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Mechanical processing of bast fibres: the occurrence of damage and implication for fibre structure

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Abstract

Currently, separation processes used for natural fibres for composite reinforcing textiles cause a significant amount of damage to the fibres. Microscopic analysis showed that low grade industrial fibres contained significantly more defects than green or retted ones and further mechanical processing did not increase the amount of defects. In this study we have shown, by analysing degree of polymerization of cell wall components by viscosity measurements that mechanically defects caused by mechanical processing do not significantly cleave the cell wall polymers. Acid hydrolysis, however, induced more degradation to the defected fibres than intact ones. This shows that defects are more susceptible to certain chemical reactions, which in turn might cause problems for example, during chemical modification of fibres due to heterogeneous reactivity.

Keywords:

Natural fibre

Mechanical processing

Defects

Hemp

Flax

1. Introduction

Over the past two decades or more, growing environmental concerns have led to resurgent interest in the use of natural plant fibre in technical applications. One of the most exciting application areas is that of composite reinforcement and today many automotive manufacturers use several kilogrammes of natural fibre composite materials in car interior components (Hännien & Hughes, 2010). The low density of plant fibre compared to, say, glass fibre offers advantageous weight savings, important in transportaion applications, whilst the renewability and low embodied energy of the fibres engender good environmental credentials. However, despite the admirable tensile mechanical propeties often reported for fibres such as flax (*Linum ussitatissimum*) and hemp (*Cannabis sativa*), the properties of natural fibre reinforced composites (NFRCs) rarely, if ever, match the potential shown by the fibres and as such the applications of NFRCs have been non-structural. There is, however, much interest in using natural plant fibre in more demanding roles, since this would broaden the application areas. So why are the properties of NFRCs so inadequate? Partly this can be explained by the lack of suitable reinforcement textiles and partly by the inherent properties of the fibre.

Hemp and flax are amongst the most commonly used fibres for NFRCs. The fibres used in composites are prepared by separating them from the stems and further refining them by mechanical action. Traditionally, the extraction of flax fibres begins after retting with the dry straw being scutched. Scutching involves the breaking-up of the straw mechanically, with the shives being removed from the technical fibres. The final operation is termed hackling, which involves pulling the fibres through a set of pins to align them and to remove any remaining woody material (Summerscales et al. 2010). This processing, however, tends to damage the fibres, which can be seen as disruptions in the fibre cell wall structure and frequently takes the form of kink bands. These kink bands, commonly referred to as dislocations, but may also be known as nodes or slip planes, are reminiscent of the failures seen in the cell walls of wood fibres that have failed in compression (Dinwoodie, 1968). Whilst the dislocations are thought to form mainly during fibre separation, they have also been observed in unprocessed fibres (Hartler, 1995; Thygesen, 2011; Thygesen and Asgharipour, 2008).

The effect that dislocations in wood fibres have on their properties, and on the properties of paper and wood, has been extensively studied over the years (Dinwoodie, 1968; Page et al., 1972; Mott et al., 1996; Ander et al., 2006). Davies and Bruce (1998) studied the effect that dislocations had on non-wood fibre (nettle and flax), finding that fibre damage negatively affected the tensile mechanical properties. Similar findings have been reported by several other authors (e.g. Baley, 2004; Thygesen et al., 2007a). In addition to negatively affecting the properties of the fibre, dislocations have been shown to give rise to matrix stress concentrations, when such fibre is used as composite reinforcement, that leads to matrix cracking (Hughes, 2000) and to the onset of inelastic behaviour at low levels of applied stress (Hughes et al., 2007). It has also been speculated that, in addition to reducing the mechanical properties of the fibres, defects may prove to be more susceptible to chemical attack than the intact areas of fibres (Hänninen et al., 2011) as well as causing chemical reactions to take place heterogeneously in the fibres. Good evidence for this has been provided by Thygesen (2005) who used acid hydrolysis as a means of quantifying the dislocations in hemp (and spruce pulp) fibres.

