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Review Article

Physical and Chemical Modifications of Plant Fibres for Reinforcement in Cementitious Composites

R. Ahmad,[1] **R. Hamid**,[1,2] **and S. A. Osman**[1,2]

[1]*Smart and Sustainable Township Research Centre, Faculty of Engineering and Built Environment, University Kebangsaan Malaysia, 43600 UKM, Bangi, Selangor, Malaysia*
[2]*Civil Engineering Programme, Faculty of Engineering and Built Environment, University Kebangsaan Malaysia, 43600 UKM Bangi, Selangor, Malaysia*

Correspondence should be addressed to R. Hamid; roszilah@ukm.edu.my

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This paper highlights the physical and chemical surface modifications of plant fibre (PF) for attaining suitable properties as reinforcements in cementitious composites. Untreated PF faces insufficient adhesion between the fibres and matrix due to high levels of moisture absorption and poor wettability. These conditions accelerate degradation of the fibre in the composite. It is also essential to reduce the risk of hydrophilic PF conditions with surface modification, to enhance the mechanical properties of the fibres. Fibres that undergo chemical and physical modifications had been proven to exhibit improved fibre-matrix interfacial adhesion in the composite and contribute to better composite mechanical properties. This paper also gives some recommendations for future research on chemical and physical modifications of PF.

1. Introduction

The incorporation of fibre in cementitious material composites as a reinforcement can enhance the flexural limit during splitting, durability, ductility, and break resistance compared to the unreinforced matrix [1]. In addition, the benefit of fibre as reinforcement in cement composites is the ability to control crack growth and to increase ductility [2]. Much work has been carried out to identify the potential use of natural fibre (NF) compared to artificial fibre in strengthening composites. Efforts have been made to utilize NF-reinforced composites as part of building material in the construction industry. By exploiting the benefits of NF due to its lower density, tool wear, and cost, NF has overtaken artificial fibre in many applications and is well suited for use as a reinforcement either in a cement matrix or in polymer composites [3]. Additionally, the renewable and biodegradable characteristics of NF make the fibres easy to dispose of by composting or incineration compared to artificial fibre.

More recent evidence shows that sustainability, renewable sources, and broad use of NF have introduced several plant fibres (PF) into the biocomposites field [4]. PF also have a great potential to replace glass in many applications [5], and they cause less dermal and respiratory irritation than glass fibre [6]. Other observations by several researchers also indicate that PF can yield the same flexural strength and a higher Young's modulus compared to glass fibre [7–10]. The use of PF can easily be adopted in cement composites for economic and environmental reasons if the hydrophilic nature, low processing temperature, wettability, incompatibility, and high moisture absorption of the fibres are defeated. Hence, this paper reviews the challenges in resolving the above issues of using PF in composite. The research on the physical and chemical treatments of PF to enhance its mechanical and physical properties is ongoing so that PF can compete with other synthetic fibre-reinforced composites and wood products and bring many potential applications in the construction industry [11].

2. Natural Fibres

Fibres are classified according to their origin and are divided into synthetic fibre and NF. NF is extracted from plant, animal, and mineral sources [12]. Examples of fibre classification are

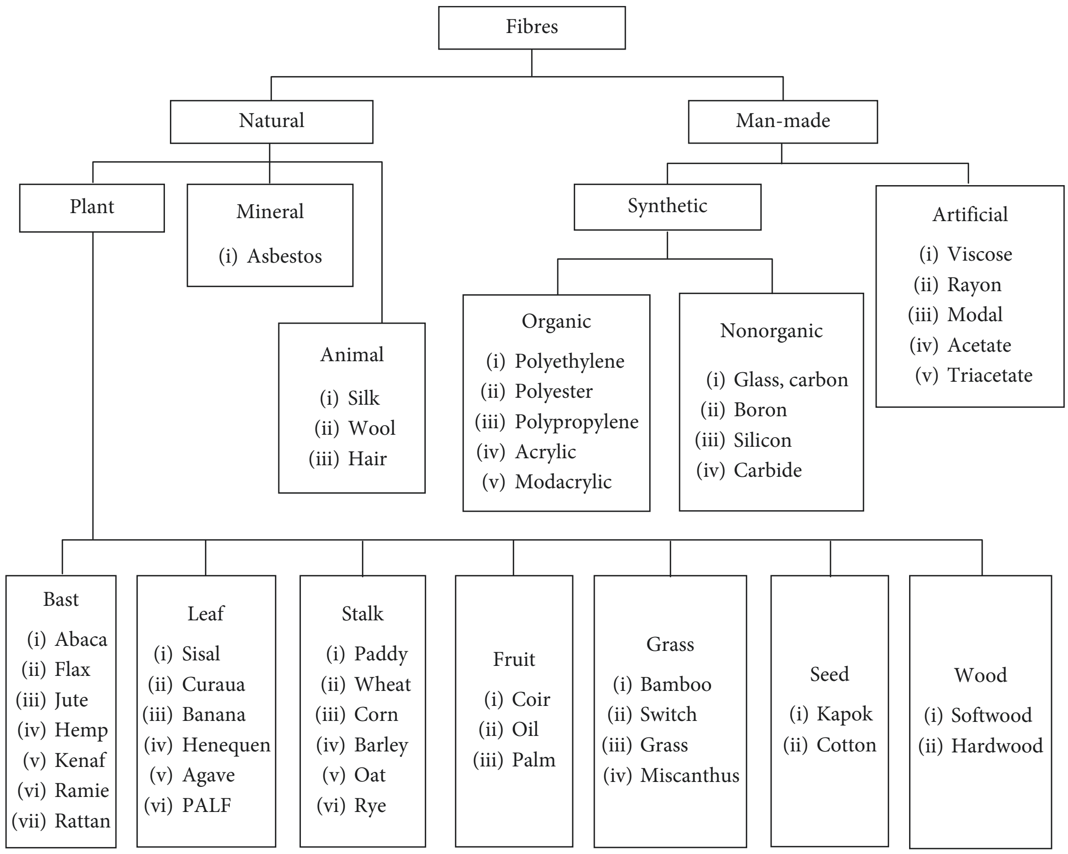

Figure 1: Fibre classification.

