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Research paper

Differential behaviour of nodes and internodes of wheat straw with various pre-treatments

Seyed Hamidreza Ghaffar, Mizi Fan*

College of Engineering, Design and Physical Sciences, Brunel University, Uxbridge, Middlesex, UB8 3PH, United Kingdom

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This study is aimed at fully understanding wheat straw by investigating important properties for the processing of wheat straw (node and internode) for further production of bio-composites and bio-fuels as they server as great renewable resource. A combination of mild physical treatments and the synergetic effect of each physical treatment were investigated in terms of chemical, surface and mechanical properties. Functional groups changes were monitored in two anatomical sections of wheat straw stem inner and outer surface and the results showed a reduction in the intensities assigned to aliphatic fractions of waxes and silica after each stage of pre-treatments when compared to the untreated samples. The penetration rate (wettability) and hydrophobicity of wheat straw internode outer surface was analysed through contact angle measurements which indicated 35% decrease in hydrophobicity of straw surface therefore increasing the wettability of surface after the combinational pre-treatment. The relationship of surface characteristics to the mechanical properties of wheat straw was also investigated by testing the single strand tensile strength of nodes and internodes before and after pre-treatments. Mechanical test revealed 35% increase in tensile strength of wheat straw after the pre-treatment in comparison to untreated wheat straw. The thermogravimetric analysis clearly illustrate that the thermal stability of the wheat straw increased after the pre-treatment which is encouraging for the bio-composite application.

1. Introduction

Wheat straw is a promising biomass for the production of bio-products which could present a novel source for the eco-building materials and systems. The European countries (27 countries) produce 137.49 million tonnes of wheat collectively which is 16.59% higher than that produced by China [1]. However, most of the straws are burnt in the field which causes significant environmental and health problems. Compared to wood, straw is much lighter in weight and has more water resistance. The main chemical constituents of wheat straw are similar to those of wood; cellulose, hemicellulose, lignin and extractives, however, wheat straw has higher content of hydrophobic waxy cuticle layers and a high amount of inorganic silica and extractives. The research concerning wheat straw pre-treatment and optimisation for bio-products have developed extensively over the past few years [2–17]. Finding the limitations with the straw biomass for the targeted bio-product (i.e. bio-composite) is the key to an outstanding research and development; hence the detailed study of straw is required to finding the limitations associated with it in different application. In this study wheat straw stem is investigated as a whole, rather than particles or fibres and is pre-treated in order to optimise its surface chemical distribution and mechanical properties so it could be utilised in bio-composite. Research on the bio-composites from wheat straw has been on utilising straws in particle form with very short length (i.e. [9,13,18–21]), whereas the mechanical properties could potentially be increased by using longer straws and also the energy consumption of the processing of raw materials could be lowered. Limited research has been done to characterise the surfaces of wheat straw and specifically on the differences between node and internode.

The waxy layer on the outer surface of wheat straw is one of the main inhibiting factors for the reduction of bonding quality in bio-composites and on the other hand it makes the straw less accessible to the main components such as cellulose and hemicellulose which could be utilised as bio-energy source. Waxes appear primarily in

* Corresponding author.
E-mail address: Mizi.fan@brunel.ac.uk (M. Fan).

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the cuticular and epidermal tissues along with micro-sized silica particles (phytoliths) that are unique to each plant species [9]. Straw biomass stems are covered with semi-crystalline wax particles, 1–2 μm across and 2–3 μm high [22]. The wax is normally made up of a mixture of primarily long chain fatty acids and fatty alcohols, sterols and alkanes [23]. The bio-composites from straw biomass will experience low quality without any pre-treatments to address the associated issues raised from the waxy layer on surface [24]. On the other hand the pre-treatments should not only be environmentally sustainable but also should not deteriorate the mechanical integrity and rigidity of each individual straw. Combinatorial pre-treatment strategies are identified as an emerging pre-treatment technology in enhancing the biomass digestibility and overcoming the relative bottlenecks in utilisation and optimisation of biomass. Traditionally wax layer was extracted by the organic solvents like ethanol/benzene. Han et al. [25] reported that the wettability of wheat straw surface was enhanced through ethanol/benzene extraction and the bond-ability of particleboards made from extracted wheat straw was improved due to the removal of wax-like substances and other nonpolar extractives from the straw surface. Other approaches have been used to increase interfacial adhesion between straw surface and resin system, such as steam explosion [26,27], acid or alkali treatment [28,29], coupling agents modification [30], and enzyme treatment [12,31].