The work reported herein, builds upon previous work (Hänninen et al. 2011) and aims to explore the relationship between fibre processing, the occurrence of damage and the accompanying changes to the fibre structure. In this study the progressive accumulation of fibre damage, caused by processing, was assessed microscopically, whilst the extent of damage to the cell wall polymers was investigated by dissolution of the fibres in cupriethylenediamine (an established solvent for lignocellulosic fibres) and subsequent measurement of the viscosity of the solution. The viscosity of the solution is known to correlate with the average degree of polymerisation (DP) and is an established method in the paper industry for analysing the extent of chemical degradation during pulping and bleaching (da Silva Perez and van Heiningen, 2002). The increased chemical reactivity of the fibre in the area of the dislocations was assessed by measurement of viscosity following acid hydrolysis of the fibre.

2. Materials and methods

Flax fibres were provided by Ekotex, Namysłów, Poland and hemp fibres by BaFa GmbH, Malsch, Germany. The flax samples were supplied as green stem (i.e. before retting), as retted stem and as two fibre products (grades) known as "noils" and "kotonina" (Table 1). Noils are flax fibres mechanically separated from the stem and cleaned of shive. Additional mechanical processing of the noils, separating the fibres further, yields kotonina. The hemp fibre was mechanically separated from the stem and had the shive removed (in processes known as 'scutching' and 'carding' respectively), but had not undergone any further processing.

To investigate whether increasing levels of damage induced during a particular mechanical process affected the susceptibility of hemp fibre to chemical degradation a series of artificially damaged fibres were prepared. Hemp fibres were passed between intermeshing cogwheels a varying number of times and the damage to the fibres reported as the number of passes (0, 5, 12 and 22) that the fibres had made through the equipment.

Single flax fibres were carefully extracted manually from fibre bundles. The fibres were cleaned by ultrasound in water to remove surface impurities so as to make the defects visible. The fibres were imaged using a Leitz optical microscope equipped with polarisation filters. Cross polarised light was used to make the defects appear as bright or dark line patterns. The intensity of the light, and the angle of the fibre relative to the polarisation filters must be strictly controlled to obtain reliable results (Thygesen et al., 2006). Five fibre samples, corresponding to a total fibre length of about 30 mm, for each fibre type were analysed. The fibre samples were scanned from one end to the other, and the defected sections were identified and their widths measured. The following defect quantities were measured: defect count per length (mm^{-1}), width of defect free sections (mm), width of defect sections (mm), and defect content (%).

A gravimetric method was used to determine the contents of: wax, pectin, lignin, hemicellulose and cellulose in the fibres (Browning, 1967). The method consisted of 5 steps: (1) wax was extracted using a Soxhlet apparatus using a mixture of 66 % toluene, 17 % acetone and 17 % ethanol for 5 h. (2) Pectin was extracted in 30 g/L ethylene-diamine-tetra-acetic acid. The composition of the remaining residue was measured by strong acid hydrolysis of the carbohydrates. Dried and milled samples (160 mg) were treated with 72% (w/w) H_2SO_4 (1.5 ml) at 30°C for 1 h. The solutions were diluted to a total volume of 42 ml with water and were autoclaved at 121°C for 1 h. The hydrolysates were filtered and the lignin content was determined as the weight of the filter cake less the weight of the ash content (Kaar et al., 1991). Glucose, xylose, arabinose and ethanol were determined in the subsequent analysis procedures in a HPLC-system (High Pressure Liquid Chromatography; Shimadzu) with a Rezex ROA column (Phenomenex) at 63°C using 4 mM H_2SO_4 as eluent and a flow rate of 0.6 ml/min. Detection was done by a refractive index detector (Shimadzu Corp., Kyoto, Japan). Conversion factors for dehydration on polymerization were 162/180 for glucose and 132/150 for xylose and arabinose.

Acid hydrolysis of air dry fibres (10 g) was carried out by boiling in 0.1 M HCl for 60 min at 2 % (w/V) concentration. Concentration of acid was prevented by using a condenser with water cooling. After hydrolysis the fibres were filtered in Bühner funnel and washed with excess water and air dried.

The intrinsic viscosities of flax and hemp fibres, both before and after acid hydrolysis, were determined according to SCAN-CM 15:99 “Viscosity in cupriethylenediamine solution” standard. The fibre were cut to less than 1 cm long pieces to improve dissolution. The fibres were dissolved in 0,5 mol /l cupriethylenediamine (CED) solution according to the standard, however, to obtain the complete dissolution in CED the mixing time was extended from 30 minutes to 45 minutes. After dissolution, the viscosity of the solution was measured with a capillary viscometer.