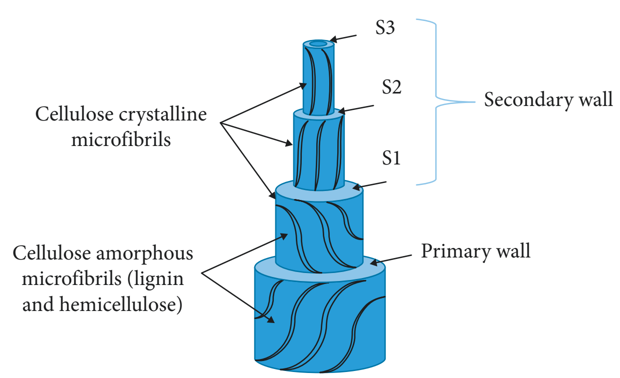

Figure 2: Schematic representation of PF structure (source: [13]).

shown in Figure 1. The fibres extracted from plants are further classified into bast, leaf, stalk, fruit, grass, seed, and wood. Figure 2 shows the PF structure of the amorphous cellulose held together by a lignin and hemicellulose oriented randomly in cell walls. Hemicellulose, lignin, pectin, and the waxy substances contained in lignocellulose are the utmost chemical components in PF [14]. The hemicellulose provides cementing material in the cell wall and forms a matrix surrounding the cellulose microfibrils, whereas the amorphous lignin gives additional strength and coupling to the hemicellulose-cellulose network, which becomes a protective barrier in fibres [15]. From Figure 2, the crystalline cellulose microfibrils in secondary walls (S2) determine the fibre mechanical properties and provide good mechanical properties when used alone rather than combined with individual fibres for composite applications [16]. The chemical composition of PF after surface modification has a strong impact on the mechanical properties by removing lignin and hemicellulose [17], which are responsible for the bonding behaviour and degradation of PF in composites.

In addition, the properties of PF also vary depending on the internal structure, fibre diameter, microfibril angles, cell dimensions, crystal structure, and defects [18]. Other factors influencing the PF properties are different fibres taken from different parts of plants, such as fibres extracted from the stem, leaf, or seed, and different growing conditions [19]. The advantages and disadvantages of bast fibre are shown in Figure 3. Based on the weaknesses of PF shown in Figure 3, researchers have initiated detailed studies to identify the effect of physical treatment on PF for improvement in its mechanical properties and to improve the properties of the composite materials. Table 1 shows that physical treatments contribute significantly to enhancing the fibre tensile strength and the mechanical properties of the polymer composites. However, fibre strength is not the dominant factor related to composite strength, whereas good fibre orientation/dispersion and excellent bonding between the fibre-matrix proved to be the promoting factors in accelerating the mechanical properties in flexural and tensile strength [27]. The most important limitation lies in the hydrophilic nature of cellulose fibre that affects the mechanical properties and performance of PF in composites [28].

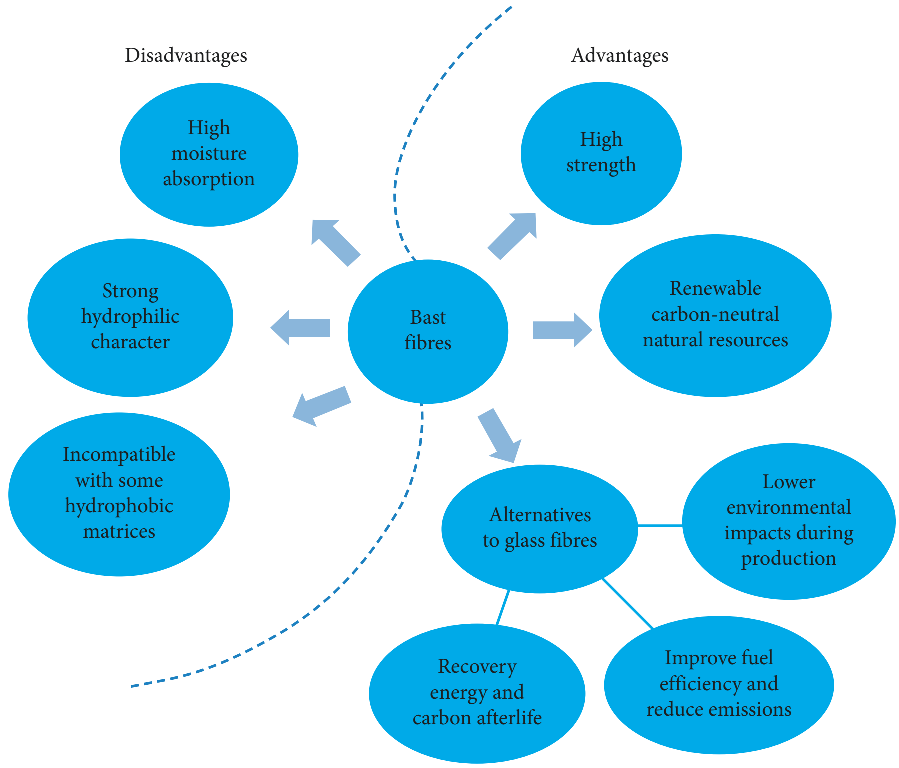

Figure 3: Advantages and disadvantages of bast fibres (source: [20, 21]).

Table 1: Effect of PF physical treatment on the mechanical properties of composite materials.

Fibres			Matrix	Mechanical properties (MPa)		Ref.
Types	Treatment	Tensile (MPa)		Compressive	Flexural	
Jute	Argon cold plasma	—	Unsaturated polyester resins	NA	180	[22]
Arundo Donax	Plasma	192.32	Epoxy composites	NA	75	[23]
Jute	Ionized air	547	Phenolic composites	37.5	NA	[24]
Piassava	Electron beam	—	Composites	23	17	[25]
Kraft	Fibre beating	45	PP composites	41	NA	[26]

PP, polypropylene, NA, not available.

3. Composites

The cement mortar composite material should be developed to acquire properties that cannot be achieved by any of the materials independently. Shanks [29] has indicated that the reinforcement and matrix are the two main constituents in composites, where reinforcements are fibre structures that give strength to the materials and the latter surrounds the fibres with elastic interaction that holds them in place, transfers force between the fibres, and resists a small portion of the loading [30]. The combination of the properties of cement and fibrous materials treated with chemical can contribute in the performance of mortar as building material [31]. Mishra et al. [32] found that the performance of the composites can be enhanced persistently through thorough experimentation by blending two or more fibres or fillers, and Kwon et al. [33] indicated that the composite strength and stiffness are transferred by the fibres. Although synthetic fibres are the most common type of fibre reinforcements used in composites, the interest in NF as reinforcement has grown because of its low cost, weight reduction, nontoxicity, ease of recyclability, and biodegradability [34, 35].

