



## Effects of chemical treatments on hemp fibre structure



M.M. Kabir<sup>a,\*</sup>, H. Wang<sup>a</sup>, K.T. Lau<sup>a,b</sup>, F. Cardona<sup>a</sup>

<sup>a</sup> Centre of Excellence in Engineered Fibre Composite (CEEFC), Faculty of Engineering and Surveying, University of Southern Queensland, Toowoomba, Queensland 4350, Australia

<sup>b</sup> Department of Mechanical Engineering, The Hong Kong Polytechnic University, Kowloon, Hong Kong Special Administrative Region

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### ABSTRACT

In this study, hemp fibres were treated with alkali, acetyl and silane chemicals. Fibre constituents such as cellulose, hemicellulose and lignin constituents were separated from treated fibres. The chemical and thermal influences of these constituents on the treated fibres were examined by using scanning electron microscope (SEM), fourier transform infrared (FTIR) spectroscopy, thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC). Experimental results revealed that, hemicellulose was degraded faster than that of cellulose and lignin. Cellulose exhibited better thermal stability and lignin was degraded in a wide range of temperatures. The hydrophilic nature of the fibres was predominantly caused by the presence of hemicellulose and then lignin constituents. Hemicellulose and lignin were mostly removed by the alkalisation with higher concentrations of NaOH, followed by acetylation. Silane treatment could not remove the hemicellulose and lignin, rather this treatment facilitated coupling with the fibre constituents.

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### 1. Introduction

Among various plant based natural fibres, hemp fibres are widely available as lignocellulosic reinforcing materials in composite applications. The structure of hemp fibre consists of three main constituents, i.e. celluloses, hemicellulose and lignin. Fig. 1 presents the structural organisation of fibre. Cellulose is considered the major framework component of the fibre structure. Celluloses are covered by hemicellulose and lignin constituents and provide strength, stiffness and structural stability of the fibre [1–3]. The cellulose structure of the fibres is distinguished through crystalline and amorphous regions, whereas hemicellulose and lignin are completely amorphous. In cellulose crystalline region, large numbers of strongly linked hydroxyl groups are present. These hydroxyl groups are inaccessible and so other chemicals have difficulty penetrating the crystalline region (Fig. 2) [4]. However, in the amorphous region, the hydroxyl groups are loosely linked with the fibre structure and are relatively free to react with other chemicals. Due to this freedom, hydroxyl groups present in the amorphous region can easily combine with water molecules from the atmosphere. The hydroxyl groups present in the amorphous hemicellulose and lignin initially give the access of water molecules to penetrate the fibre surface. Water molecules then combine with the hydroxyl groups that are present in cellulose (in the amorphous region) and

stay in the fibre structure. This makes the fibre hydrophilic and polar in character. Hydrophilic fibres worsen the ability to develop adhesive characteristics with most hydrophobic binder materials during composite processing. As a result strong bonding between fibre and the binder materials are compromised which reduced the composites mechanical properties. This problem can be addressed by treating these fibres with suitable chemicals. Treatments can decrease the hydroxyl groups that presents in the amorphous region, remove the lignin and hemicellulose coverings from the fibre surface and expose the cellulose structure to react with binder materials. Chemical reactions instigated by alkalisation, acetylation and silanisation are widely used to modify fibre structures.

Alkali (NaOH) treatments reduce amorphous hydroxyl groups from the fibre ( $\text{Fibre-OH} + \text{NaOH} \rightarrow \text{Fibre-ONa} + \text{H}_2\text{O}$ ). Due to this hydrophilic nature of the fibre reduced. Treatment also takes out a certain portion of hemicellulose and lignin covering materials. As a result, larger number of possible reaction sites of cellulose is available for matrix (binder) adhesion [5,6]. Acetyl treatment is the esterification method for the replacing of hydroxyl groups from the fibre with the acetyl group ( $\text{CH}_3\text{CO}$ ) [7,8]. As a result, the hydrophilic nature of the fibre decreases. Treatment also breaks down the hemicellulose–lignin covering from the fibre surface and expose cellulose surface for matrix adhesion. On the other hand, silane is a multifunctional molecule which is used as a coupling agent to modify fibre surfaces [9]. Silane molecules also act as a surface coating on the fibre. During silane treatment, several stages of hydrolysis, condensation and bond formation take place [7]. Silanols form in the presence of moisture in the fibre (hydrolysis). During the condensation process, silanol reacts with the cellulose

\* Corresponding author at: P-9, CEEFC, USQ, Darling downs, Toowoomba, QLD 4350, Australia. Tel.: +61 7 4631 1336; fax: +61 7 4631 2110.

E-mail address: [kabirm@usq.edu.au](mailto:kabirm@usq.edu.au) (M.M. Kabir).

