Chapter

An Overview of Alkali Treatments of Hemp Fibres and Their Effects on the Performance of Polymer Matrix Composites

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Abstract

The alkali treatment is aimed to modify the surface chemistry of natural plant fibres effectively through several factors. This treatment has been carried out at ambient and high temperature. Natural plant fibres treated with alkali have been seen to have benefits such as improved separation of fibres from fibre bundles, improved removal of unwanted surface constituents, increased tensile strength and stiffness, better thermal stability, and enhanced interfacial adhesions compared to other standard treatments. Hemp fibres are an attractive reinforcement for natural plant fibres as they are environmentally friendly compared to other natural plant fibres and exhibit good mechanical properties. This chapter mainly provides an overview of alkali treatments on hemp fibres.

Keywords: alkali treatment, hemp fibres, polymer matrix composites

1. Introduction

Projections of continuing demand for materials across the world is driving the development of more sustainable materials. In addition, the low energy consumption requirements and recyclability found within the spectrum of natural fibre composites have led to increased interest in improving these sustainable materials. Although the use of natural fibre composite materials has been documented in early civilisations, growing environmental concerns coupled with technological advancements have encouraged the expansion of their use in recent times. However, there are still significant issues, including their limited mechanical performance, that limit the ability to compete for future use.

A major area of recent technological development has been that of natural plant fibre composites (NPFCs). The main constituents of NPFCs include plant fibres as the reinforcement and often polymer-based matrix. Natural plant fibres (NPFs) are broadly classified as non-wood fibres and wood fibres, of which non-wood fibres, such as flax, hemp, jute, kenaf and harakeke (Phormium-tenax), are stronger. Among natural plant fibres, hemp fibres are an attractive alternative reinforcement to synthetic fibres due to their favourable mechanical properties as well as availability. **Table 1** represents the specific properties comparison of hemp and glass fibres. As can be seen, hemp fibres have a higher specific Young's modulus. Most of the

Property	Hemp fibres	Glass fibres
Density (g/cm^3)	1.48 - 1.55	2.55
Tensile strength (MPa)	550 - 1110	1400 - 3000
Young's modulus (GPa)	58 - 70	70
Specific tensile strength (MPa per g/cm^3)	370 - 740	800 - 1400
Specific Young's modulus (GPa per g/cm^3)	39-47	29

Table 1.
Specific properties comparison of hemp and glass fibres [1, 2].

interior parts in automobiles are mainly designed for low density and high stiffness [1], and hemp fibres are well-suited for this application [3]. Additionally, compared to other natural fibres, hemp fibres are more valuable for the bio-based economy due to environmental benefits such as being grown without pesticides and a high yield of technical fibres.

Plant fibres are lignocellulosic, and the presence of numerous hydroxyl groups make them hydrophilic in nature [4]. Polymeric matrices, although generally hydrophobic, are preferred for NPFCs due to their low density and ability to process at low temperatures [5]. There are usually limited interactions between the hydrophilic natural fibres and hydrophobic polymer matrices, which commonly leads to their poor mechanical performance [4]. Additionally, a weak fibre-matrix interface increases the moisture uptake of these composites, which affects their long-term performance [6].

Physical treatment methods such as corona, plasma and heat treatment often require highly sophisticated equipment. Therefore, chemical treatments are commonly seen in the literature. The most popular treatment is alkali. The treatment is aimed to modify surface chemistry effectively through several factors. Natural plant fibres treated with alkali have been seen to have benefits such as improved separation of fibres from fibre bundles, improved removal of unwanted surface constituents, increased tensile strength and stiffness, better thermal stability, and enhanced interfacial adhesions compared to other standard treatments.