4. Natural Fibre-Reinforced Composites

Hassanin et al. [36] stated that the largest advantages of using NF in cement composites are the low cost of materials, sustainability, and density. The properties of fibre-reinforced composite materials are controlled by a number of factors as follows: (1) magnitude and proportion of the fibre-matrix elasticity; (2) type and properties of the matrix, such as ductile or brittle; (3) fibre content, length, and orientation; and (4) interfacial bond strength in the fibre-matrix [37]. However, the bond strength solely depends on the effectiveness of the fibre as reinforcement in composites [38]. Due to poor interfacial bonding between the hydrophilic fibres and the hydrophobic polymer matrices, Romanzini et al. [39] conducted a study and found that the addition of treated stiff fibres in composites can produce a new material with outstanding mechanical properties. Other observations by Bentur and Mindess [40] confirm that adding fibres can enhance the abrasion resistance, impact resistance, and fatigue characteristics of cementitious composites.

However, fibre-reinforced cement composites tend to be exposed to degradation during wet/dry cycles from accelerated ageing. This condition will affect the durability of cementitious composites. However, according to Mohr et al. [41], the degradation of fibre-reinforced cement composite can be mitigated with fibre and matrix modifications. Fibre modifications will stimulate chemical composition, dimensional stability, and bond strength in PF cementitious composites. Meanwhile, matrix modifications containing blended cementitious materials as a partial replacement of OPC are effective in preventing degradation [42]. This suggests that the future of PF-reinforced composites seems to be bright because they are cheaper, lighter, and environmentally superior to glass fibre composites [43]. Since strength and water absorption are the two main factors that limit the performance and the urge to increase the future use of PF-reinforced composites, a thorough discussion covering physical and chemical treatments for modification of PF will be presented.

5. Mechanical Properties of Plant Fibres

Strengthening mechanisms have triggered interest among researchers to enhance the mechanical properties such as compression, tensile, flexural, or impact strength, and wear behaviour that signify the high achievement of good materials. There is a strong indication that the fibre-matrix properties are important in improving mechanical properties that determine the capability of material under extreme loading as well as in critical conditions, and this pointed to the performance of the composites [7]. Studies have been performed by Fazal and Fancey [44] on PF-reinforced composites, and they have discovered that the mechanical properties of the composites are strongly influenced by a number of parameters such as the volume fraction of the fibres, the fibre length, orientation and aspect ratio, adhesion in the fibre-matrix, and stress transfer at the interface.

When selecting a suitable fibre, the mechanical properties are extremely important and become a major element to resolve in using PF as a reinforcement in composites [45]. Various studies have been carried out to determine the effects of physical and mechanical properties on PF, and the results displayed in Table 2 show that the tensile strengths of bast and leaf fibres were the highest. As mentioned by Bledzki et al. [51], bast and leaf fibres are classified as hard fibres and are suitable for use as reinforcements in composites as they give a high stiffness and strength to the materials compared to other fibres. Hence, the technical brief of the fibre is also an important factor to determine the PF structure as well as the characteristics [52]. There are several physical properties that are significant in selecting a suitable PF for use in composites, such as fibre structure and dimensions, defects, crystallinity, variability, and cost [53]. PF has potential for use as a reinforcement material in cement composites, but it is compulsory to utilize the strong reinforcing of PF in order to produce high mechanical strength for composite materials [54].

6. Fibre Surface Properties

Surface properties are generally defined as the surface free energy that is used for characterizing the interaction between solid surfaces related to the adhesion properties of materials [55]. Findings of Dai and Fan [56] show that the surface properties of a fibre are the main factors that affect the interfacial adhesion on the surface of fibres and the mechanical properties of the composite reinforced with PF. Adhesion is needed since the fibres and matrices are chemically dissimilar, and Valadez-Gonzalez et al. [57] highlighted that the fibre surface must consequently be modified to improve the properties and interfacial interaction of PF for use as a reinforcing material in composites.

However, bast fibres will degenerate easily in the alkaline environment of the cementitious matrix, giving low impact strength and brittle composites [58]. Cordeiro et al. [55] investigated the surface properties of raw and modified lignocellulose fibres by inverse gas chromatography (IGC) treatments and found that bast fibres offered higher surface dispersive energy compared to leaf fibre. Later, Praveen et al. [59] reported that plasma-induced modification changed the surface topography and generated high water absorption of coir fibres. The test indicates that different surface property treatments resulted in different interfacial shear strength. Thus, improvement in fibre surface properties is needed and is dependent on the following factors: (1) fibre morphology, (2) chemical composition, (3) extractive chemicals and processing conditions, and (4) modification of plant fibres [60].

7. Fibre Surface Modification Methods

Fibre surface modification can improve fibre-matrix interfacial bonding, roughness, wettability, and the hydrophilic nature and can decrease moisture absorption, which can enhance the tensile properties of PF in cementitious composites [46]. However, the impurities and waxy substances that lay on the PF surface will develop poor surface wetting and reduce bonding in the fibre-matrix [61]. Thus, the PF will need to undergo surface treatment before its application as reinforcement in cement composites. On the other hand, Fiore et al. [6] stated that physical or chemical modification or a combination of both must be proceeded to strengthen the poor properties of the PF surface by reducing the polar component through (1) removal of impurities, (2) changing the crystallinity and chemical composition, (3) improving the fibre-matrix interface, and (4) attaining good adhesion in the fibre-matrix. The fibre modification methods shown in Figure 4 can be divided into three groups: (a) physical treatments to improve the properties of PF, such as strength, modulus, and elongation; (b) chemical treatments to improve the interfacial properties of the fibre-matrix and the durability of the fibre

Table 2: Physical and mechanical properties of PF.

Group	Fibres	Density (g/cm^3)	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)	Ref.
Bast	Abaca	1.5	400	12	3–10	[3]
	Flax	1.5	345–1035	27.6	2.7–3.2	[3]
	Jute	1.3	393–773	26.5	1.5–1.8	[3]
	Hemp	1.48	690	70	1.6	[3]
	Ramie	1.5	560	24.5	2.5	[3]
	Kenaf	1.45	930	53	1.6	[46]
	Roselle	0.75–0.8	170–350	17	5–8	[47]
Leaf	Sisal	1.5	511–635	9.4–22	1.5	[3]
	Curaua	1.4	500–1150	11.8	1.4	[48]
	Pineapple	0.8–1.6	400–627	1.44	0.8–1.6	[3]
	Poplar	0.25–0.39	—	2.9–8.4	—	[49]
	Banana	0.75–0.95	180–430	23	3.36	[47]
Fruits	Coir	1.2	593	4–6	4.4	[50]
	Oil palm	0.7–1.55	248	3.2	0.7–1.55	[3]
Seed	Cotton	1.51	400	12	3–10	[5]
	Kapok	1.47	45–64	1.73–2.55	2–4	[18]
Grass	Sea grass	—	453–692	3.1–3.7	13–26.6	[18]
	Bamboo	0.6–1.1	140–230	11–17	—	[3]
	Bagasse	1.25	222–290	27.1	1.1	[3]

