REVIEW PAPER

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Treatment methods for plant fibers for use as reinforcement in cement-based materials

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Abstract The use of natural fibers in cement-based building materials has become an emerging field in the building and construction industry. Natural plant fibers are green, sustainable, and low-cost renewable resources that can be found in all parts of the world. Besides their biodegradability, studies have also established that the natural fibers also positively impacted the mechanical properties of the resulting composites. However, their utilization has also presented some challenges due to their high-water absorption, limited tensile strength, and open surface morphology that is exposed to cement hydration products and microbial degradation. Also, the fibercement matrix bonding and adhesion is another challenge limiting natural fiber utilization. Successful research on these challenges has been conducted and improvements were observed for the fiber-matrix adhesion and durability. Thus, herein we critically review the articles, discuss the science behind the treatment methods and suggest some intriguing issues for further investigation.

 $\label{eq:keywords} \begin{array}{ll} \mbox{Natural fibers} \cdot \mbox{Cement composite} \cdot \mbox{Fiber} \\ \mbox{treatment} \cdot \mbox{Fiber-cement matrix} \end{array}$

Introduction

Cement-based materials are the most commonly used building materials today (Wei and Meyer 2014a) and it was once said that human beings are in the world of cement-based materials (Lan 2005). However, the brittleness of cement-based materials, the tendency to crack and shrink, as well as the low tensile strength are serious drawbacks of cement-based structures (Turatsinze and Garros 2008). It was reported that when the strength of the cement-based material increases, it resulted in an increase in brittleness, and as such cracks are caused which usually cause serious damage to the cement-based structures (Turatsinze and Garros 2008). To overcome this issue, the utilization of synthetic fibers for the reinforcement of the cementbased materials to produce a cement-fiber composite with enhanced properties and durability have been studied extensively and the findings of the studies were very positive (Izaguirre et al. 2011). Also, study has reported the use of mineral fibers as reinforcement in cement composites (Khandelwal and Rhee 2020). However, it has been established thatsome mineral fibers such as asbestos represent a health hazard and they are classified as cancer-causing chemical/physical toxicants (Finkelstein 2019). The fibers mentioned above are expensive, non-biodegradable and their production was reported to result in environmental pollution (Saikia and De Brito 2012).

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The aforementioned reason attracted the attention of researchers towards investigating the natural fibers for the reinforcement of the cement-based materials to produce the composites (Ferreira et al. 2015). The natural fibers were employed because they are relatively cheap, green, and environmentally friendly with their utilization resulting in the production of relatively lightweight composite with high tensile strength and Young's modulus. However, challenges of natural fiber utilization such as high water absorption capacity, poor interfacial adhesion (Bilba and Ouensanga 1996) were reported to negatively affect both the fiber and the resulting composite in terms of durability and performance. Interestingly, various research groups have extensively carried out studies to investigate some treatment methods towards tackling the challenges, and positive results achieved were published by the different groups. However, despite these efforts, these published reports have never been reviewed to the best of our knowledge. Thus, herein some of the published articles are critically reviewed, their major findings were discussed for future improvement suggested in the area. These will undoubtedly provide the basis for further studies and sustainable utilization of the natural fiber in cementbased structures

Treatment of the natural fibers for cement composites

To improve the durability and properties of the natural fibers, the various treatment efforts were classified into physical, chemical, and mixed methods as discussed below.

Physical treatment methods

The physical treatments methods ensure that the chemical composition of the natural fibers is not altered, but the physical state and surface morphology are modified. The physical treatment has affected the surface morphology, structure of natural fibers, and reinforced dimensional stability of the fibers, thus greatly improved the adhesion of the fibers to the matrix. Also, water absorption capacity of the fiber was substantially reduced (Ferreira et al. 2015), possibly due to the changes in surface morphology and dimensional stability that have affected the

porosity of the fiber cell walls. Details of the physical methods steam blasting, heat treatment, plasma, and hornification treatments are discussed below.