2. Fibre and matrix selections for composites

2.1 Fibre Selection

It is very important to incorporate high strength reinforcing fibres in order to manufacture high-performance plant fibre composites. The mechanical properties of plant fibres depend upon many factors other than botanical type. These include chemical composition and structure, harvesting time, extraction method, treatment and storage conditions. Among the different types of plant fibres, bast fibres have the highest specific moduli and tensile strengths, which is considered to be mainly due to their higher cellulose content and their cellulose microfibrils aligned more in fibre direction [2]. **Table 2** shows the mechanical properties of some NPFs.

Hemp is one of the most utilised bast fibres. It exhibits high tensile strength ranging between 550 and 1110 MPa, specific Young's moduli ranging between 39 and 47 GPa/gcm^{-3} [2], and also environmentally friendly since it can be grown without pesticides and herbicides [7]. Hemp is being considered as a suitable NPF reinforcement for use in the present research because of its local availability and good mechanical properties.

NPFs	Tensile strength (MPa)	Young's modulus (GPa)	Density (g/cm^3)	Specific tensile strength (MPa/gcm^{-3})	Specific Young's modulus (GPa/gcm^{-3})
Flax	345-1830	27-80	1.5	230-1220	18-53
Hemp	550-1110	58-70	1.5	370-740	39-47
Jute	393-800	10-55	1.3-1.5	300-610	7.1-39
Harakeke	440-990	14-33	1.3	338-761	11-25
Sisal	507-855	9.4-28	1.3-1.5	362-610	6.7-20

Table 2.
Mechanical properties of some of the NPFs [2, 5].

2.2 Industrial hemp fibre

Industrial hemp is the term utilised for hemp grown for industrial use, selected such that it naturally attains a tetrahydrocannabinol (psychoactive chemical) content below 0.6%. It is a fast growing annual plant, which has a height of up to 5 m (1.2-5 m) and stem diameter between 4 and 20mm [5]. It has separate male plants and female plants. Male plants are taller, more slender and with a small number of leaves surrounding the flowers. Female plants are characterised as shorter, stockier and have more leaves meeting at each inflorescence.

A dried hemp stalk is shown in **Figure 1**. Dried hemp stalk. Each dried stalk consists of a hollow core (called 'hurd') which contributes 65 to 70% of the total weight. The bast fibre, of composite interest, is located between the hurd and epidermis, which contributes 25 to 30% of the total dry weight of a stalk [6]. Apart from the general classification of plant fibres, the bast fibres are of two types: primary and secondary bast fibres. Primary bast fibres are larger, stronger and contain more cellulose. The bast fibres are bonded together as fibre bundles. These can be separated into single fibres through alkali treatments. The average hemp fibre length and the average fibre width are 25 mm (5 to 55 mm) and 25 µm (10 to 51) µm, respectively [5]. Humans have used hemp for food, textiles, paper, fabric and fuel oil for thousands of years. Industrial hemp fibre applications include a wide range of composites for automotive, insulation materials and construction.

2.3 Matrix selection

The matrix is important in a NPFC, as it holds the plant fibres together within the composite. It can protect the fibres from adverse environments (e.g. water,

Figure 1.
Dried hemp stalk.

Thermoplastic polymers	Density	Water absorption (24h @h 20 °C)	Tensile strength (MPa)	Elastic modulus (GPa)	Izod impact strength (J/m)
Polypropylene	0.899 - 0.920	0.01 - 0.02	26 - 41.4	0.95 -1.77	21.4-26.7
Low Density Polyethylene	0.910 - 0.925	<0.015	40 -78	0.055- 0.380	>854
High Density Polyethylene	0.94 - 0.96	0.01- 0.2	14.5 – 38.0	0.4 -1.5	26.7-1068
Polystyrene	1.04 -10.6	0.03 - 0.10	25 - 69	4 -5	1.1

Table 3.
Properties of common thermoplastic polymers used in NPFCs [12, 13].

chemicals and impact properties) and transfers the applied load to the fibres. NPFCs include either a thermoset or thermoplastic polymer matrices [8].

