property of the hemp surface due to minimal oil and waxy substances. Authors also reported that higher alkalinity may enhance the removal of oil and waxy substance on hemp surfaces to increase surface roughness of the fiber, all of which can improve interfacial adhesion with the host matrix, thereby improving the mechanical properties of the resultant composite. In another comprehensive study, Thomsen et al. [82] investigated the effects of various pre-treatment techniques, such as wet oxidation, hydrothermal treatment and steam explosion and reported that, while all pre-treatment techniques increased the cellulose content of fiber by degrading and dissolving non-cellulosic material such as pectin and waxes, alkaline steam explosion produced fibers with the highest cellulose content (86–90%), which are highly desirable for use as reinforcement in composite materials (Figure 7); however, it is noteworthy that steam explosion requires higher temperatures and more expensive reactor materials for better biomass treatment, while the use of some acids in catalytic steam explosion technology may lead to the dissolution of cellulose and hemicellulose, which leads to loss of dry matter, reduced product quality and yield [83]. On the other hand, wet oxidation technologies can be costly since it requires oxygen and a catalyst, which can be considered a major limitation.

In recent years, several novel technologies have been implemented for the pre-treatment of lignocellulosic biomass, such as microwaves, ultrasound, gamma ray, electron beam, pulsed-electric field, high hydrostatic pressure and high pressure homogenization [84]. Microwave irradiation is an energy-efficient process that offers significant advantages compared to conventional heating, such as faster heat transfer, which shortens reaction time, lowers degradation and the formation of side products, as well as increases the removal of acetyl groups in hemicellulose due to the hot spot effect from microwave irradiation [85]. On the other hand, the ultrasonic pretreatment of biomass can be considered as an effective green technology where surface structure is altered to produce oxidizing radicals to chemically attack the lignocellulosic matrix. In a study by Sun et al., it was reported that 90% of hemicellulose and lignin were removed via the ultrasound irradiation technique at an ultrasound power of 100 W and sonication time of 2 h in distilled water at 55° [86]. Some other studies have used a combination of microwave and ultrasound to achieve higher effectivity for pretreatment of lignocellulosic biomass. The combination of these techniques allows for the selective degradation of waxes and lignin, wherein the microwave technique will be capable of removing the waxy layer from the surface of biomass to increase availability of surface area for enzymic actions [84]. Other techniques such as electron beam irradiation is capable of disrupting the structure of cell wall polymers, such as lignin, cellulose and hemicellulose, when beams of electron irradiate the lignocellulosic biomass. In a study by Mante et al., it was reported that electron beam irradiation at dosages of 1000 kGy depolymerized cellulose and hemicellulose structures to increase yield of phenolics [87]; however, it is noteworthy that this technique is primarily effective for the depolymerization

of cellulose; as such, it requires a combination of other techniques, such as steam explosion or alkali pre-treatment, for the hydrolysis of hemicellulose and lignin.