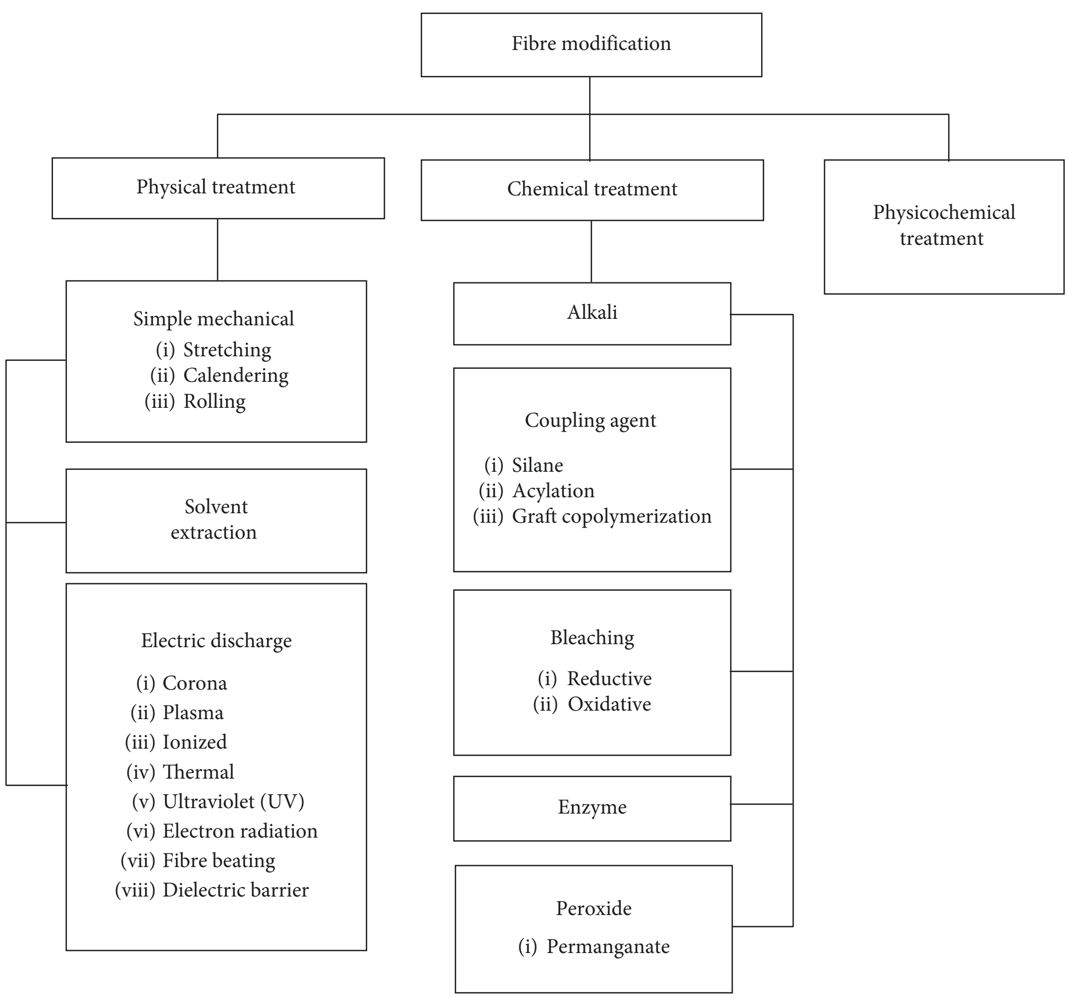

Figure 4: Fibre modification method.

in cement-based composites; and (c) physicochemical treatments that provide clean and fine PF or fibrils that have very high cellulose content [62–64].

8. Physical Methods

Physical methods used to treat PF can effectively change structural and surface characteristics, improving thermal properties and influencing the mechanical bonding of the composites without changing the chemical composition of the PF [55, 56]. These modification methods are implemented on PF for (1) separation of the fibre bundles into individual filaments and (2) improvement of fibre surfaces for composite applications [65], and they can be divided into three main treatments: (1) mechanical treatment, (2) solvent extraction treatment, and (3) electric discharge treatment as shown in Figure 4. The advantages and disadvantages of fibre physical treatments are summarized in Table 3. From Table 3, it can be concluded that each physical treatment offered different benefits in terms of increment in mechanical properties and surface area, high crystallinity, and improved durability of the treated PF.

8.1. Simple Mechanical. Field of the application of different types of physical methods used to treat PF is constantly increasing and can be divided into simple mechanical, solvent extraction, and electrical discharge methods. Simple mechanical treatment such as stretching, calendering, rolling, or formation are conventional mechanical methods for surface treatment of long PF that influence the bonding of fibres with the polymer matrix. Stretching process has the potential to give a maximum tensile strength but the process also can trigger elongation in which the PF could glide over one another during stretching resulting in elongation and extra extension [89]. Calendering process with the applied pressure by calender rollers converts the PF into uniform continuous sheet that can be trimmed and fitted into desired mould. Gupta and Gupta [90] stated that the calendering process has fulfilled the objectives of improvement in the surface smoothness and density of PF. Thus, the pore size has been reduced and larger particles are held back into PF sheet. Rolling and swaging are used to yield PF bundle separation and the rolling effect resulting in enhancement of dispersability and adhesion with polymeric matrices [91].

The conventional treatments can reduce the potential loss to the fibres and increase the surface area for fibre-matrix interaction in the composite [92]. This contrasts with Varshney and Naithani [19], who found that fibres tend to tangle and lead to high energy consumption during treatment. However, the PF chemical structure will not change through the entire process and will improve the performance of the composite as reinforcement [93]. This finding was later proved by Rana et al. [94] that the fracture energy of composites using PF was considerably enhanced though it did not give significant influence on strength and stiffness.

8.2. Solvent Extraction. Solvent extraction or partitioning is the easiest method using mechanical fractionation that can increase the surface area and remove soluble impurities for short PF and fillers [95]. Solvent extraction method proved that lignocellulosic fibres can be separated from PF sources by selective solvent action, obtaining fibres with high content of cellulose. This treatment is an effective method to separate a compound based on the solubility of blended water with an organic solvent [96]. However, this treatment was not widely used because PF will encounter degradation due to a decreasing fibre aspect ratio. In addition, during the solvent extraction process, hazardous steam formed and contaminated the water by leaching into ecosystems, which is harmful to the environment [64]. According to Płotka-Wasylka et al. [72], many new bioderived solvents have been discovered lately but due to some specific requirements for solvents to be used for this application, apparently, not all of it can be used.