Steam blasting treatment

The steam blasting treatment is carried out by applying high pressure to the fiber which causes the fiber to expand and rupture, resulting in profound changes in the fiber structure (Claramunt et al. 2016). Firstly, the fibers are fibrillated which forms a relatively rough surface with an increased surface area of the plant fibers. Furthermore, the treatment method has also facilitated the separation of the various components such as lignin, pectin, and hemicelluloses. After removing small molecules, the surface area of the fiber increases, thereby improving contact with the composite material.

Heat treatment

During heat treatment, the moisture content in the natural fibers is significantly reduced with the application of heat thereby reducing the probability of voids and internal stresses generated inside the cementbased materials (Wei and Meyer 2014a). By the thermal process, the lignin is rearranged and hemicelluloses are degraded (Wei and Meyer 2014a). Moreover, the hydroxyl content of the fiber surface is usually reduced, which causes an improvement in the bond strength between the fiber and the cement matrix (Wei and Meyer 2014a). The heat treatment does not affect the general composition of the fiber except the slight change in the number of hemicelluloses, cellulose, and lignin that was reported to occur (Onésippe et al. 2010).

Plasma treatment

In cold plasma treatment, the cold plasma is generated from a pure organic gas (e.g. methane) or mixed with other gases resulting in a collision between energetic electrons and gas molecules which forms a series of reactive fragments. The reactive fragments are recombined to give rise to a solid polymeric material that is deposited on the surface to be treated (Kim et al. 2006). It provides an effective and reliable mechanism to alter the surface properties of the material without affecting the overall properties of the treated fiber (Carlsson and Stroem 1991).Due to the physical process involved, the treatment does not use water and chemicals so it is fast and environmentally friendly without resulting in any pollution.

Hornification treatment

In the hornification treatment, natural fibers are placed in water to let them attain their maximum water absorption capacity before placing them in an oven to dry at a suitable temperature and the process is repeated about ten times. It has been found that some microfibers such as soft kraft pulp and cotton linter fiber use fewer cycles of drying and rewetting to achieve hornification (Claramunt et al. 2011; Ballesteros et al. 2019). This treatment effectively promotes the change in fiber microstructure, resulting in dimensional stability (Lima et al. 2012). The treatment process also leads to irreversible shrinkage of the fiber cell cavity and the formation of internal hydrogen bonds which make the fiber structure firmer, reduce its hydrophilicity, increase the stiffness, and ultimately enhance the fiber strength by the alternate swelling and drying (Ferreira et al. 2015). At the same time, the process promotes a reduction in fiber volume changes, thereby enhancing the bonding between the fiber and the cement matrix (Claramunt et al. 2011).

Therefore, it could be observed that generally the physical treatment methods of the natural fiber usually resulted in the physical structure of the natural fiber surface which could be noticed in the fiber's morphology, dimensions, shape, and texture as shown in Fig. 1, such as rearrangement of cellulose and hemicellulose. The possible reasons are that they could be attributed to these physical changes, with inducements of high pressure or temperature involved in the methods and the presence of water in the natural fibers which can be drained out by squeezing the fiber because of the force application. However, a possible change in chemical compositions of the natural fibers due to the physical treatment methods that may also occur remains a puzzle. Also, a comparative study of the various physical methods discussed considering their efficiency and energy expenditure is presently lacking.

Chemical treatment methods

Chemical treatments can be used to improve natural fibers. The method exposes more reactive groups on the surface of the fiber which promotes efficient coupling to the substrate resulting in the enhancement of the mechanical properties of the composite (Dash et al. 2000). It has been established that the chemical treatment methods change both the chemical composition and structural features of the natural fiber (Snoeck et al. 2015). Chemical treatment methods, such as alkaline, silane, acetylation, benzoyl peroxide, acrylic, and polymer graft co-polymerization, are discussed below.