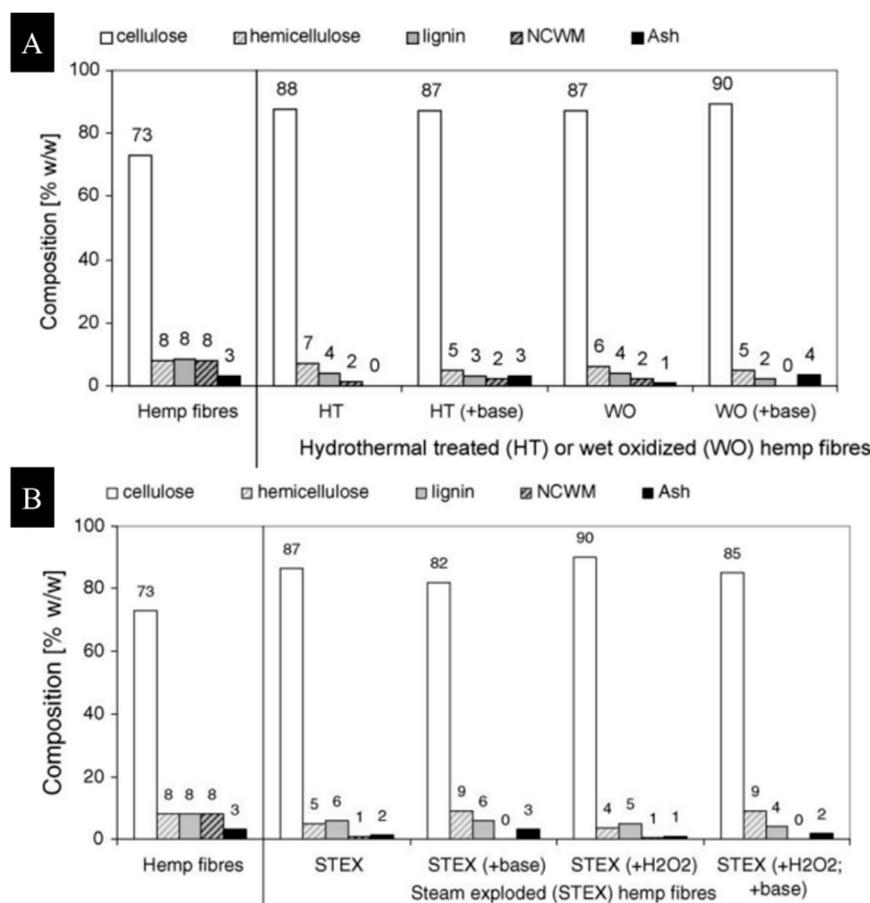


Figure 7. Chemical composition of (A) hemp fiber raw material in wt.% and the chemical composition after wet oxidation and hydrothermal treatment, (B) steam-exploded hemp fibers (with and without impregnation) and untreated fibers in wt.%; reproduced with permission from Elsevier 2006 [82].

3.2. Effects of Fiber Alignment

Several studies have investigated the effect of fiber alignment on the mechanical properties of composites. For instance, Hargitai et al. [88] studied the anisotropy of the mechanical properties of PP-reinforced hemp fiber resulting from carding technology. The same authors reported that both tensile strength and Young's modulus of the composites were higher when fibers were aligned in the parallel direction compared to in the perpendicular direction. For PP composite reinforced with 50 wt.% hemp fiber, the Young's modulus and strength were approximately 62.5% and 42.9% higher, respectively, in the parallel direction. This is because fibers laid in direction perpendicular to load will not be capable of bearing sufficient load in the composite material upon loading, thereby causing failure. Similarly, Baghaei et al. [89] found that composites with fibers oriented at an angle of 0° exhibited higher flexural strength, modulus and impact strength compared to fibers along other orientation angles (Figure 8). Furthermore, it was observed that the strength and stiffness of the composite decreased with increasing fiber orientation. For instance, in comparison to neat PLA, the modulus increased by 225% for treated-hemp-fiber composite and by 203% for untreated-hemp composite with a fiber orientation angle of 0°. The composites with fibers oriented along 45° and 90° exhibited the lowest properties, since on-axis properties, such as stiffness and load-carrying capability, are dependent on fiber properties [90,91], whereas off-axis properties are dependent on matrix properties [92]. Moreover, matrices with off-axis fillers will not be capable of carrying significant load

due to the presence of porosity within the matrix, which may hamper the properties of the composite compared to neat PLA, which has lower porosity. Similar to findings reported in Section 3.1, it should be noted that pre-treatment showed optimal enhancement in mechanical properties, which can be related to stronger interface formed as a result of increased potential hydrogen bonding [62]. Although the optimum mechanical properties can be achieved in the composite by aligning the hemp fibers parallel to the direction of loading, the composite can perform poorly when load is applied perpendicular to the hemp fibers. Creating a composite with aligned fibers and optimum mechanical properties in all directions will incur additional cost and several manufacturing challenges.

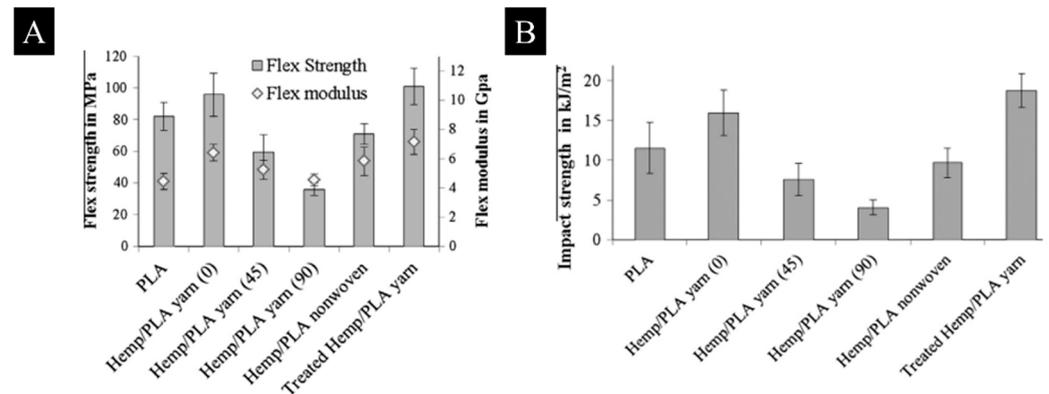


Figure 8. (A) Flexural modulus/strength and (B) impact strength as a function of fiber orientation and pre-treatment; reproduced with permission from Elsevier 2014 [89].