Thermosets cannot be melted once cured, while tthermoplastics can be repeatedly melted by the application of heat and solidify on cooling. This repeatability is one of the main advantages of thermoplastics, as they can be recycled without much affecting their physical properties. Some thermosets used as matrices include unsaturated polyester, epoxy and vinyl ester. Commonly used thermoplastics include polypropylene (PP), polyethylene (PE) and polystyrene (PS). The selection of matrices in NPFCs are normally limited to those that can be processed at less than 200°C, although it is possible to use a maximum of 240°C for a short duration [2, 9].

Thermoplastic matrices offer several advantages compared with thermosetting matrices. These include recyclability, easier control in processing, high impact resistance, low cost, greater resistance to moisture and some industrial solvents and flexibility in design (molecules in a linear chain can slide over each other) compared to thermoset matrices (cross-linked) [10, 11]. The properties of some of the common thermoplastics used are listed in **Table 3**.

Polypropylene (PP) and polyethylene (PE) are the most widely used thermoplastic matrix in NPFCs, particularly for non-structural applications, because of its low density, low water absorption, excellent processability, good mechanical and electrical properties, good biological and chemical resistance, and good impact resistance and dimensional stability [2, 11–16].

3. Interfacial bonding between the fibre and the matrix

The strength of the interface has a significant influence on composite properties, which depends on the mechanism and amount of interaction. The mechanisms of interfacial bonding can be mechanical interlocking (rough fibre surface), chemical bonding (presence of chemical functional groups) and inter-diffusion bonding (interaction between atoms and molecules). There are possibilities of multiple bonding mechanisms occurring at an interface at the same time [2]. The interface strength also depends on the density of bonds. As already discussed, for NPFCs, there is usually limited interfacial bonding at the interface due to polar fibres and non-polar polymer matrices. This, in turn, affects the stress-transfer efficiency of NPFCs from the matrix to the fibre, thereby limiting the mechanical properties.

Most literature on interfacial bonding of NPFCs focuses on chemical treatments and coupling agents [4, 5, 17–22]. The main objective in conducting these treatments is to improve wettability and potential for chemical bonding of the fibre surface with the matrix, thereby providing interfacial strength (effective stress transfer across the interface) [23]. Wettability of the fibre by the matrix is most essential for the matrix-fibre adhesion, which can be assessed from the surface energy of the fibre and the matrix. The surface energy of the reinforcements should be greater than that of the matrix for the occurrence of fibre-matrix adhesion [24]. However, NPFs, due to their hydrophilicity, absorb atmospheric moisture when exposed to the ambient environment. This lowers their surface free energy, which may even result in their surfaces possessing lower surface energy than that of matrices. Coupling agents (also known as compatibilisers) act as a bridge between the fibre and the matrix and bond them together. Many studies have been carried out to achieve improved interfacial strength by different treatment methods on NPFCs.

3.1 Chemical methods

Chemical treatments involve reactions between fibres and reagents, including alkali, acetyl, silane, benzyl, acryl, stearic acid, maleic anhydride, permanganate, peroxide, isocyanate, titanate, and zirconate [2, 4, 5, 25, 26]. The most popular treatments are alkali, acetyl and silane [2]. The majority of these treatments are aimed to modify surface chemistry. However, alkali treatment, which has been found to be the best method [27], is effective through a number of factors; NPFs treated with alkali have been seen to have benefits such as improved separation of fibres from fibre bundles, improved removal of unwanted surface constituents, increased tensile strength and stiffness, better thermal stability as well as improved interfacial adhesions compared to other common treatments [2, 16–18, 28–30]. Here, we will be discussing the most used chemical treatment, which is the alkali treatment.