Alkaline treatment

Sodium hydroxide (NaOH) treatment of natural plant fibers is widely used to modify the molecular structure of natural fibers. For instance, as in the case of the chemical composition of the lignocellulosic natural fibers presented in Fig. 2, the alkaline treatment changes the ordered orientation of highly deposited crystalline cellulose (Fig. 3a which is a linear semicrystalline phase with the main structural component of plant fibers (Pickering et al. 2016). The main framework component of fiber structure responsible for providing fiber strength, stiffness, and structural

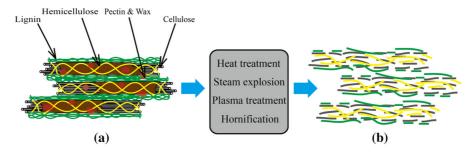
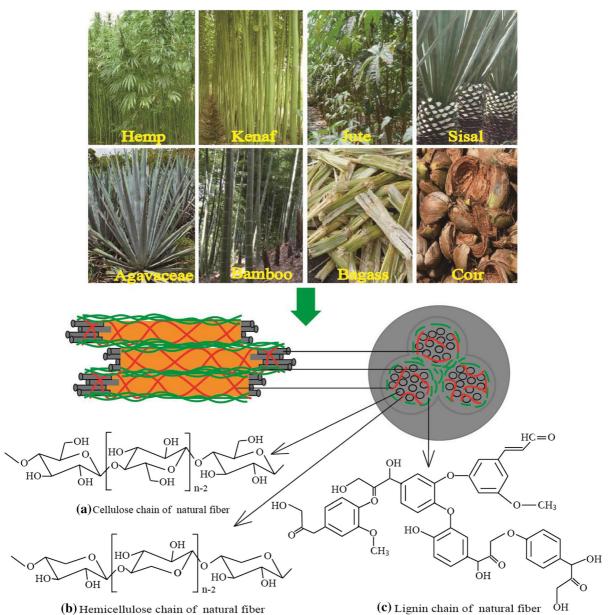


Fig. 1 Change in the physical structure of natural fiber due to physical treatment methods: a before treatment; b after treatment

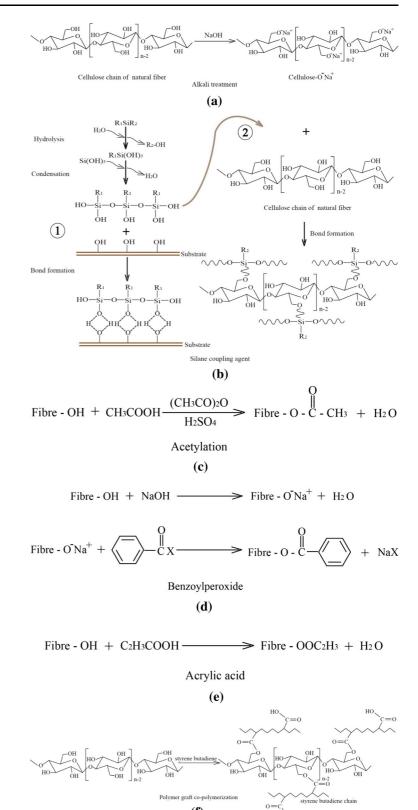






stability (Sedan et al. 2008). This provides more penetration pathways for chemical substances (Kabir et al. 2012), and then the base-sensitive hydroxyl groups (–OH) present in the molecule are destroyed. The fiber composition is also altered and its crystallinity is increased by removing the amorphous constituents which are the binding phase of the cellulose (hemicelluloses Fig. 2b and lignin Fig. 2c (Morán et al. 2008). The treatment also depolymerizes the cellulose to form short chains with increasing the surface roughness of the fibers (Li et al. 2007). By these processes, there is a possible increase in tensile strength of natural plant fibers, probably due to the increase in the cellulose crystallinity (Sawpan et al. 2011).