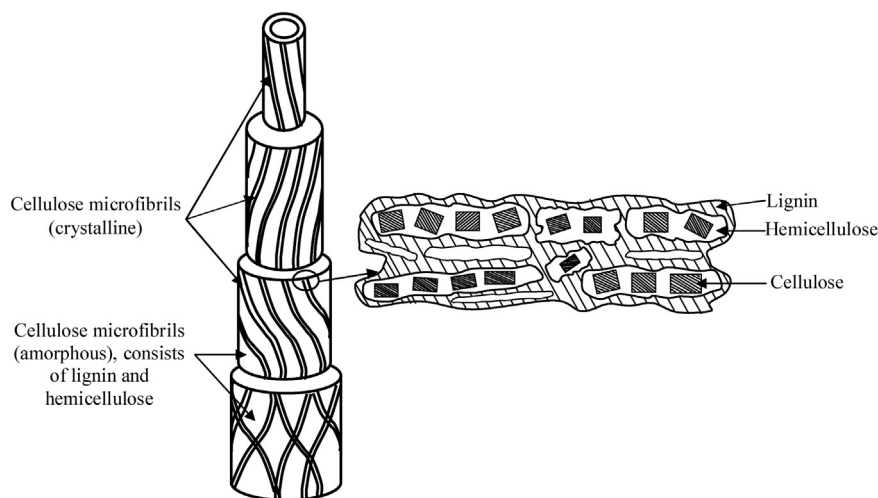


Fig. 1. Structural organisation of natural fibre [3].

hydroxyl group (Si—O—Cellulose) and forms chemical bonds (bond formation) with fibre constituents.

The treatment effect changes the contents of cellulose, hemicellulose and lignin constituents of the fibre. These constituent changes can be detected through SEM, chemical analysis, FTIR and thermal (TGA and DSC) analyses. SEM analysis detects the change on the treated fibre surface and its morphology. The quantitative estimation of fibre constituents can be evaluated through chemical analysis. FTIR helps to identify the functional groups present in the fibre, thereby highlighting the chemical differences among the fibre constituents. TGA aids in demonstrating the reaction mechanisms of fibres which occur at the molecular level of the fibre constituents as a function of progressive temperature. Fibre constituents degrade at different temperatures and results in weight loss. DSC allows a measurement of the chemical changes among the fibre constituents when subjected to heat.

Many studies had been conducted by using different chemical treatments on natural fibres. The treatment effects were reported in terms of fibre thermal properties. De Rosa et al. [10] showed

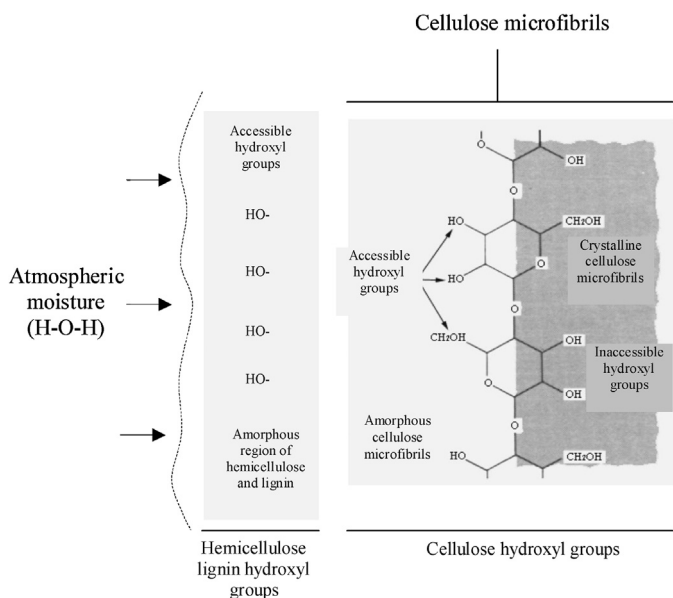


Fig. 2. Schematic presentation of the orientation of fibre constituents that absorb moisture [4].

acetyl treatments lowered the thermal stability of the okra fibre. However, Phattaraporn et al. [11] reported higher thermal stability properties after silane treatments. Vallo et al. [12] reported higher thermal properties of hemp fibres after alkali (8% NaOH) treatment, whereas Brigida et al. [13] showed lower properties. Siregar et al. [14] reported that the thermal degradation of a natural fibre occurred at a temperature region of 340–345 °C. Brigida et al. [13] reported that the cellulose and lignin degradation temperatures were 200 °C and 310 °C respectively. From all the previous studies, the treatments effects on fibre constituents specifically cellulose, hemicellulose and lignin behaviours were not clearly stated.

In this study, the main fibre constituents i.e. celluloses, hemicellulose and lignin were separated from the treated fibres. SEM, FTIR, TGA and DSC analysis of these constituents was performed separately to develop a basic understanding of the individual fibre constituent behaviours. These constituent behaviours were used to systematically analyse treatment effects on fibre thermal properties.

## 2. Experimental methods

### 2.1. Materials

Hemp fibres were obtained from Eco Fibre Industries, Australia. For alkalisation, commercial grade of NaOH was used. For acetylation, acetic acid and acetic anhydride were used and for silanisation, oligomeric siloxane was utilised. Sodium chlorite (commercial grade), acetic acid (glacial), potassium hydroxide (24% v/v), sulfuric acid (72% v/v) were used to separate cellulose, hemicellulose and lignin constituents from the fibre.

### 2.2. Fibre surface treatments

Alkaline, acetyl and silane treatments on hemp fibres were performed. Fig. 3 shows the treatment outline on the hemp fibres. The alkali treatments used different concentrations of NaOH solution. The solution was prepared by adding 0%, 4%, 6%, 8%, and 10% NaOH (by weight) in a water ethanol mixture (water:ethanol = 80:20). It was then stirred at room temperature for an hour. Hemp fibres were soaked with different concentrations of NaOH solutions for 3 h at room temperature. The fibres were then washed several times with distilled water to allow absorbed alkali to leach from the fibre. The washed fibres were dried at room temperature for 8 h, and then oven dried at 100 °C for another 6 h. The dried fibres were stored in

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