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Assessing the susceptibility of hemp fibre to the formation of dislocations during processing

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ABSTRACT

A method to assess the impact of processing on hemp fibres is presented. A testing process based on acid hydrolysis followed by fractionation, employing a Bauer McNett apparatus, was used to assess the extent to which dislocations formed in hemp fibre that had undergone different degrees of mechanical processing. Using the potential of acid hydrolysis to break the fibres preferentially at dislocations, fractionation was used to classify the resulting segments by size into eight fractions. Overall, the assessment method was shown to be a feasible and robust, as well as a reasonably fast technique. Using the same method, samples of hemp fibre taken from different stages of the production chain were used to assess the impact of the production processes on the formation of dislocations in the fibres. The results show that the first stage of processing (decortication) has the greatest impact on the formation of dislocations and that further processing steps (carding or coarse separation) also add more dislocations but with a lower impact.

1. Introduction

When wood and bast fibres are loaded in axial compression they frequently fail through buckling of the cellulose microfibrils (Dinwoodie, 1968; Nyholm et al., 2001; Hughes, 2012) forming structural deformations in the fibre cell wall that have been referred to variously as nodes, slip-planes, kinks, kink-bands, micro-compressions, micro-compressive defects or dislocations. As noted by Nyholm et al. (2001), different terminology can sometimes refer to the same type of damage. These deformations, which in this work will hereinafter be referred to as 'dislocations', can be formed in the living plant as a result of wind and water stress (Thygesen and Asgharipour, 2008) as well as during subsequent mechanical processing to extract the fibres from the stems (Thygesen and Hoffmeyer, 2005; Hänninen et al., 2012). The dislocations generally take the form of kink-bands that are characteristic of the compressive failure observed in unidirectional fibre-reinforced composites (Fleck, 1997) and in aligned polymer fibres (Deteresa et al., 1984) and can be readily seen under polarised light (Fig. 1). The term 'dislocation' used in this sense is not to be confused with dislocations found in crystals; however, since this term frequently appears in the literature (*e.g.* Thygesen and Ander, 2005; Thygesen, 2008) to describe this form of structural deformation of the cell wall, it will be adopted in this work too.

Dislocations can affect both the stiffness and strength of the fibres, compromising their potential in technical applications (Hughes, 2012). Thygesen et al. (2011), for example, found that processing decreased the tensile strength of hemp, reducing it from 998 ± 45 MPa before processing to 581 ± 44 MPa after dew retting and scutching. After a second scutching step, the tensile strength was further reduced to 396 ± 30 MPa, whilst after carding following the first scutching step, the tensile strength was found to be 503 ± 24 MPa. Thygesen et al. (2011) concluded that in addition to the processing adding damage to the fibre, the effect of scutching was greater than that of carding in terms of its effect on tensile strength.

Because of their potentially excellent mechanical properties, bast fibres such as flax (*Linum usitatissimum* L.) and hemp (*Cannabis sativa* L.) have attracted considerable interest as reinforcement in composites (Eichhorn et al., 2001). However, due to the presence of dislocations their full potential has yet to be fully realised (Hughes, 2012). Hughes (2012) widely reviewed the implications of fibre dislocations in composite materials, highlighting that their presence can be related to matrix failure, resulting from stress concentra-

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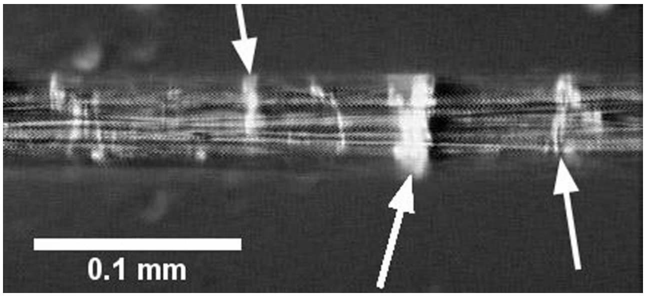

Fig. 1. Dislocations in a macerated hemp fibre seen under polarised light.

tions around the dislocations when a composite is loaded. Terziev et al. (2005) studied the impact of dislocations in paper products, and found that paper sheets made from fibres with dislocations induced by compressing the raw material exhibited lower tensile strength than those made from fibres without induced dislocations.

Whilst it is clear that dislocations are induced during growth (Thygesen and Asgharipour, 2008), processing has also been shown to have a major and detrimental effect on the fibres, leading to a significant accumulation of additional dislocations (Hänninen et al., 2012). However, whilst it is generally recognised that processing causes dislocations in the fibres, very little has appeared in the literature with regard to the relationship between processing and the amount of dislocations induced; what work there is, has generally been based on the study of different raw materials or fibres already processed industrially without mention of the characteristics of the processing (Thygesen, 2008; Hänninen et al., 2012). Clearly, if fibres such as flax and hemp are to be used more extensively as composite reinforcement and in more structurally demanding roles, then the relationship between the fibre characteristics, processing and the occurrence of dislocations must be more fully understood. This should enable processing strategies to be developed that will minimise the introduction of dislocations in the first place as well as suggesting ways in which their effects can be minimised.