3. Results and discussion

3.1 Flax fibres

The separation of the fibre from the plant stem is carried out when the materials is dry and when the mobility of the cell wall components is reduced due to the low amount of plasticising water (Salmén and Olsson, 1998), making the fibre structures brittle. Differences in extent and frequency of defects, which manifest themselves as light bands across the fibre, can be clearly seen from Figure 1. In fibre extracted from green stem, only a very few, thin light bands can be seen whilst in the cottonized fibre the defects are much more pronounced.

As may be seen from the microscopy data presented in Table 2, during retting, which does not involve any mechanical processing, no defects were induced in the fibres. This may be concluded from the fact that all the values of the green and retted fibres fall within the standard deviation. The difference between the unprocessed stem and the noils is very clear. The defect count per length and the defect content increased drastically, whilst the width of the defect free sections decreased after processing. No difference could be seen between any of the fibres in the width of the defected sections. Comparing the processed fibres, noils and kotonina, no differences could be seen in the defect count per length, the width of the defect free section, nor the defect content (Table 2). This finding would indicate that most of the defects are formed during the initial processing stages and that after a certain level of processing little further damage is introduced into fibres.

From the chemical composition of the flax fibres (Table 3), it can be seen that the proportion of pectin and other compounds (waxes and such) in the untreated fibres decreases, as expected, after retting (Mooney et al., 2001; Nykter et al., 2008; Thygesen et al., 2007b). Moreover, the cellulose content of the fibres increases significantly after the fibres have been processed. Mechanical processing most probably removes residual middle lamella particles, consisting mainly of lignin, hemicelluloses and pectin. It would appear that most of the residual middle lamella fraction is removed during the first process steps, since further mechanical treatment, in processing the noils to kotonina, does not significantly alter the chemical composition of the fibres. It must, however, be borne in mind that being commercially produced fibre, the original source of the flax fibre is unknown and thus some variations in the chemical composition are to be expected.

As may be seen from Table 3, following acid hydrolysis the chemical composition of all of the flax fibre types investigated is very much alike. Soaking the fibres in an aqueous acid solution will not only wash middle lamella particles off the surface of the fibres, but will also result in some hydrolysis of the hemicelluloses (Kontturi and Vuorinen, 2006), enhancing cleavage of the remaining middle lamella.

The intrinsic viscosity was used to analyse the average degree of polymerisation (DP) of the fibre cell wall polymers from dissolved samples, which consists of high molecular weight cellulose (DP 15000-7000) and low molecular weight lignin and hemicelluloses with DP of several hundred (Metsushitsuka and Isogai, 1996; Akin, 2010; Eder and Burgert, 2010). The intrinsic viscosity of dissolved fibres has been used to calculate the DP of the hemicellulose and cellulose fractions in chemical pulps (da Silva Perez and van Heiningen, 2002). However, due to the high proportion of pectin and other compounds, such as waxes in the solutions of flax or hemp fibres used in this study, the solution viscosities could not be directly translated into DP and the intrinsic viscosity was used only as an indicator of DP.

As may be seen from Table 3, a good correlation exists between intrinsic viscosity and the cellulose content of untreated flax fibres. This indicates that the main contributor to the intrinsic viscosity of the fibre solution is the cellulose. The increasing trend in viscosity despite the increase in defects (that accompanies the increase in the cellulose content) suggests that they are only a misalignment of the cellulose microfibrils rather than actual damage to the cellulose polymer itself that would result in a decrease of viscosity. This was earlier speculated by Hänninen et al. (Hänninen et al., 2011). Cleavage of the cellulose would have resulted in a decrease in intrinsic viscosity, whereas now the increasing value is probably due to removal of low molecular weight compounds contained in the residual middle lamella particles.

After acid hydrolysis, a clearly decreasing trend in viscosity of fibre solution can be observed as the defect content increases, whilst the cellulose content of the fibres remains constant (Table 3). A decrease in viscosity after acid hydrolysis as the defected area increases strongly suggests that the defects are indeed more susceptible to hydrolysis than defect-free regions of the fibre.