Fig. 3 Connection mechanism of chemical treatment of plant fibers: a alkali treatment, b silane coupling agent, c acetylation, d benzoyl peroxide, e acrylic acid, f polymer graft copolymerization



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Silane treatment

In the silane treatment, a silane coupling agent, a bifunctional compound with silicon atoms bonded to different functional groups, is usually used. One end of the compound interacts with the matrix while the other reacts with the hydrophilic fibers (Sepe et al. 2018). The silane forms a chemical bond between the surface of the fiber and the substrate through the siloxane bridge, which undergoes three stages namely, hydrolysis, condensation, and bond formation during the fiber treatment process (Kabir et al. 2012). The hydrolyzable alkoxy group in silanol is formed in the presence of moisture (Sreekala et al. 2000). During the condensation process, one end of the silanol reacts with the cellulose hydroxyl group (Si-O-cellulose) as shown in Fig. 3b (Sreekala et al. 2000), and the other terminal functional group reacts with the matrix (Simatrix). Therefore, the adhesion of the fiber with the matrix got improved and the properties of the composite are stabilized (Li et al. 2007). Natural fibers have micropores on their surface, so the silane coupling agent on the surface of the fiber usually penetrates into the pores and forms a mechanical interlocking structure (Kabir et al. 2012).

Acetylation treatment

Acetylation is a process that involves the esterification of natural fibers. Generally, acetic acid and acetic anhydride are not sufficiently reactive with the fibers (Kabir et al. 2012). To accelerate the reaction, the fiber is first soaked in acetic acid, and then sulfuric acid was used as a catalyst and treated with acetic anhydride (Snoeck et al. 2015). The reaction of acetyl (CH₃CO–) with the hydrophilic hydroxyl (–OH) of the fiber as shown in Fig. 3c reduces the hydrophilicity of the composite (Sreekala et al. 2000). This treatment provides a rough surface morphology that allows the fibers to be mechanically interlocked with the substrate (Li et al. 2007).

Benzoyl peroxide treatment

In benzoyl peroxide treatment, alkali-treated plant fibers expose more active hydroxyl groups (–OH) on their surface (Kabir et al. 2012) as shown in Fig. 3d. The free radicals of the peroxide react with the hydroxyl groups on the fiber surface, which reduces its hydrophilicity and increases the roughness of the treated fiber, and also increases the tensile strength of the fiber (Snoeck et al. 2015).

Acrylic treatment

In acrylic treatment, the carboxyl group in the acrylic acid forms an ester bond with the cellulose hydroxyl group, which reduces the hydrophilic hydroxyl group in the fiber structure (Narendar and Priya Dasan 2014). This coupling mechanism between the fiber and the matrix enhances the stress transfer capability at the interface, which improves the properties of the composite (Li et al. 2007). The reaction mechanism is depicted in Fig. 3e.

Polymer graft co-polymerization

In this method, graft co-polymerization of synthetic polymers was done on the natural fibers, which is an effective method for surface modification (Uthaman et al. 2006). The most common method is to create an active site on the polymer backbone. In the polymerization or condensation process, the active site may be a free radical or a chemical group, which are grafted with a reactive hydroxyl group on the cellulose (Uthaman et al. 2006) for modification.

Mixed treatment

The mixed treatment method is a process of modification that affects both the surface and even the internal structure of the fiber because of a series of treatments (at least 2 steps) carried out. It usually involves a combination of two different methods to achieve comprehensive modification of the fiber. For example, Lawan et al. (2018) first treated hemp fiber with sodium hydroxide and ACQ, and then separately treated it with coating (Paraffin wax, epoxy acrylate polymer, or engine oil) (Fig. 4).