3.3. Toughening Mechanisms

Common toughening mechanisms in hemp-fiber-reinforced composites include crack deflection due to load transfer and crack bridging due to fiber pull-out. To achieve significant improvement in mechanical properties, several parameters are important, such as sufficient fiber content capable of bearing the load and good interfacial adhesion between fiber and the matrix [93,94]. For instance, the matrix bears most of the load when reinforced with insufficient fiber content, i.e., the strength of the composite will depend solely on matrix strength and the fibers will act as flaws, thereby deteriorating the properties of the resultant composite [95–97]. Similarly, without pre-treatment, the surfaces will appear smooth since it is covered with non-cellulosic material that results in poor adhesion between fiber and the matrix, which, in turn, leads to fiber failure by severe debonding. In such cases, the fiber will lose its load-bearing capability since failure occurs mostly at the hemp fiber and matrix interface. Fiber pull-outs and breakage were observed with alkaline-treated hemp fibers while the matrix showed a smooth and brittle fracture surface. Although the fiber pull-out and breakage might have contributed to the strength and ductility of the composite, it was observed that most cross-sectional surfaces of polymer were not reinforced by the hemp fiber due to low fiber loading in the composite [98]. Similar fracture surfaces were observed for composites reinforced with untreated hemp fiber; however, the debonding was more significant, and the very smooth surface indicates that the failure was initiated as a result of the fiber debonding.

In another study, Ragoubi et al. [68] reported that PP composites reinforced with non-corona-treated hemp fibers showed significant fiber pull-outs and a large number of unbroken fibers protruding out from fracture surfaces with a high presence of porosity (Figure 9). The smooth fracture surface and protruding fibers indicate that there was very minimal adhesion of the PP matrix to the fiber surface. Thus, it is likely for the failure to occur due to fiber debonding and tensile failure. On the other hand, when fibers were corona-treated, the fibers were covered to a large extent within the PP matrix and did not indicate signs of pull-out which suggests the excellent interfacial adhesion of fibers with the matrix. In this case, it is likely that the composite material fails by local shear yielding of the matrix around the fiber compared to the debonding of hemp fiber or tensile failure.

