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Research Paper

Application of cryogenic and mechanical treatment in degumming of hemp stems

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Natural fibre reinforced composites are lightweight materials widely applied to automotive interior parts. However, there are some problems such as volatile organic compound (VOC) emissions exceeding standard levels and weak interfacial bonding, which limits their use in industrial applications. In order to overcome the existing problems, a new degumming method (cryogenic and mechanical treatment) is proposed in this paper. Firstly, hemp fibre bundles became loose after cryogenic treatment, and there was some micropore creation or microcracking. Secondly, mechanical treatment separated hemp fibres from the fibre bundles. Finally, hemp fibres were cleaned with alkaline liquid. The influences of cryogenic and mechanical treatment on the fibres were investigated by using scanning electron microscope (SEM), Fourier transform infrared (FTIR) spectroscopy and thermogravimetric analysis (TGA). The results revealed that cellulose content was increased from 66.25% to 78.93%, and hemicellulose and lignin were reduced to 7.16% and 2.82%, respectively. Decreases in the diameter and tensile properties of the treated hemp fibres were also observed. In addition, in differential thermogravimetric analysis (DTG), the untreated hemp fibres had a significant peak at about 336 °C, while the significant peaks of treated hemp fibres were approximately at 360 °C. The significant peaks at 336 °C and 360 °C represent the maximum oxidative decomposition rate, and indicate that the treated fibres had a higher thermal stability than the untreated fibres. It was found that cryogenic and mechanical treatment is feasible for the degumming of hemp fibres.

1. Introduction

Natural fibres are available, cheap, renewable, and have good properties such as higher specific stiffness and strength, degradation as well as good flexibility. Therefore, they have been identified as attractive reinforcement materials for composites. Among the variety of natural fibres, hemp fibres have gradually come into view (Beckermann & Pickering, 2008; Islam, Pickering, & Foreman, 2011; Lu & Oza, 2013).

Similarly to other natural fibres, hemp fibres mainly contain 6 components (Liu, Thygesen, Summerscales, & Meyeret, 2017; Liu et al., 2015; Zhang et al., 2009): cellulose, hemicellulose, lignin, pectin, waxes and minerals. They form a natural composite material in essence where the gum (hemicellulose, lignin and pectin) and single-celled fibres are

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interwoven into a network. The fibres are particularly long and contain rich cellulose, which makes hemp a promising source of natural fibres for biocomposites. It is worth mentioning that hemp fibres have other advantages such as low cost, excellent sound absorbance, strong ultraviolet resistance, corrosion resistance, good antistatic and adsorption performance (Kostic, Pejic, & Skundric, 2008; Sepe, Bollino, Boccarusso, & Caputo, 2018; Zhang et al., 2009). Nevertheless, hemp fibres contain a lot of the gum. Thus, it is necessary to degum hemp fibres.

Nowadays, traditional retting (Placet, Trivaudey, Cisse, Gucheret-Retel, & Boubakar, 2012), chemical treatment (Oza, Ning, & Ferguson, 2014), physical treatment (Qu, 2005), biological treatment (Angelini et al., 2015; Chiliveri, Koti, & Linga, 2016) and united degumming (Liu et al., 2015; Qu, 2005) are considered for hemp fibres. Liu, Ale, et al. (2017) and Liu, Thygesen, et al. (2017) compared mechanical properties of fibre/epoxy composites by using the traditional field retting and Phlebia radiata Cel 26 retting. After Phlebia radiata Cel 26 retting, the effective stiffness of the fibres was increased slightly. By contrast, it was decreased after traditional field retting. Kabir, Wanga, Lau, and Cardona (2013) evaluated effects of chemical treatments on hemp fibre structure. It was found that the hemicellulose and lignin were mostly removed by the higher concentration of NaOH, followed by acetylation. Although silane treatment would not remove them, it could facilitate coupling with fibre constituents. Nair, Lyew, Yaylayan, and Raghavan (2015) studied microwave assisted-degumming/retting of hemp stems. The changes of the cellulose, hemicellulose and lignin in microwave were investigated by near infrared (NIR) spectrometer analysis. The results showed the efficiency of microwave treatments in degumming. Liu et al. (2016) proposed that the removal of pectin could increase composite stiffness and ultimate tensile strength. The removal of hemicellulose could increase composite stiffness, but decrease ultimate tensile strength. Composites with 0.5% EDTA, 0.2% EPG and 10% NaOH treated fibres had the highest stiffness and the lowest porosity factor.