8.3. Electric Discharge. The purpose of electric discharge of this treatment is to separate cellulose, increase the melt viscosity, and improve the mechanical properties of PF [19]. Electric discharge is an appropriate treatment method to improve the compatibility between hydrophilic fibre and matrix through roughening of the fibre surface and structure [19]. In contrast with solvent extraction, the electric discharge method has a low impact on the environment [64]. Thermal treatment is the most popular electrical discharge method to change the physical properties of the material and preserve the chemical composition of the PF [97]. Heating PF to temperatures between 100 and 200°C for various durations will separate the lignocellulose fibre bundles into single filaments due to dry-up [98]. Other noncellulose/chemical constituents with lower glass transition temperatures or similar with lignin will release or depolymerize from the fibre bundles [99]. Rong et al. [100] also recorded that the crystallinity of PF is increased when exposed to lower temperature ranges due to fibre stiffness and improved physical adhesion between fibre-matrix. The exact temperature for PF to increase in crystallinity has been found at 150°C and concluded that thermal treatment resulted in higher strength and modulus compared to chemical treatments [101].

Another way of fibre treatment with electrical discharge methods is plasma treatment and it is very effective in substrate surface activation for PF. The plasma treatment is a chosen method to limit the use of chemicals for surface treatments due to increasing concern for environmental pollution [102]. This treatment is functional for optimizing the fibre-matrix interface of polymer composites, and it was an effective and stable treatment to modify the surface of PF [103] without using a chemical solvent. This conventional method only modifies the outer surface layers resulting in significant changes of PF surface morphology [104] with improved wettability.

However, it was found that plasma-treated PF generated lower strength value due to the degradation of the PF after the treatment and is not advisable to be used as reinforcement in composites [22].

Corona treatment is a type of atmospheric plasma technique along with dielectric barrier. Corona surface

Table 3: Advantages and disadvantages of fibre physical treatments.

Treatment	Advantage	Disadvantage	Remarks	Ref.
Stretching	Better load distribution in composite as a result of a reduction of fibre rigidity and density	High heating rate during stretching can generate shrinkage on the fibre surface	Heat treatment will improve the fibre strength and develop high tensile modulus	[66, 67]
Calendering	Enhancement of surface area available for matrix interaction by yielding bundle separation of long fibres	Fibres exposed to potential damage during the process	Fibres became tightly packed, and the filtration efficiency is increased by regulating density and permeability	[68, 69]
Rolling	Fibres became plasticized and will enhance the performance of the composite when added into the matrix	Longer processing will decrease fibre performance and disallow matrix interface benefits	Surface and structural properties of the lignocelluloses fibre were partially changed	[70, 71]
Solvent extraction	High fibre cellulose content was extracted from plants due to removal of impurities	Discharge from the solvent extraction process is harmful to the ecosystems and produces environmental pollution	New renewable and biodegradable bioderived solvents are of great interest and can contribute to an environmentally sustainable solvent extraction	[72, 73]
Corona treatment	Fibre wettability improved through fibre surface polarity and compatibility between hydrophilic fibres and the hydrophobic matrix	Surface ablation and etching can reduce fibre firmness	Usable as standalone surface treatment or early treatment to activate cellulose for chemical modification such as grafting	[74, 75]
Plasma treatment	Simple process without any pollution to modify the surface of NF without altering the bulk properties of the fibres and increase in the wettability of the fibre-matrix interface	Degradation and changes on the fibre surface occur from an etching mechanism that generates pits on the fibre surface area exploiting the plasma properties	PF have stronger interaction with the matrix and enhanced the mechanical interlocking of the fibre-matrix in composites	[76, 77]
Ionized air treatments	Altering the NF structure by separating the fibre bundles through removal of impurities from fibre surfaces	Mechanical properties and the performance of PF as a reinforcement were affected by longer ionized air treatment	Increases the wettability of fibre, but the interfacial property enhancements are lower compared to chemical treatments	[24, 78]
Thermal treatment	Improved thermal stability, crystallinity, and physical and mechanical properties of the NF	Longer thermal exposure decreases the moisture content and changes the physical and chemical composition of the fibre	The combination of chemical and thermal treatment can increase the initial strength and improve the durability of fibre	[79, 80]
Steam explosion	Environmentally friendly and low cost	Does not have much influence on fibre strength, crystallinity, or thermal stability	The temperature is varied between 120–220°C with a variation in time period from 30 min to 2 h	[81, 82]
Ultraviolet (UV)	Polarity of the fibre surfaces, wettability, and fibre-matrix interfacial along with mechanical properties of biocomposites are increased	Fibre strength decreased	Surface oxidation improved the mechanical properties and adhesion between the fibre-matrix compared to traditional electrochemical treatments	[83, 84]
Electron radiation	Improves the interfacial bonding of NF and matrix due to free radicals, which ensure crosslinking between the fibre-matrix	Degradation of high cellulose content under the effect of irradiation will reduce the maximum thermal decomposition temperature	Development of composite materials with good mechanical properties and morphology but lack of durability	[25, 85]
Fibre beating	An effective treatment to mitigate the fibre degradation in composites related to accelerating ageing conditions	Decrease the average fibre length, curling, and tensile strength	Improvement in terms of durability but lower mechanical strength due to defibrillation	[26, 86]
Dielectric barrier	Increased the incorporation of fibres and polymerization into the matrix	Fibre strength decreases due to primary decomposition and short decolourization of low energy consumption and time	Surface roughness increases and leads to improvement in wettability due to changes in fibre surface microstructure but a reduction in tensile strength	[87, 88]

treatment uses low-temperature corona discharge plasma to transmit changes in fibres properties and alter the surface characteristic of PF [74]. This method is an electrical discharge applied on a surface energy of PF at or near atmospheric pressure using electric current that changes the surface energy of the cellulose fibres [105]. Corona treatment are not widely used due to the difficulties to use on three-dimensional fibrous materials [106], inherent complexity, and insufficient number of investigations dedicated to understanding their behaviour on PF [65]. However, these physical methods offers many advantages such as no need for specific conditions during modification, low-cost process with low energy consumption, and high volume of material can be applied in large scale during the treatment and can benefit the industrial production line of PF [107].

Whilst, the dielectric barrier through the plasma process provides a nonthermal, nonequilibrium plasma and modifies surface properties of fibres at atmospheric pressure [108] and similar with corona treatment process. The free electrons in the plasma discharge are heated up to 10,000–100,000 K, while the gas itself can be kept at moderate temperatures between room temperature and 100°C [109]. High-energy electrons are generated through collisions during discharge and able to produce radicals and electronically excited particles efficiently. The treatment process can promote the surface activation of the PF, but the discharge is not completely uniform and has a short duration [110]. Although the ionized air treatment using electric discharge is a similar method to corona discharge, the treatment will minimize the aggregation of fibre bundles [111] In addition, the separation of the fibre bundles and penetration of the ionized air through the PF increases the PF loading and matrix interactions at the interface during the treatment [112] and revealed significant changes of the surface roughness after the treatment, which could also enhance the PF wettability.