This research evaluates whether a combination of mild pre-treatments are sufficient for an economically feasible and environmentally sustainable system for the optimised utilisation of biomass for bio-products. The objective of this study is to investigate in detail the differences in node and internode of wheat straw when subjected to various pre-treatments in order to be able to select the best possible anatomical section of the wheat straw stem after the pre-treatments, for the application of bio-composites. The reason for the very detail investigation of surface of node and internode (inner and outer surface), is that when it comes to bio-composite production, inevitably various scenarios of interfacial connection between different anatomical sections is going to occur, hence it is useful to know whether these properties play an important role in interfacial bonding quality; this bonding quality determines the consequent physical and mechanical properties of the overall composite.

2. Material and experimental plan

Wheat straw (*Triticum aestivum* L.) was obtained from the Dixon Brothers Porters Farm in Rickinghall, Norfolk, England (East of England). The straw was harvested in summer 2012 and wheat straw bales were prepared and dried directly on site and then the bales were collected and homogenised. Straw was stored in an ambient room temperature atmosphere to confirm air-dryness. The stems of wheat straw with the best condition were selected, cleaned and grouped for each pre-treatment. To separate the nodes and internodes the stems were cut carefully above and below the nodes. For analysis of the surface, the stem was cut longitudinally in half using a razorblade. All the samples were oven dried for 24 h at 104 °C prior to testing and pre-treatments.

2.1. Pre-treatment methods

2.1.1. Combinational pre-treatment (H+S)

Hot water temperature at 100 °C is the starting temperature of pre-treatment when the straws were introduced to the pressure cooker and the lid was then closed (the pressure cooker with lid closed has an inside pressure of approximately 0.103 MPa). The temperature inside the pressure cooker was monitored for duration of the pre-treatment (30 min), with advancing of time and pressure inside the pressure cooker, temperature also increased to a maximum of 105.7 ± 1 °C. Pre-heating time for the water at room temperature to reach 100 °C (starting temp. of pre-treatment) inside pressure cooker was 10 min. The solid to liquid ratio of 1:20 (by weight) was used for this pre-treatment. Hot water treatment (H) was followed instantly by steam treatment (S); wheat straw was taken out from the pressure cooker and was placed in a mesh basket positioned above hot water inside the pressure cooker so that steam at 100 °C passes through wheat straw for another 30 min (H+S see Fig. 1).

2.1.2. Hot water and steam pre-treatment (0.5H and 0.5S)

The hot water and steam pre-treatments were carried out exactly as the combinational pre-treatment procedure. In 0.5H the untreated wheat straw was pre-treated for 30 min in hot water in a pressure cooker and for 0.5S the untreated wheat straw was pre-treated for 30 min in a mesh basket placed above boiling water. These treatments were used individually for the characterisation purposes in order to understand the synergetic effect and also to evaluate where the most contribution to each characteristic arises from.

2.1.3. Mild alkaline pre-treatment (0.5NaOH)

A mild alkali pre-treatment is also carried out on wheat straw in order to compare and evaluate the designed physical pre-treatment. Sodium hydroxide (NaOH) solution concentration of 10 g kg^{-1} was used for pre-treatment of wheat straw for 30 min of incubation, samples in this solution were kept in an environmental chamber with 25 °C and a 55% relative humidity. After the pre-treatment the samples were washed with distilled water several times.

2.2. Analytical techniques

2.2.1. ATR-FTIR

ATR-FTIR spectra were recorded on a PerkinElmer Spectrum one Spectrometer. Wheat straw was mounted on an ATR equipped with 3× bounce diamond crystal and an incident angle of 45° was used. The instrument was operated under the following conditions: 4000–650 cm^{-1} range; 4 cm^{-1} resolution; 16 scans. The results are average of 5 sub-samples for each section and each surface.

2.2.2. Contact angle

The contact angle between water and the surface of wheat straw was measured using First Ten Angstroms FTA 1000 Analyzer System. The outer surface of pre-treated and untreated wheat straw (10 mm × 5 mm) was tested for 10 different specimens. A drop of water (2 μL) was placed on the outer surface of wheat straw using a micro syringe at 18 ± 2 °C. Wettability is expressed as the advancing contact angle of distilled water on the outer surface of the wheat straw. The spreading and penetration rate of the surface pre-treatment is also assessed through this test. Only internode outer surface is investigated as node's surface is not flat. The moment the contact angle of droplets fell the solid surface is the initial contact angle (θi) and after some time of penetrating and spreading the straw surface, it will reach the equilibrium value (θe) which in this study is after 500 s.

2.2.3. Tensile testing

For the tensile testing 3 different batches of samples were selected from each pre-treatment along with untreated wheat straw and from each batch 20 individual samples were randomly selected and tested for their tensile strength. In total for each pre-treatment and untreated straw 60 samples were tested. In order to reduce the variance of the result the following steps were