However, the degumming methods have the following problems, to a greater or lesser extent (Duval, Bourmaud, Augier, & Baley, 2011; Kim, Lee, & Kim, 2016; Kuglarz et al., 2014, 2016; van der Werf & Turunen, 2008). Traditional retting consumes large amounts of fresh water and excessively depends on climate. Chemical treatment has disadvantages that include the consumption of many chemical reagents with serious threats to the environment. Physical degumming is not thorough. Biological treatment also involves some issues such as requirements for special instruments and high cost of enzyme preparation in practical application. There are biochemical and physicochemical treatments and so on, but they cannot achieve the goal of being more efficient-less polluting.

In this study, cryogenic and mechanical treatment is proposed as a new degumming method. In this regime, the fibre bundles first become loose after cryogenic treatment, and some micropores or microcracking appears. Then the fibres are separated from the fibre bundles by means of mechanical treatment. Eventually, the fibres are cleaned with alkaline liquid. The surface morphology of the untreated and treated fibres was investigated by scanning electron microscope (SEM). The chemical and thermal influences for hemp fibres were studied using Fourier transform infrared (FTIR) spectroscopy and thermogravimetric analysis (TGA). Additionally, the diameter and tensile properties of the treated fibres were also measured.

2. Materials and methods

2.1. *Materials*

As shown in Fig. 1, the non-retted hemp stems (Fig. 1a) were obtained from the field in Heilongjiang Province, China in the autumn of 2017. The middle of stems (Fig. 1b) were chosen before the following treatments were applied.

2.2. *Fibre surface treatment*

2.2.1. *Cryogenic treatment*

Cryogenic treatment was conducted using a temperature-programmed control cryogenic chamber (SLX-30, Technical Institute of Physics and Chemistry CAS, China). The schematic diagram of cryogenic treatment process is shown in Fig. 2. Hemp stems were cooled from room temperature down to 193 K at a constant rate, which took 35 min, and then maintained at that temperature for 2 h. Then a third of them were quickly taken from the cryogenic equipment. Subsequently, the stems were dropped down to 153 K in 30 min. Half of them were taken out after the hemp stems had been subject to that temperature for 2 h. The fibres were then cooled to 77 K in 50 min, and were maintained at the temperature for 2 h. All the stems were then taken out and placed into the room air to naturally return to room temperature.

2.2.2. *Mechanical treatment*

Using a stripping machine, hemp stems were crushed to break down the woody core into short pieces (hurds or shives), and the hurds were then removed from the fibre surfaces. The fibres were passed through the mechanical processing equipment (Fig. 3a) in the next step. The fibres were spread out and fed into the feeding area. Driven by a motor, the upper and lower grooves alternately moved in two directions, with the forward movement faster than the reverse, which could repeatedly squeeze and rub fibres in the mechanical rubbing area. In the slapping and dusting area, particles and dust on the fibres were mechanically oscillated and removed.

2.2.3. *Alkaline liquid cleaning*

The alkaline liquid cleaning involved the use of a 20 g L^{-1} solution of NaOH, in which the fibre (g) to liquid (cm^3) ratio was 1:40. The fibres were cleaned in the alkaline liquid for 1 h at 50 °C. Hemp fibres were then washed several times with distilled water to allow absorbed alkali to leave the fibres. The washed fibres were oven dried at 100 °C for 6 h.

2.3. *Scanning electron microscope*

Hemp fibres were positioned on aluminium stubs with a copper sheet using double-sided carbon conductive adhesive tape. The aluminium stubs was then placed into a vacuum