The steam explosion treatment is one of the most efficient methods for removal of hemicelluloses fibres with rapid process in addition to alkaline extraction [113]. The steam explosion methods require less energy consumption, hazardous chemical, human toxicity, and environmental impact in comparison with the alkaline treatment in order to gain higher fibre yielding [114]. This method involves in treating PF with saturated steam at various temperatures and reaction times, activated by shear force generated from moisture expansion and acetic acid form the hydrolysis of acetyl groups in hemicellulose obtained from PF [115]. This type of treatment physically shatters the fibres from within to release impurities and form fibrils without changes in chemical composition. Degradation may occur in some cellulose fibres depending on the gradation of treatment, time, and temperature [116], and the PF are not significantly damaged due to greater strength, crystal structure, and limited water uptake compared with the rest of the PF.

The electron radiation method is a surface modification treatment applied to PF to develop interactions between PF and the polymeric matrix for property improvement [117]. The established reaction depends on (1) type of fibre/polymer, (2) additives, (3) temperature, (4) pressure, (5) dose, (6) dose rate, (7) morphology, (8) crystallinity, and (9) surface volume ratio [118]. Free radicals on the surface of the PF generated from electron radiation process can initiate grafting polymerization of functional groups and the modified PF can be used in composites to enhance the bonding between fibre-matrix, or used as an independent functional material such as heavy metal ion adsorption and wastewater purification [119]. Hence, the irradiation will significantly transform the structure, reactivity, mechanical properties, and physicochemical properties of cellulose [120].

It was found that fibre beating is a well-established process of physical surface modification in the paper industry that induces better performance of the PF cementitious composite [121]. Beating or refining is a mechanical treatment to develop a controlled amount of smaller fibrils, improve fibre bonding, and develop optimum strength in preparing the pulp for the paper manufacturing process. It is widely used in wood related material but was implemented in PF as well [49]. In general, the three main effects of beating/refining are as follows: (1) internal fibrillation increases the flexibility of fibres by the breakdown of fibre walls into separate lamellae, (2) external fibrillation is described as the creation and/or exposure of fibrils on the surface of the fibres, and (3) the generation of fines from fibres when they are no longer able to sustain compressive and/or shear forces during the treatment [122]. According to Li et al. [123], an optimum degree of beating at 60°SR (degree of beating) leads to higher porosity and a greater extent of fibrillation. The PF fibrillation degree obtained from the beating process enables the control of the microstructure and mechanical properties of PF [124].

The least popular physical method is ultraviolet (UV). UV rays are suitable for textile surface treatment with shorter wavelengths than visible light that ranges from 10 to 400 nm and can trigger chemical reactions with several organic molecules [107], thus, leading to greater effects rather than simple heating effects. It was found that UV treatment resulted in a greater degree of surface oxidation when compared to conventional electrochemical treatment [125]. Bast fibres such as raw hemp, flax/jute, kenaf, abaca, and grey cotton provide good UV protection through natural pigments, lignin, waxes, and pectin that act as UV radiation absorbers [126]. In addition, techniques involving UV has proved to be clean methods and are widely attractive because UV sources are relatively cheap, flexible, and easy to install [127].

9. Chemical Methods

The purpose of chemical treatment is to modify and activate the fibre structure using a hydroxyl group that can change the composition of the material by introducing new elements to interact with the matrix [57]. Using chemical regents for fibre modification will increase the mechanical properties of the fibre and the strength of the fibre-reinforced cement composite and will improve the adhesion between the fibre surface and polymer matrix by reducing the water absorption of the composites. Alkali, coupling agents, bleaching, enzymes, and peroxide are among the chemical treatments that are reviewed here, and the advantages and disadvantages of using these treatments during fibre modifications on PF are presented in Table 4.

9.1. Alkaline. Alkali treatment or mercerization is effectual, low cost, and the most commonly used chemical treatment for PF modification. The treatment is a process to increase the surface roughness by shattering the internal hydrogen bonding that changes surface topography, crystallinity, unit cell structure, moisture absorption, and orientation of fibrils, enhancing the mechanical properties of the fibre [146]. During the treatment, lignin, wax, and oils that conceal the exterior surface of the fibre cell wall will be partly removed, as well as hemicellulose, and that will trigger the cellulose decomposition and expose short length crystallites [147]. Table 5 displays the effect of alkali on the tensile properties of NF. From Table 5, it is obvious that the tensile strength increase correlated with the percentage of alkali used in the treatment, indicating that severe or more than 10% alkali resulted in weakening or damaging the fibre and reduction in fibre tensile strength. A mild alkali added between 6 and 9% will increase the fibres tensile strength approximately 30% compared to untreated fibres.

Several researchers conducted studies to analyse the correlation between sodium hydroxide (NaOH) solution and the mechanical properties of both PF and PF-reinforced composites [151–153]. Their results proved that the thermal and mechanical properties of PF were directly affected by alkali treatment that provides a rough surface topography through removal of natural and artificial impurities. Barreto et al. [154] found that composites containing alkali-treated fibre bundles have better mechanical properties than those with untreated fibre bundles. However, the effectiveness of the alkali treatment of PF can only be increased further by conducting the treatment at elevated temperatures as the heat energy would provide some additional catalytic effect in breaking the hydrogen bonds within the fibrils [155]. In this treatment, the concentration of the alkali solution, operational temperature, temperature treatment time, material strength, and the applied additives are parameters to be considered.

9.2. Coupling Agent. Coupling agents have been developed to improve the interfacial bonding for adhesion that relates to enhancement of the mechanical strength and durability of the fibre-matrix. The coupling agents are compatible with the fibre-matrix in reducing water absorption, eliminating the leaching effect and enhancing the wettability of fibres by the polymer chains [156]. The surface modification procedures applied to PF using coupling agents such as silanes, acetylation, and graft copolymerization are intended to enhance the chemical bonding of the oxide groups on the fibre surface with the polymer molecules that link together the hydrophilic fibres and hydrophobic polymers for excellent composite mechanical properties [157]. This confirms previous findings by Bledzki and Gassan [8] that the chemical treatment due to coupling agents causes impressive improvements in the characteristic values of composites depending on the fibre, matrix, and type of surface treatment used.

