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Peeling experiments for hemp retting characterization targeting biocomposites

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A B S T R A C T

The control of hemp plant harvesting, in particular the retting process, represents the major step for the development of high-performance hemp biocomposites. In this study, the consequences of hemp retting on the ease of fibre extraction and tensile properties of elementary hemp fibres is explored. Stem peeling experiments are combined with scanning electron microscopy (SEM) to investigate the influence of the retting degree in plants. After studying the evolution of fracture energy at the fibre/woody core interphase within the stem, this work investigates fracture mechanisms during peeling experiments which aims to understand the evolution of fibre bundle cohesion during retting. A drastic drop of fracture energy reveals the impact of retting on the ease of peeling the outer tissue of the stem (containing fibre bundles), leading to less damage of fibres during the extraction process. The positive impact of retting on various fibre defects combined together leads to a 33% increase in the tangent modulus between non-retted and retted fibres, highlighting the importance of understanding the retting process to open up to new markets for biocomposites.

1. Introduction

The use of natural fibres is a real opportunity to satisfy an increasing demand for high-performance eco-designed materials. Although mainly intended for the pulp and paper industry, hemp fibres possess intrinsic mechanical characteristics that favour their potential valorization in new technical and high-value-added industrial applications including non-woven materials for thermo-compressed parts, injection-moulded composite materials and textiles (Beckermann and Pickering, 2008; Pickering et al., 2007; Placet, 2009; Placet et al., 2017). However, accessing these major markets involves the production of high-quality raw materials that meet strict specifications. This, in turn, requires the transformation of hemp straws before defibration by stem retting, inspired by the example of textile-targeted cultures such as flax. Many studies have already demonstrated the impact stem transformation (Lühr et al., 2018; Müssig and Amaducci, 2018; Wang et al., 2018) and of retting on fibre quality, in terms of the chemical composition and mechanical behaviour as well as the surface and cohesive properties of fibrous elements (Liu et al., 2015a; Müssig and Martens, 2003; Tahir et al., 2011). Moreover, certain authors have pointed out the impact of extraction processes performed on green stems, which affect the mechanical properties of obtained natural fibres, by inducing various defects in their structure (Aslan et al., 2010; Bos et al., 2002; Hernandez-Estrada et al., 2016; Thygesen et al., 2011). To reduce them, the solution is to facilitate defibration by adapting the retting conditions according to the targeted application.

Abandoned in the middle of the 20th century due to water pollution (eutrophication) by stem fermentation products, water-retting was replaced by field-retting for the cultivation of plants and is currently the most used retting method in Europe. The retting process could bring a prior separation of the fibres from the woody core and within fibre bundles by degrading the pectic constituents present in the middle lamellae (Akin and Gamble, 1996; Meijer et al., 1995; Saleem et al., 2008). Currently, hemp producers determine the achieved degree of retting in a very empirical way by using organoleptic properties, manual mechanical tests and the color or toughness of straws. These parameters only very partially reflect the quality of the fibres and are known to be perfectible. Thus, it is very difficult for hemp producers to supply straws with a controlled level of retting to meet the specifications of the intended applications. Given the importance of the impact of retting on the quality of hemp fibres and the precise requirements of the fibre-using industries, it is essential to firstly define indicators to measure the degree of retting and then develop decision-making tools to control the homogeneity and quality of the raw material. This approach could promote defibration by a softer process leading to less fibre damage and thus a better quality control and an improvement in

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the mechanical performances of hemp fibres.

The main objective of this study is to use stem peeling experiments to investigate the effect of field retting process on the tissue cohesion of hemp stems and its impact on the tensile properties of elementary fibres. Existing literature mentions methods allowing the evaluation of retting degree by investigating several factors: chemical composition, morphology, bacterial/fungal colonization (Djemiel et al., 2017; Li et al., 2009; Liu et al., 2017b; Mazian et al., 2018). However, these methods do not allow to quantify in a mechanical way the decohesion capacity of the stems like it has been done in this paper. First of all, peeling conditions and sample moisture content are determined precisely to ensure valid comparison between similar samples. Indeed, previous studies (Gardon, 1963; Gent and Petrich, 1969; Kaelble, 1964; Kendall, 1973) have shown the influence of sample state (moisture content) and peeling conditions such as temperature, test rate and test angle on the fracture behaviour and peel force of adhesives. After describing the peeling process in stems showing different retting degrees, combined with microscopic observations of peeled surfaces, an analysis of fracture energy at the fibre/woody core interphase is presented. Finally, the impact of retting degree on the mechanical performances of elementary fibres is explored.