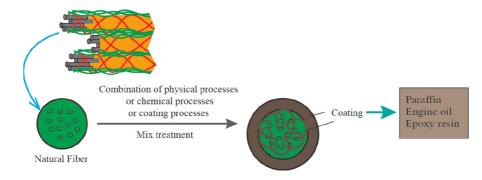


Fig. 4 Mix treatment methods

Effects of treatment methods on the properties of the fibers

Effects of the physical treatments

The various physical treatment methods discussed in "Physical treatment methods" section were found to tremendously affect the natural fibers in several ways as reported from the various studies. For instance, a study reported by Ali and Chouw (2013) established that boiled coconut fiber was found to increase its tensile strength as well as the bond strength between the fibers and the cement matrix. Similarly, the tensile strength of sisal fiber after thermal treatment got improved (Wei and Meyer 2014b). Also, in another physical method, Barra et al. (2015) reported that the results of the pull-out test showed that the sisal fibers with cold plasma treated have higher pull-out load and shear stress than untreated fibers. This reveals that the plasma treatment has resulted to surface modification of the fiber which consequently improved the interfacial adhesion between the fibers and the cement matrix. More so, a noticeable reduction in water absorption capacity as well as the increase in tensile strength achieved with the treated method could be as a result of the physical changes that occurred after the fibers treatment. For instance, it has been found that fibers obtained from slash pine trees (Pinus elliottii) have excellent performance in fiber cement composites when treated by both air-cured (naturally aged) and autoclaved processes (Morton et al. 2010). The rougher surface of the fiber that results as a result of the majority of physical treatment is the mechanism responsible for the increase in bonding between the fiber and the cement matrix (Tonoli et al. 2010).

In another development, Ferreira et al. (2015) opined that hornification of sisal fibers resulted to increase in adhesion between the fiber and cement matrix due to the formation of hydrogen bonds at the surface of the treated fiber. Also, an increase in tensile strength from 453.23 to 474.86 MPa and an appreciable reduction in water absorption were recorded. Jiang et al. (2015) established that spraying sodium silicate solution, pure acrylic polymer emulsion, or organosilicon waterproof emulsion on poplar leaf fiber surface was effective in providing a surface coating with a thin layer of modification substances which was effective in the reduction of water absorption.

Summing up the effects of physical treatment methods as discussed above reveals that the main reason responsible for the improvement of natural fibers properties desired in the cement composite revolves around the change in the physical structure of the natural fiber because of the physical treatment. Table 1 presents the summary of the major findings achieved.

Effects of the chemical treatments

The effects of some chemical treatment methods on the natural fibers were reported by various research groups, among them is the report of Sedan et al. (2008) 6 wt% NaOH treated hemp fiber to improve adhesion between the fiber and matrix and also increase the tensile strength of the fiber. The AlCl₃ treated fibers were reported to remove impurities and waxy from the surface, which have a little effect on the fiber's morphology, and increased the adhesion to the cement matrix. Ming et al. (2017) in their work treated bamboo fibers with 10% NaOH, and they found that the surface area of the fibers increased. The matrix of

Fiber	Specific method	Finding	References
type			
Sisal	Thermal treatment	Tensile strength from 214.65 to 310.54 MPa	Wei and Meyer (2014b)
Sisal	Plasma	Water absorption from 136.64 to 68.39% and Bonding force from 2.56 to 4.64 N	Barra et al. (2015)
Sisal	Hornification	Water absorption from 194.04 to 176.47%, Tensile strength from 453.23 to 474.86 MPa and Bonding force from 3.37 to 3.68 N	Ferreira et al. (2015)
Coconut	Boiled	Increased tensile strength and bond strength between fiber and cement	Ali and Chouw (2013)
Poplar leaf	Spray sodium silicate solution, Pure acrylic polymer emulsion, Organosilicon waterproof emulsion	Reduced water absorption from 214 to 125.6%, 143.4%, 210.4% respectively	Jiang et al. (2015)

 Table 1 Effect of physical treatment methods on natural fiber

cement closely enwrap treated fiber, indicates that better adhesion is formed between the alkali-treated fiber and the cement matrix. Snoeck et al. (2015) also treated hemp fibers with 10% NaOH which increased the binding of fibers with the calcium-rich cement matrix due to the formation of nodules on the fibers; Besides, acetylation makes natural fibers more hydrophobic due to the reaction of hydroxylic groups with acetyl groups (-CH₃CO). The peroxide treatment increased the tensile strength properties of the fibers. Onésippe et al. (2010) treated bagasse fibers with 5 wt% Ca(OH)2 solution, Ca(OH)2 reacts with hydroxyl groups of hemicellulose (cementing material) and it contributes to the destruction of the structure and thereby the fibers split into filaments. This fibrillation increases surface area and then increases the contact and adhesion with the matrix. Ferreira et al. (2015) also treated sisal fibers with Ca(OH)2 which resulted in the partial dissolution of hemicellulose and lignin, thereby destroying the hydrogen bonds in the network structure, which lead to an increased surface roughness as well as improving the ability to bond with the cement matrix. The styrene-butadiene treatment of the sisal fibers revealed an improved bond between the fibers and the Portland cement matrix. At the same time, both chemical treatments reduce water absorption and increase tensile strength. Narendar and Priya Dasan (2014) treated coir pith with sodium hydroxide, dicumyl peroxide, sodium hypochlorite, acrylic acid,