3.1.1 Alkali treatment

Among different chemical treatments, the alkali treatment with sodium hydroxide (NaOH) is one of the most widely used treatments. This treatment removes hemicellulose, lignin, pectin, wax and fat from the NPFs. The removal of hemicellulose, lignin, pectin (cementing materials) from the NPFs results in fibre separation and enhances exposure of hydroxyl groups on the fibre surfaces, thereby improving interfacial bonding and fibre roughness and increasing thermal stability [2, 18, 19]. Modest treatments have been seen to bring about increased cellulose crystallinity which is considered to be because of the removal of the abovementioned amorphous materials, whereas harsher treatments have been shown to convert crystalline cellulose to amorphous cellulose and possibly result in chain scission **Figure 2** [31].

The chemical reaction reported by some researchers, which occurs between fibre cell wall and NaOH (sodium hydroxide), are represented in **Figure 3**. The hydroxyl (OH) groups in the fibre break down and react with water molecules (H-OH). The water molecules are thus driven out. The remaining reactive groups in the fibre (i.e., Fibre cell-O) may form Fibre-cell-O^-Na^+ groups between the cellulose molecular chains, which could significantly improve tensile properties of the fibres. However, alkali treatment is commonly carried out to remove the cementing materials.

Different researchers have carried out these alkali treatments in different ways, including at ambient temperature (AT) and high temperature (HT). AT treatments have many advantages, such as simplicity, low cost and can be easily carried out in large volumes, compared to HT treatment which requires fully controlled methods. Oushabi et al. investigated the effect of alkali treatment on date palm fibres with various concentrations of NaOH (0 wt %, 2 wt %, 5 wt %, 10 wt %) at 25 °C for one hour and found an increase in tensile strength of date palm fibres compared to raw

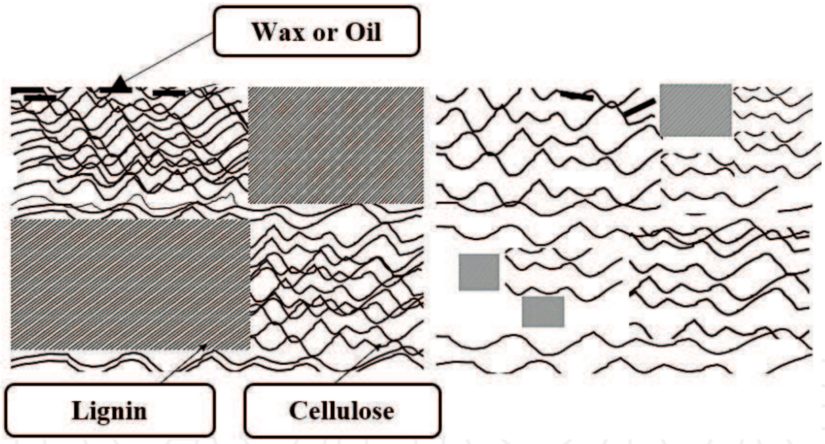

Figure 2.
Change in crystalline cellulose structure before (left side) and after treatment (right side).

$$\text{Fibre-cell-OH} + \text{NaOH} \longrightarrow \text{Fibre-cell-O}^{-}\text{Na}^{+} + \text{H}_2\text{O} + \text{impurities}$$

Figure 3.
Chemical reaction between fibre cell and NaOH [4].

fibres [32]. Mishra et al. reported that alkali treatment at 30°C for one hour with 5 wt% NaOH concentration resulted in better strength for sisal/glass fibre polyester hybrid composites compared to 10 wt% NaOH [33]. Mohanty et al. carried out alkali treatment for sisal fibres at 30°C with 5 wt% NaOH for one hour and reported a slight improvement in mechanical properties of sisal/polypropylene composites [34]. **Table 4** lists some of the recent works on AT and HT alkali treatment of hemp fibres. As it can be seen, for different high temperature treatments significant improvement in average tensile strength was reported for hemp fibres treated with 5 wt% NaOH and 2 wt% Na_2SO_3 (sodium sulphate) at 120°C with a holding time of 60 minutes compared to 10 wt% NaOH and untreated fibres [18].