2. Materials and methods

2.1. Materials

Hemp stems (*Cannabis sativa* L., cultivars Fedora 17) were grown at the La Chanvrière cooperative farm (Bar-sur-Aube, France). Stems were mechanically harvested at the end of August 2016 after pre-harvesting the seeds. The stems were then laid out for field retting for 37 days. 10 stems were randomly collected before retting (sample R0), after 17 days of retting (R1) and after 37 days (R2) for the purpose of this study, then naturally air dried. Stems were kept in a controlled environment (T = 23 °C and RH = 50%) before testing. Fibres were extracted using an industrial mechanical process that involves breaking and scutching of the straws.

Precipitation and temperature was recorded during the retting period from end of September to early November (Fig. 1). From harvest to the first retting stage (R1), equivalent to 17 days, the average temperature reached 10.6 °C and precipitation were very little with 0.9 mm. From the first to the last stage of retting (20 days), slightly lower

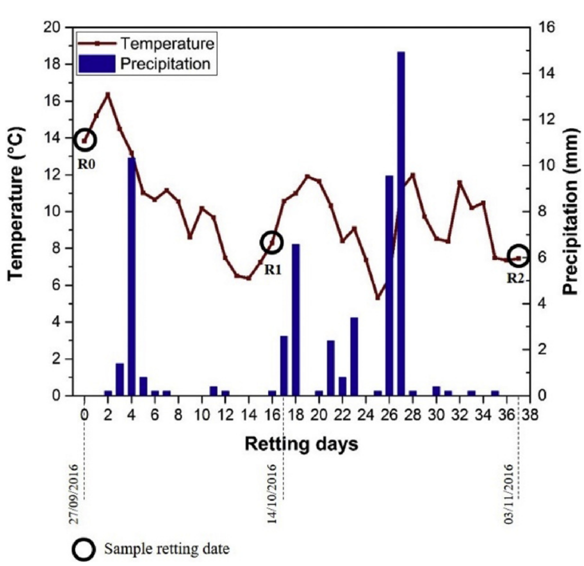

Fig. 1. Daily average temperature and precipitation during the period of hemp field retting.

temperature was recorded with 9.5 °C and precipitation was more important with 2.1 mm.

2.2. Moisture content measurement

Stems were kept in a controlled environment (T = 23 °C and RH = 50%) for 48 h for moisture content stabilization. Following this step, the stems were oven-dried at a temperature of 105 °C (commonly used to remove free and bonded water (Toury-Triboulot and Triboulot, 2012)) under atmospheric pressure until a constant weight was attained. During this process, stem samples were periodically removed from the oven for weighing. The percentage loss of water is stem samples, denoted by M_t, was measured with the following formula (Eq. (1)):

$$M_t = \frac{W_0 - W_t}{W_0} \tag{1}$$

where W_0 and W_t are the weight of stems before oven drying and the weight of dry material, respectively.

All the samples taken together show an average moisture content of 8.5 ± 0.3%. Only a slight variation of 0.5% was found between samples with different retting degrees. Previous studies have shown the influence of change of moisture content in wood on fracture toughness (Wang et al., 2003), reaching a maximum value at 82% relative humidity. Fracture toughness increases as the material dries out, reaching a maximum at 16.8% moisture content as the wood becomes less ductile. Booth et al. (2004) also observed the influence of stem moisture content on work required to peel due to changes in mechanical properties of the cell walls.

In the present study, all stems are assumed to have similar moisture contents, thus allowing us to study the influence of retting degree between comparable materials.

2.3. Peeling tests

Peeling tests were carried out at controlled temperature (23 ± 1 °C) and relative humidity (50 ± 1%). A universal MTS tensile testing machine equipped with a 2 N capacity load cell was used to measure the force required to peel the fibre-rich peripheral tissue ("outer tissues") from the woody core ("inner tissues") of the sample. The influence of displacement rate was first analyzed before testing. Before peeling, 220-mm-long samples were cut from the middle-height of the stem (initially measuring between 2–3 m high) and two longitudinal parallel cuts of 1.5 mm were made using a razor blade. Special attention was paid to perform the peeling test between two nodes of the stem to avoid any disruption. Samples were then clamped on a holder free to move horizontally at the same velocity as the outer tissue was being peeled vertically from the stem, maintaining a 90° angle between the peel and the substrate during the test. The developed experimental setup is illustrated in Fig. 2. The average fracture energy, G_f, is calculated from the force versus displacement graph after stabilization of the force, between 5 and 15 mm.

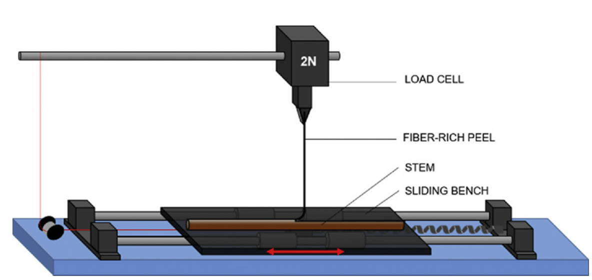

Fig. 2. Peeling test experimental set-up.