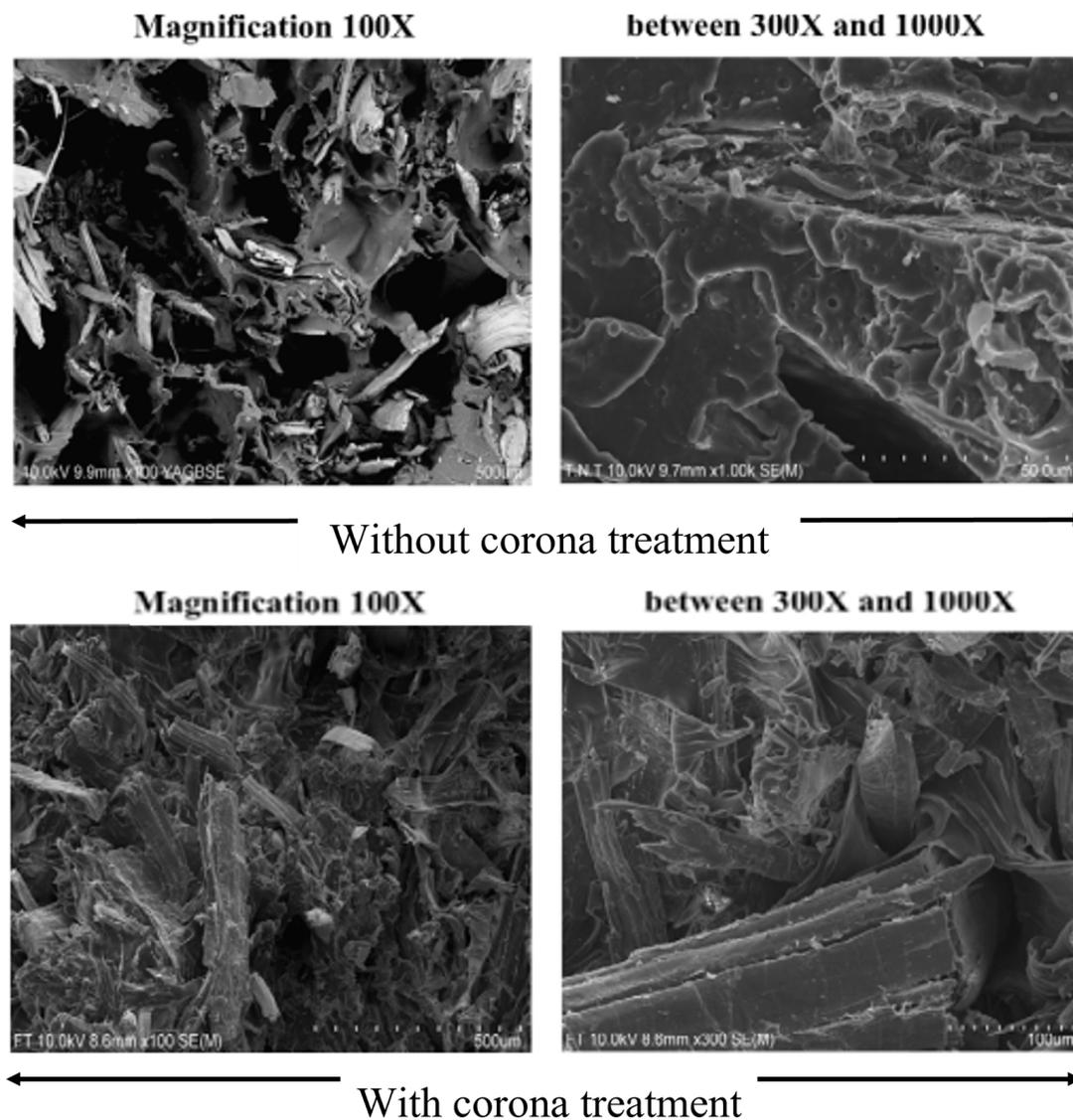


Figure 9. Effect of corona treatment on fiber pull-out and breakage upon fracture; reproduced with permission from Elsevier 2010 [68].

The degree of interfacial bonding between fiber and the matrix can be examined from microstructural examinations. For instance, the surface of fibers without matrix residues upon fiber pull-out indicates poor interfacial bonding or adhesion due to the incompatibility between fiber and the matrix [99]. On the other hand, presence of residues still attached to the surface of fibers after pull-out indicates excellent interpenetration of fibers within the host matrix, which results in enhanced mechanical properties of the composite. In an ideal situation, external load applied to hemp-fiber-reinforced composites should be transferred to the fibers, which allows them to take major share of the load. For example, optimal loading of hemp fibers in the matrix ensures efficient load transfer and stress distribution from matrix to the filler, which minimizes stress concentration points. It is important to minimize stress concentration points since it is postulated that fractures propagate from this point, as the entire polymer fails in a rapid manner due to the presence of a small crack. Nevertheless, other studies have reported that fiber-reinforced composites with low interfacial bonding exhibit higher energy absorption due to the loose bonding that provides a pathway for impact energy to be dissipated as kinetic and heat energy [100]. Table 3 summarized the effect of hemp fibers as reinforcement on the mechanical properties of various composites.

Table 3. Effect of hemp fibers as reinforcement on the mechanical properties of various composites. (a: loading was reported in volume percent, the density of hemp fiber (1.25 g/cm³) was used to convert to a weight percent loading).

Matrix	Filler Composition [wt.%]	Tensile Strength [MPa]	Tensile Modulus [GPa]	Flexural Strength [MPa]	Flexural Modulus [GPa]	Ref.
Epoxy	0.00	30.00	1.10	34.69	0.60	[101]
	9.00	36.48	1.43	85.59	1.78	
Epoxy	0.00	38.11	1.38	40.30	1.32	[102]
	37.50 ^a	50.46	1.72	76.69	3.79	
Epoxy	42.20	71.55	5.85	124.52	4.15	[103]
HDPE	0.00	18.70	0.34	17.10	0.60	[100]
	50.00 ^a	60.20	2.31	44.60	2.42	
HDPE	0.00	n/a	n/a	17.80	0.47	[104]
Polyester	50.00 ^a	n/a	n/a	44.6	1.49	[105]
	10.00	111.05	2.65	60.01	4.37	
Polyester	0.00	30.00	0.60	42.00	5.81	[106]
	32.50 ^a	65.00	0.68	84.00	8.05	
Polyester	0.00	23.19	0.42	50.31	1.46	[102]
	25.00 ^a	31.46	0.51	60.06	1.86	
Polyactic acid (PLA)	0.00	35.00	3.50	n/a	n/a	[98]
	50.00 ^a	54.60	8.49	112.70	n/a	
Polypropylene (PP)	0.00	25.00	n/a	57.50	n/a	[107]
	37.50 ^a	32.50	n/a	62.00	n/a	
Polypropylene (PP)	0.00	27.10	0.67	n/a	n/a	[68]
	20.00	37.80	1.22	n/a	n/a	
Polyurethane (PU)	0.00	26.52	0.04	7.55	0.157	[108]
	50.00 ^a	27.23	0.54	22.14	0.587	