9.2.1. Silanes. Silanes are efficient coupling agents and are extensively used to promote adhesion to hydrophilic composites and adhesive formulations, and various types were found to have effectively modified the interface properties of the natural fibre-polymer matrix interface, wood-polypropylene, mineral-filled elastomers, fibre-reinforced epoxies, and phenolic [13]. The chemical structure for silane coupling agents generally consists of $R(4\text{-}n)$-Si-$(R1X)n$, where R is alkoxy, $R1$ is an alkyl bridge connecting a silicon atom, and X is organofunctionality [158]. Nonswelling behaviour, high chemical resistance, and an increase in tensile strength are the outcome of cross links between silanes, treated fibres, and matrix that accelerates the efficacy of composites [159]. Trialkoxysilanes and γ-aminopropyltriethoxysilane (APS) are the types of silanes that are frequently used as coupling agents to reduce the number of hydroxyl groups, forming silanols that are adsorbed on to the fibre-matrix surface [160]. The fibre-matrix interaction depends upon the organofunctionality of silanes and the matrix.

9.2.2. Acylation. Acylation or esterification methods are divided into acetylation (using acetate) and valerylation (using valerate) for plasticizing PF. An acetyl group during acetylation reacts with hydrophilic hydroxyl groups of the fibre to generate esterification that reduces its hydrophilic nature by absorbing moisture from the fibre [161]. As a result, after acetylation, the dimensional stability was improved as well as the dispersion of fibre into polymeric matrices, thus increasing the hydrophobic nature of the fibre due to the substitution of hydroxyl groups with acetyl groups. Generally, alkaline treatment was done before acetylation and has been found to reduce the impact strength and stiffness and improve the interfacial bonding, as well as the dimensional stability and thermal stability and resistance to fungal attack in PF composites. The mechanical and other physical properties of the composite are usually dependent on the fibre content, which also determines the possible amount of coupling agents in the composite [162].

9.2.3. Graft Copolymerization. Graft copolymerization is a prominent cross-linking agent used since 1943 to chemically increase the compatibility of PF or wood with a suitable solution that forms free radicals on the cellulose molecules by reaction through selected ions with hydrophobic matrices [163]. The method involves the grafting of various vinyl monomers, acrylonitrile, and methyl methacrylate [164]. Through this method, functional groups that can interact with cellulose or other constituents of the natural fibres are grafted into the same polymers or polymers that have a resemblance to a given matrix [56]. Thus, the grafted systems are able to act as bridges to minimize the mismatches in polarity between the hydrophilic fibres and hydrophobic matrices. The functional groups that are actively used

TABLE 4: Advantages and disadvantages of fibre chemical treatments.

Treatment	Advantage	Disadvantage	Remarks	Ref.
Alkaline	Rough fibre surfaces provide better mechanical interlocking and stronger interfacial strength between the fibre-matrix	More than 6% increment of alkaline concentration and longer than 24-hour soaking periods damage the fibres and reduce tensile strength	The types and concentration of the alkaline solution, time of treatment, and the temperature used for modification affected the efficiency	[128, 129]
Silanes	Increases toughness and reduces water adsorption of the fibres resulting in hydrophobic composites	Treated fibre tensile strength decreased due to cellulose microfibrils receiving less support against tensile loading	Silane treatments could be more effective when combined with physical treatment	[130, 131]
Acylation	Increases crystallinity and reduces water adsorption	Increasing the degree of acetylation decreases the mechanical properties due to degradation of cellulose and cracking of fibres	Acylation may not be as effective as alkaline treatments for improving the interfacial interaction between fibres and matrices	[132, 133]
Graft copolymerization	Increases fibre-matrix adhesion, reduces water adsorption, thermal stability, and enhances the mechanical properties of the composites	High initiator concentration, temperature, and fibre loading influence the grafting effect	Maleic anhydride (MA) is the most prominent functional group due to cost, performance, and commercial availability	[134, 135]
Reductive bleaching	Fibres are highly hydrophobic and bright	Not as efficient as oxidative bleaching	Bleaching effects have a positive reaction with a 1–2% dosage of reductive bleaching agents and higher than 60°C temperature	[136, 137]
Oxidative bleaching	No separate pretreatment is needed due to the combined effect of bleaching and cleaning with fibre quality intact	Expensive treatment requires high temperatures, and fibre-coloured compounds are destroyed and cannot reform due to the permanent bleaching process	Combination treatment with coupling agents is needed to modify the wet-out and interfacial bonding	[138, 139]
Enzyme	Effectively used to produce homogenous fibre surfaces with improved thermal properties by removal of the hygroscopic pectin and hemicellulose content	Differences in enzyme quality including contamination of enzymes during preparations with others of varying specificity will require careful and detailed analysis	Variation types of fibre and enzymes have different reactivity to the targeted components because of geometrical shapes	[140, 141]
Peroxide	Increases crystallinity, thermal stability, and mechanical performance	Possibility of forming mechanical and chemical bonding at the fibre surface is mainly dependent on the surface morphology and chemical composition of the fibres	It may facilitate both mechanical interlocking and the bonding reaction due to the exposure of the hydroxyl groups to the chemical	[142, 143]
Permanganate	Rough fibre surfaces provide better mechanical interlocking and stronger interfacial strength between the fibre-matrix	The use of hazardous chemicals causes environmental pollution	4% is the maximum dosage of the compound to yield tensile strength	[144, 145]

include methyl groups, isocyanates, triazine, benzoylation, maleic anhydride, and organosilanes. However, the best functional group for compatibility by graft copolymerization is maleic anhydride (MA) due to cost, performance, and commercial availability [64].

9.3. Bleaching. Bleaching treatments including peroxide bleaching, alkali and enzyme treatments, and biobleaching can improve the appearance of bast fibres. The purpose of the bleaching was to isolate individual microfibres through dissolving and eliminating lignin and hemicellulose from the matrix surrounding the cellulose microfibrils [165]. Bleaching treatments are environmentally safe, and most of the PF used for textile fabrics are bleached with hydrogen peroxide. The temperature and pH must be controlled when using hydrogen peroxide because high temperature and alkalinity will damage the fibres. The effect of bleaching on PF may be seen visually as a change in appearance and other aesthetic properties, but it may result in deliberate loss of fibre tensile strength [166] due to the loss of lignin as a cementing material and result in a decrease in the tensile

Table 5: Alkali effect on tensile properties of PF.