Regarding hemp fibres, in order to quantify dislocations, two groups of techniques have thus far been used. The first approach is based on directly imaging the fibres using polarised light microscopy (Thygesen and Hoffmeyer, 2005; Mortensen and Madsen, 2014). The second employs indirect methods based on inducing cleavage of the fibres at the dislocations using, for example, acid hydrolysis, followed by testing to evaluate the degree of segmentation (Hänninen et al., 2012; Thygesen, 2008). The dislocations, in bast fibres like flax are particularly sensitive to attack by cellulases resulting in a weakened fibre (Foulk et al., 2008). Polarised light microscopy (PLM) of the partially degraded fibres after incubation with enzymes like Viscozyme, show attack at the nodes by the cellulase component in the enzyme mixture. Attack and degradation that takes place at nodes during acid hydrolysis resembles that induced by cellulase (Akin, 2010).

Since crystalline cellulose is a birefringent material (Thygesen and Hoffmeyer, 2005), PLM is a useful technique to visualize dislocations. This technique is explained in detail by Thygesen and Hoffmeyer (2005) and has been used for visualizing dislocations in pulp fibres (Thygesen and Ander, 2005; Thygesen et al., 2006) as well as in crop fibres such flax (Nilsson and Gustafsson, 2007; Hänninen et al., 2012) and hemp (Thygesen and Hoffmeyer, 2005; Mortensen and Madsen, 2014). Scanning electron microscopy (SEM) has also been used to image dislocations but is limited to the visualization of dislocations that are large enough to deform the entire cell wall structure of the fibre (Thygesen et al., 2006).

Since PLM is laborious and time consuming, other approaches to measuring the degree of fibre damage have been attempted. Relying on the propensity of dislocations to undergo enzymatic or chemical attack, an alternative approach utilising acid hydrolysis to cleave the fibres at the dislocations has been used to quantify the defects in hemp fibres and other fibre types such as flax (Hänninen et al., 2012) and wood fibres (Ander et al., 2005). Following acid hydrolysis, the resulting segments are analysed either by directly measuring the number and size of the segments (Thygesen, 2008) or indirectly, by subsequent dissolution of the fibre followed by measurement of the solution viscosity (Hänninen et al., 2012).

Thygesen (2008) analysed the segment length distribution of 5 mm lengths of hemp fibre yarn that had undergone acid hydrolysis, following the work done previously by Ander et al. (2005) with pulp fibres. The findings highlighted the feasibility of acid hydrolysis as a method to study the amount of dislocations in hemp fibres. Later, Hänninen et al. (2012) measured the viscosity of a solution of acid hydrolysed hemp samples to assess the dislocations induced in fibres that had been passed a different number of times between intermeshing gearwheels (to simulate the decortication and carding process). It was found that the viscosity of the acid hydrolysed fibres decreased when the number of passes increased, suggesting that the amount of dislocations in the fibres is greater when more work is done in the processing. Interestingly, Hänninen et al. (2012) observed that the difference in viscosity values is small, suggesting that the fibres had already been processed and that after a certain number of passes through the gearwheels, the viscosity appeared to level-off, suggesting the existence of a threshold level of dislocations.

Whilst dissolution of acid hydrolysed fibres followed by viscosity measurement is revealing, it is rather complex and time consuming for routine work where a more direct measurement of the segment length is to be preferred. Classification of pulp fibre using a series of screens of differing mesh size has been routinely used in wood fibre pulp classification and in this work a similar procedure was adopted. The Bauer McNett fibre classifier is a laboratory screening device based on a series of tanks arranged in a cascade. Each tank has a sieve installed with a characteristic mesh opening to retain fibres of a specific size and allow smaller fibres to pass through the sieve to the next tank and so on. The fractionation process is performed with a continuous flow of water from the upper tank to the lower one; the fibres are distributed amongst the tanks according to their length (Levlin and Söderhejelm, 1999; Gooding and Olson, 2001) and the fibres in each tank are retained by a filter when the tank is emptied. The Bauer McNett classifier has been widely used in the paper making field to screen fibres (Jones, 1970; Gooding and Olson, 2001), since fibre length has an impact on many of the performance parameters of end products such as paper sheets (Clark, 1942). Whilst the Bauer McNett device has been widely used for wood pulp fibres (Gooding and Olson, 2001) it has also proven its utility with non-wood fibres such as kenaf (Azizi Mossello et al., 2010) and bamboo fibres (Sood et al., 2005).

The overall aim of this work was to assess the influence of processing on the formation of dislocations in hemp fibres during decortication and separation, using a combination of acid hydrolysis followed by analysis using a Bauer McNett device. A further aim was to develop a robust testing method to assess such influence by using small samples of fibre. Our working hypothesis was tested by determining the influence of decortication and separation on the formation of dislocation in two different hemp (*C. sativa* L.) varieties.

2. Materials and methods

The hemp (*C. sativa* L.) varieties used in this work were USO-31 and Felina-34. The USO-31 stems were used to assess the influence of the production process and were thus processed in different