Among the two different alkali treatments (high temperature and ambient temperature) for hemp fibres, it has been reported that most of the high temperature treatments maintained or increased the fibre tensile strength (or reduction reported for tensile strength was lower) compared to that of untreated fibre, whereas most reported ambient temperature treatments reduced the fibre tensile strength [40]. The removal of weak components has explained the increase in tensile strength of the high temperature treated fibre, and thus, the remaining material is stronger. Furthermore, the removal of weak components from the fibre cell walls could be leading to close packing and alignment of cellulose chains. The close compaction could have enhanced the adhesion between cellulose microfibrils, thereby providing better fibre tensile strength towards the loading direction compared to untreated fibres.

The ambient temperature treatments reported removed some of the weak components, resulting in a significant reduction in fibre strength, suggesting that cellulose degradation had occurred during these treatments. Literature has reported that chemical reagents first react with the chain ends at the surface of the crystallites, as they cannot diffuse into the crystalline region, thus limiting crystalline damage to open some of the hydrogen-bonded cellulose chains. The chemical reagent then diffuses into the crystalline region, reacting with the cellulose and generating more amorphous cellulose (cellulose degradation) [41,

Methods of applications	Fibres or composites produced	Observations on properties of fibres or composites
Soaked hemp mats in 0.16 wt% NaOH for 48 hours	Non-woven hemp mats in euphorbia resin	Increase in tensile strength of composites produced with treated fibre mats [37]
Immersed pre-dried hemp fibres in 5 wt% NaOH solution for 30 minutes, *FSR- 1:20	Hemp fibre	Average tensile strength of the fibres increased [17]
Hemp fibres were soaked in 0%, 4%, 6%, 8% and 10% for 3 hours at room temperature. NaOH solutions were prepared in water ethanol mixtures.	Hemp fibre	All treatments reduced the tensile strength [38]
Hemp fibres were treated with 5 wt% NaOH and 2 wt% Na_2SO_3 solution in *SSCs at 120°C for 60 minutes	Hemp fibre/ polylactic acid	Interfacial shear strength increased as a result of alkali treatment, thereby improving mechanical properties of composites produced [39]
Hemp fibres were treated with 5 wt% NaOH and 2 wt% Na_2SO_3 solution in *SSCs at 120°C for 60 minutes	Hemp fibre/epoxy	93% of lignin was removed after the treatment. Improved tensile strength and Young's modulus of short fibre/epoxy composites [19]
Two different alkali treatments. In first method, fibres were treated with 10 wt% NaOH to a maximum of 160°C for 45 minutes in *SSCs, FSR - 1:6. In the second method, 5 wt% NaOH and 2 wt% Na_2SO_3 solution in *SSCs to a maximum of 120°C for 60 minutes, FSR-1:7	Hemp fibre	5 wt% NaOH and 2 wt%Na_2SO_3 treatment improved tensile strength and Young's modulus. Improved fibre separations of fibre bundles were also resulted with both methods of alkali treatments [18]
Three different alkali treatment; 5 wt% NaOH, 10 wt% NaOH, 5 wt% NaOH and 2 wt% Na_2SO_3, solutions in *SSCs at 160°C for 30 min	Hemp fibre	5 wt% NaOH and 2 wt%Na_2SO_3 improved fibre separation. Average tensile strength of the fibre reduced [29]

*Note the following: *SSCs- stainless steel canisters and FSR- fibre to solution ratio.*

Table 4.
Some of the recent works on alkali treatment of hemp fibres or composites produced [4, 17–19, 29, 35, 36].

4. Conclusions

While considering preliminary treatments for industrial hemp fibres, high temperature alkali treatments seem best to produce strong and stiff fibres because low temperature treatments are most likely to bring about degradation of the crystalline cellulose chains in the microfibrils or bonding between cellulose microfibrils before sufficient removal of weak components from the fibres.

Declarations

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