A severe drop in viscosity by acid hydrolysis can be expected due to hydrolytic cleavage of the glycosidic linkages between the cellulose monomers. Hydrolytic cleavage takes place randomly along the cellulose chain, which results in a much more severe decrease in the DP of the polymer than, for example, end-peeling (Klemm et al., 1998). Strong acid hydrolysis has been used to degrade cellulose to the levelling-off DP (LODP), which is the point at which the decrease in the DP of cellulose becomes insignificant (Klemm et al., 1998). The acid concentration used in our study was much lower than the one used for determining LODP.

Degradation of hemicelluloses and pectins also take place during acid hydrolysis. However, due to their low DP and low quantity in the samples, their influence on the drop in intrinsic viscosity, which corresponds roughly with the average DP of the sample, is most probably insignificant.

The increased susceptibility of defects to acid hydrolysis can be explained by their nature. The defects, which can be seen by polarized light microscopy due to their birefringent properties, are mainly the result of disruptions to the structure of the secondary cell wall (Bos and Donald, 1999; Hartler, 1995; Thygesen, 2005). As the non-defected parts of the cell wall is tightly packed it is able to keep aqueous solutions from penetrating the wall, any loosening of the tightly bound structure within the defects might enable reagents to access the inner parts of the cell wall, which in turn increases reactivity.

The susceptibility of the defects to acid hydrolysis has been used earlier in the quantification of defects in combination with microscopy (Ander, 2005; Thygesen, 2008). The methods are based on mechanically breaking the fibre at defected areas weakened by acid hydrolysis and measuring the fibre sections with a fibre analyser. Mechanical treatment, however, causes fibres to also break within the non-defected sections, leaving some of the defects intact. Defects can be analysed by microscopy only, however, the method is tedious and the automatic methods available are applicable only to short fibres, such as wood fibres (Joutsimo and Robertsen, 2004; Robertsen and Joutsimo, 2005). To our knowledge, our study is the first one to show the correlation between the chemical degradation of the cell wall components due to acid hydrolysis and the amount of defects in the fibres.

3.2 Artificially damaged hemp fibres

As may be seen from Table 4, the there was little change in the chemical composition due to mechanical processing, however, it must be remembered that the starting materials in this case was fibre that had already been processed and may be thought of as being analogous to the flax noils. Nevertheless, the slight increase in cellulose content is reflected as an increase in the intrinsic viscosity of the fibre solution. The change in the chemical composition is likely to be due to removal of residual middle lamella particles, which causes the apparent increase in the cellulose content of the fibres.

After acid hydrolysis, the chemical composition of the fibres is similar in all of the fibre types. Although a decreasing trend in the viscosities of the acid hydrolysed fibres can be seen (Table 4), the difference between samples is much smaller than in the industrially processed flax fibres. This is most likely due to the different starting material and the fact that the industrially processed flax fibres have been through several operations.

As with the industrial flax fibres, after a certain number of passes, corresponding to increasing levels of damage to the fibres, the viscosity of the acid hydrolysed fibres appears to level-off. This might indicate that there could be a sort of threshold level of damage that can be induced by any particular type of mechanical process. Reaching a point where the amount of damage levels-off in mechanical processing is a very interesting phenomenon and similar tendency has been seen in the beating of unbleached pulp fibres (Rauvanto et al., 2006), however, this is the first time it is reported for the processing of flax and hemp fibres.

Defects in natural fibres are clearly an issue that should be addressed among NFRC manufacturers and fibre producers. Defects have been acknowledged to be an issue in several research papers (Baley, 2002; Davies and Bruce, 1998; Hughes et al., 2000; Bos et al., 2002), but very little has been done to improve fibre quality, although more gentle ways to separate fibres have been studied (Nykter et al., 2008; Thygesen et al., 2007b; Wang et al., 2003; Zhang and Zhang, 2009). This study shows that a significant amount of defects are already formed in low grade fibres. The harsh mechanical fibre separation methods that originally were developed for the textile industry, where the damage to the fibres is not as problematic, are clearly not suitable for load bearing composite applications where high quality defect free fibres are needed. Defected fibres do not only diminish the strength properties of fibre and properties of NFRCs, but can cause problems if fibres need to be modified, for example, to increase fibre-matrix compatibility. As shown in this study, defects are indeed more susceptible to some chemical reactions than intact region of fibres, which causes heterogeneous reactivity along the fibres.