Fibres	Treatment: Chemical	Treatment: Physical	Tensile strength (MPa)			References
Kenaf	NaOH soaked for 24 h	Oven dried at 220°C (10 h)	Untreated	—	289	[97]
			Treated	5%	355	
				10%	261	
				15%	213	
Kenaf	NaOH soaked for 24 h	Oven dried at 80°C (10 h)	Untreated	—	16.8	[148]
			Treated	3%	17.5	
				6%	17.6	
				9%	16.8	
Abaca	NaOH soaked for 0.5 h	Oven dried at 80°C (2 h)	Untreated	—	755	[146]
			Treated	5%	847	
				10%	840	
				15%	840	
Mulberry	NaOH soaked for 0.5 h	Dried at room temperature (48 h)	Untreated	—	463.84	[149]
			Treated	5%	605.61	
				10%	532.24	
				15%	519.30	
Date palm	NaOH soaked for 1 h	Oven dried at 80°C (24 h)	Untreated	—	350	[150]
			Treated	2%	370	
				5%	460	
				10%	250	

strength of the composite. Generally, bleaching was divided into reductive and oxidative bleaching.

9.3.1. Reductive Bleaching. Reductive bleaching is a temporary process performed on protein fibres containing high cellulose using sodium dithionite ($Na_2S_2O_4$) associated with the reduction of chromophores (coloured fibres) to leucochromophores (uncoloured fibres). Although it is stable under most conditions, it will decompose in hot water and acid solutions. Due to significant losses in durability, reduction bleaching is rarely explored for surface modification of PF for composite purposes [64]. This enables oxidative bleaching to be frequently used treatment method using sodium hypochlorite (NaClO), hydrogen peroxide (H_2O_2), or sodium chlorite ($NaClO_2$). NaClO attacked the hydroxyl groups in the lignin to form aldehyde groups (CHO) that can reduce the lignin content, whereas existing aldehyde groups will cause the degradation of cellulose in NF. However, $NaClO_2$ bleaching can reduce lignin and pectin with minimal amounts of cellulose degradation at a reasonable cost compared to NaClO and H_2O_2. However, the bleaching process using H_2O_2 is gradually replacing other oxidative bleaching processes due to its environmental friendliness, although the chemical cost is higher than many other bleaching agents with similar capabilities [167].

9.4. Enzymes. Enzyme or biological modification is an effective method for fibre surface treatment through removal of lignin and hemicellulose, and it requires lower energy input. The fungus or bacteria release enzymes that can deteriorate or remove the pectinase glue that bonds the fibre bundles to release the cellulosic fibres. The treatment resulted in an increase in the surface hydrophilic interface between the fibre-matrix and improved the mechanical properties of the composites. Yi et al. [168] found that the crystallinity and thermal properties improved after separating hemp fibre bundles using enzymes into individual bundles. It also influences the structure, chemical composition, final fibre quality, and properties of the fibres [169]. Enzyme treatment is progressively popular, with benefits related to environmental friendliness [170], and the reaction catalysed from this treatment is very specific, focusing on the particular performance required. It can be recycled after each use [62], but the discharge will affect the environment [171].

9.5. Peroxide. Peroxide treatment is a process similar to the initiation step of the free-radical polymerization, which induces adhesion in cellulose fibre-reinforced thermoplastic composites. Chemicals that can be used as initiators of the polymerization include benzoyl peroxide (BPO) and dicumyl peroxide (DCP) [172]. An alkali pretreatment is required before the peroxide treatment to separate wax, hemicellulose, and lignin. Since the peroxide treatment is the first step of free-radical polymerization, it works best as an interface modifier when the curing mechanism of the matrix polymer is free-radical polymerization, whilst permanganate is not technically a peroxide treatment, but it has a similar treatment method. The permanganate treatment uses the oxidation property of $KMnO_4$ to reduce the hydrophilic nature of cellulosic fibres. The degree of reduction in hydrophobicity is found to grow with increasing $KMnO_4$ concentration. Peroxide treatments can significantly reduce the water absorption of natural fibres/fillers, which in turn can improve the interfacial adhesion between fibres/fillers and hydrophobic polymer matrices [173]. Meanwhile, the tensile properties of peroxide-treated natural fibre-reinforced thermoplastic composites showed clear improvement. On the other hand, the rate of peroxide decomposition can also negatively affect the mechanical properties of treated composites.

10. Physicochemical Treatments

The combination of chemical and physical treatments is known as a physicochemical treatment and combines physical treatments with chemical treatments to produce support to the chemical reactions and improved separation of fibre bundles [174]. These types of treatments provide clean and fine natural fibres or fibrils with high cellulose content. The mechanical properties of these fine fibres are close to those of pure cellulose fibres, which can significantly improve the appearance and mechanical properties of PF [64].

11. Summary

The combination of alkali and thermal treatments for surface modification of PF is popular among the various combinations of chemical and physical techniques that provide better adhesion between the fibre-matrix. Chemical treatment plays a major role in the interfacial properties of the fibres, and thermal treatment improves the surface area exposed for treatment and matrix interaction by assisting in the separation of the fibre bundles. Thermal treatments also increase the hydrophobicity of lignocellulose fibres, crystallinity, and dimensional stability. While NaOH is the most common alkaline solution used in the physicochemical treatment, the use of sodium bicarbonate ($NaHCO_3$) started getting attention lately due to its similar effect on PF and cost effectiveness. High temperatures have been reported ranging from 100°C to 200°C. However, low temperatures below 100°C had a significant effect on the adhesive properties of PF and showed that this parameter affects the fibres as reinforcements. In summary, the combination of both alkalization and thermal treatment provides benefits to improve the reinforcement potential of cellulosic fibres, but treatment parameters such as concentration, time, and temperature are essential to achieve the optimum efficiency of the fibre in biocomposites.

12. Conclusions and Recommendations

The ability of surface modification treatment for improving fibre surface properties has generated interest in employing PF with composites. The aim of using PF in composite materials can be carried out following a variety of physicochemical methods that can be applied, leading to the enhancement of interface bonding between the cellulose surface and matrix. PF is increasing in demand for biocomposites because of its availability, environmental friendliness, and consistent quality of a wide range of fibres provided that the fibres must be treated to stimulate their properties to be used as reinforcements in cementitious composites. A strong fibre-matrix interface is important, and fibre surface treatments can initially improve the interfacial adhesion between the fibre-matrix and enhance good mechanical properties of composites.

Physicochemical surface treatments provide good compatibility and interface bonding in the modification of PF. The effort to find the most suitable physicochemical combination for PF surface modification must be continuous as the reinforcement fibre is the major contributor to the mechanical properties of the composite. When the effects of various fibre treatments were put into consideration for selecting suitable fibres, it can be seen that physical or chemical surface modification treatment alone can be applied depending on what properties we want to achieve. However, it is suggested to use physicochemical treatment for potential excessive changes in fibre surface properties with improvement in the compatibility of different matrices and also to overcome the hydrophilic nature of PF surface properties. Moreover, the surface free energy, the mechanical interlocking at the interface of the composite, and the mechanical properties of the composites also increase due to improvements in fibre-matrix adhesion related to various types of physical and chemical modifications.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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