acetic acid, sulfuric acid respectively, and reduced water absorption. The decrease in water absorption of fibers treated with sodium hydroxide is relatively small and is minimal after sulfuric acid treatment. Zukowski et al. (2018) using 1% Ca(OH)₂ solution to impregnate the curauá fibers, removed hemicellulose, and lignin from the fiber wall, and increased the surface roughness by calcium deposition, the deposition of calcium on the surface of the fibers was found to improve the frictional bond between the fibers and the substrate. Overall, it could be concluded that the improvement in the natural fiber properties is mostly the change in the chemical properties of the fibers after the treatment. The summary of the main findings of the chemical treatment methods reviewed are shown in Table 2.

Effects of the mix treatments

Besides the physical and chemical treatment methods, certain groups have investigated the possible usage of more than one method by combining two or more treatment method and their findings have also been reported. Tonoli et al. (2013) reported a combination of heat treatment and impregnation of the kraft pulp fibers which resulted in an improvement in the adhesion of fibers with the cement matrix. Chakraborty et al. (2013) reported the treatment of jute fiber with NaOH solution followed by a carboxylated

Table 2	Effect of	chemical	treatment	methods	of	natural	plant	fiber
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Fiber type	Specific method	Finding	References	
Hemp	NaOH	The improvement in adhesion at the interface and the fiber tensile strength	Sedan et al. (2008)	
	AlCl ₃	Little effect on the fiber's morphology, and increased the adhesion to the cement matrix		
Bamboo	NaOH	Better adhesion is formed between the alkali-treated fiber and the cement matrix	Li et al. (2007)	
Hemp	NaOH	Increasing the combination of natural fibers and calcium- rich cement matrix	Snoeck et al. (2015)	
	Acetylation	More hydrophobic		
	Peroxide	Increased the tensile strength properties of the fiber		
Bagasse	Ca (OH) ₂	Increasing adhesion to the cement matrix	Onésippe et al. (2010)	
Sisal	Ca(OH) ₂ ; styrene butadiene	Water absorption from 194.04 to 168.97%, 156.91% respectively; Tensile strength from 453.23 to 708.70 MPa, 722.07 MPa respectively; and Bonding force from 3.37 to 4.85 N, 5.71 N respectively	Ferreira et al. (2015)	
Curauá	Ca(OH) ₂	Improved the frictional bond between the fibers and the substrate from 1.37 to 2.47 N	Zukowski et al. (2018)	
Coir	Sodium hydroxide, dicumyl peroxide, sodium hypochlorite, acrylic acid, acetic acid, sulfuric acid	Reduced water absorption from 141.65 to 132.89%, 109.84%, 127.46%, 118.92%, 124.68%, 101.71% respectively	Narendar and Priya Dasan (2014)	