4. Application Perspectives of Hemp Fiber Reinforced Composites

Hepworth et al. [44] reported that unretted hemp fibers subjected to decortication treatment resulted in enhanced strength and stiffness, which is useful as structural material in composite manufacturing; however, in another study by Caprino et al. [109], low-impact testing was performed on unidirectional- and bidirectional-hemp-fiber-reinforced polymer laminates, and it was reported that, although unidirectional laminates were more sensitive to impact loads than bidirectional laminates, hemp fiber in general had no potential to replace glass fiber in structural applications. Exploring other potential application of hemp-fiber-reinforced composites, Kymäläinen and Sjöberg [110] investigated the suitability of using flax and hemp fibers as raw materials for thermal insulators. The same authors reported that natural fibers can be utilized as insulators in the future, owing to their biodegradable nature. However, owing to the high susceptibility to contamination and microbial attack, the properties should be carefully monitored, although additives may prove beneficial to offset the negative effects of fibers on indoor air quality. In an interesting study, Scarponi and Messano [111] conducted a preliminary investigation on the suitability of using E-glass and hemp-fiber composites for helicopter devices as well as semi-structural applications, such as electronic racks. In particular, authors successfully reported the substitution of a steel electronic rack mounted on an Eurocopter AS 350 Ecureuil with a hemp-fabric-reinforced epoxy composite. A weight reduction of approximately 55.6% was obtained with this substitution, which translates into lower fuel consumption, lower pollution and lower cost. Figure 10 summarizes various other potential applications of hemp-fiber-reinforced composites; such as strengthening unreinforced masonry walls using natural hemp fibers [112]; as a loose-fill insulation material [113]; in automobile

parts [114,115]; for biomedical applications, such as for joints and bone fixtures to alleviate pain in patients [116]; or for shoe material, owing to the good moisture absorption and higher evaporation rate among other materials in high-humidity environments [117–119].



Figure 10. Application perspectives of hemp-fiber-reinforced hemp fiber reinforced composites.

5. Conclusions and Perspectives

In conclusion, progress has been made in using natural fibers, such as hemp fiber as reinforcement in polymer matrices, in the past decade. Pre-treatment for lignocellulosic materials is important for the removal of lignins and other non-cellulosic components as well as for easing the separation of fibers from the fiber bundle. Most studies have consistently reported the importance of lignin, pectin and hemicellulose removal to attain fibers with high properties, which can be used as reinforcement in composites. Pre-treated fibers, when used as reinforcement in polymer matrices, have enhanced tensile and flexural stiffness and strength and impact the strength of the resultant composites.

With the knowledge on pre-treatment of hemp fibers and their reinforcement mechanism, there are still many challenges that can be addressed, such as:

- The effects of hemp-fiber alignment on the mechanical properties of the composite have not been well investigated. Techniques to achieve fiber alignment can be explored to attain excellent properties in a specific direction;
- Research can also be intensified on additive manufacturing of hemp-fiber-reinforced composites. This provides the opportunity to fabricate structures with complex geometry and near complete design freedom at a lower cost and with fast turnaround time. Moreover, exploring 3D printing for fabrication of hemp-fiber-reinforced composites minimizes material wastage since the technique only adds the material required for fabricating a structure;
- Studies have commonly reported the deterioration of mechanical properties at very high fiber loading. As such, it is important to investigate the fundamental idea of stress transfer and interfacial bond strength that influences properties of the composites (e.g., theoretical analysis).

Furthermore, it is important to intensify the interdisciplinary research and collaboration on various aspects of hemp fiber to fully exploit the potential of hemp-fiber-reinforced polymer composites. Despite the gaps in research, hemp-fiber-reinforced composites have established their significance by enhancing mechanical properties of the resultant composite. As such, hemp-fiber composites are expected to have a brighter future to be used in many applications. If overriding problems such as quality of hemp fibers, suitable pre-treatment of hemp fiber and achieving isotropic properties with suitable processing technique for bulk manufacturing can be addressed and research continues apace, it is

very likely to expedite application perspective of hemp-fiber-reinforced composites to the marketplace.

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