4. Conclusions

The amount of defects was shown to increase drastically to certain extent by mechanical processing of flax fibres. The viscosity measurements indicated that the processing did not cleave the cellulose chains, but merely disrupt the cell wall integrity. After the acid hydrolysis the viscosity values correlated well with the amount of defects, which indicated that the defected regions of the fibres were more susceptible to acid hydrolysis. These results were also verified with hemp fibres that were systematically damaged in laboratory.

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Figures:

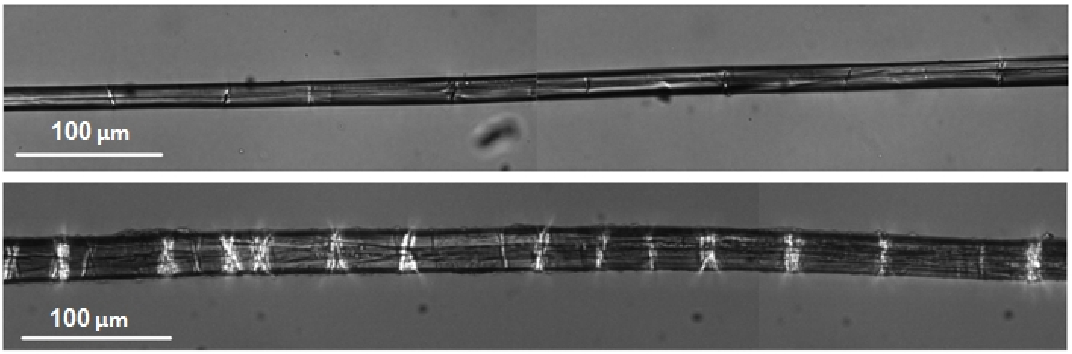

Figure 1. Polarized optical microscope images of two types of flax fibres: Green stem (above) and Kotonina (below). The defects can be seen as bright and dark lines across the fibres.

Tables:

Table 1. Sample names, preparation process and type of process

Sample name	Fibre preparation process	Type of process
Green stem	Harvesting	-
Retted stem	Retting	Chemical
Noils	Scutching and carding	Mechanical
Kotonina	Cottonization	Mechanical

Table 2. Defect data from image analysis of micrographs

	Defect count per length [mm-1]		Width of defect free sections [µm]		Width of defected sections [µm]		Defect content [%]	
Sample name	Mean	Stdv.	Mean	Stdv.	Mean	Stdv.	Mean	Stdv.
Green stem	17.3	2.0	43.1	34.5	14.1	8.8	24.4	6.8
Retted stem	19.7	10.1	44.8	68.4	9.7	5.2	19.6	12.1
Noils	27.6	5.5	23.1	25.5	13.7	10.0	37.4	7.0
Kotonina	30.8	4.7	21.2	17.6	11.9	6.8	36.2	4.7

Table 3. Chemical compositions and viscosities of flax fibres

Untreated fibres	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pectin (%)	Other (%)	Intrinsic viscosity (ml/g)
Green stem	59	9	3	8	7	1554
Retted stem	70	12	2	5	3	1874
Noils	79	8	2	2	3	2027
Kotonina	79	7	4	5	2	2039
Acid hydrolysed fibres						
Green stem	82	8	6	2	2	974
Retted Flax	82	7	4	5	3	1058
Flax Noils	84	7	3	4	2	399
Kotonina	84	8	4	2	1	487

Table 4. Chemical composition of hemp fibres

Untreated fibres	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pectin (%)	Other (%)	Intrinsic viscosity (ml/g)
0 Passes	74	11	6	4	7	1600
5 Passes	74	11	6	5	6	1617
12 Passes	77	11	3	3	5	1702
22 Passes	76	11	5	3	7	1767
Acid hydrolysed fibres						
0 Passes	83	10	2	3	4	634
5 Passes	83	10	2	3	2	626
12 Passes	82	10	3	3	5	556
22 Passes	82	9	5	3	2	548