styrene-butadiene copolymer emulsion, and found it to enhance the interfacial bonding between the fiber surface and the cement matrix. And reducing water absorption and increasing the tensile strength of the fiber. They found that during pretreatment of jute with the NaOH solution, some of the hydroxyl groups in the fibers reacted with Na⁺ ions to form alkali-exchanged cellulose fibers with O-Na⁺ groups (Sreekala and Thomas 2003). The alkali exchanged cellulose fibers reacted with the -COOH groups of the carboxylated styrene-butadiene copolymer emulsion form an ester bond, and another part of this co-polymer forms bond with the calcium ion of the hydrated cement. Claramunt et al. (2016) reported steam explosion and ironed flax fibers to effectively prevent loss of fiber-matrix adhesion due to fiber size changes and reduce water absorption of the fiber. Similarly, Ferreira et al. (2015) changed the mixed method where they used hornification and styrene-butadiene mixed treatment on sisal fibers. They found that the fibers had the best adhesion to the cement matrix attributing it to large amount of intramolecular and intermolecularly bonded hydroxyl groups of cellulose in the sisal fibers. At the same time, the water absorption is reduced with an increase in the tensile strength of the fiber. Javadian et al. (2016) reported coated bamboo fibers with epoxy resin and adhered sand on the surface. They reported the creation of friction due to the interlocking mechanism between the sand and the concrete aggregate thereby enhancing its bonding. Tian and Zhang (2016) removed the non-cellulosic part of the bagasse fiber in NaOH solution and then covered it with silane, which effectively improved the adhesion of the fibers with the surrounding cementitious matrix. Lawan et al. (2018) treated hemp fibers with NaOH, alkaline copper quaternary with three different coatings which include; paraffin wax, epoxy acrylate polymer, and waste engine oil. Their findings revealed that the treatment was effective for the reduction of water absorption of the fiber and increase in the tensile strength. They also carried out anti-microbial treatment, where the NaOH-treated hemp fibers used were soaked in an alkali copper quaternary solution. It was also found that the treated fibers showed no significant difference between the results obtained after the exposure to the fungus and the results obtained before

Table 3 Effect of Mixed treatment methods of natural plant fiber

exposure to the fungus. It indicated that the mixed treatment inhibited brown rot and white rot fungi, and determined that the decay caused by fungi was mainly on hemicellulose and lignin (Curling et al. 2002), the damage of fiber by brown rot fungi is stronger than that of white rot fungus (Witomski et al. 2016). The effect of mixed treatment methods of natural plant fibers was summarized in Table 3.

Future perspective of natural fiber utilization in cement composites

Presently, findings have so far revealed that the established treatment methods have suggested some solutions by modification of plant fibers, which in most cases improved the adhesion at the interface between the fiber and the cement matrix, which can result in increase in load transfer efficiency between the cement matrix and the fiber. This improvement has in most cases resulted as a result of the increase in the surface area of the fiber which consequently increases the contact area between the fibers and the cement matrix. More so, the simultaneous increase in the surface roughness of the fibers also contributed to the said effects on the fibers and the cement matrix. However, in the case of the chemical treatment

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method, if the fibers and the cement matrix are chemically compatible, higher interfacial compatibility can be achieved, which will cause the fibers to support the stress together with the cement matrix. The main product of silicate cement hydration is calcium silicate sol, it can be considered to use silicon as a transition material to treat plant fibers, silicon as an intermediate can be better to achieve the effect of interface bonding. Treating fibers through a combination of organic and inorganic will be a positive impact on interfacial adhesion, which wouldultimately results to improvement in the durability of the composite.

Plant fiber is a natural green, environmentally friendly reinforcement material that is biodegradable. However, when used as reinforcement for cementbased materials, the biodegradability becomes a disadvantage, because the fiber degradation couldaffect the durability of the fiber, which could also result to failure of the cement-based materials under certain tension and bending loads. In addition to alkaline attack, study has also shown that microbes could to cause significant degradation of natural fiber when used in cement matrix (Lawan et al. 2018). Therefore, studies that will suggest ways of protecting the fiber from both alkaline and microbial attack would be the future research direction in the area.

Conclusion

Results obtained from the various studies conducted reveals that the strength and durability of a cement matrix reinforced with plant fiber depend on the strength and adhesion of the plant fiber used in the matrix. Thus, an effective treatment method and excellent interface compatibility between the plant fiber and cement matrix are needed. Findings have shown that studies mainly focused on simple physical or chemical surface modification treatment of plant fiber, which was still found to have some challenges. Therefore, more studies that focus on the effective treatment method and chemical interaction between the cement matrix and the treated plant fiber needed to be deeply investigated. Also, the effect of each treatment method on degradation that results from a microbial attack should be studied.

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Declarations

Conflict of interest The authors have no conflict of interest with regards to the submission and publication of this article.

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