Tensile and compressive properties of flax fibres for natural fibre reinforced composites

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Mechanical properties of standard decorticated and hand isolated flax bast fibres were determined in tension as well as in compression. The tensile strength of technical fibre bundles was found to depend strongly on the clamping length. The tensile strength of elementary flax fibres was found to range between 1500 MPa and 1800 MPa, depending on the isolation procedure. The compressive strength of elementary flax fibres as measured with a loop test lies around 1200 MPa. However, the compressive strength can be lowered severely by the decortication process. The standard decortication process induces kink bands in the fibres. These kink bands are found to contain cracks bridged by microfibrils. The failure behaviour of elementary flax fibres under compression can be described as similar to the failure behaviour of a stranded wire.

1. Introduction

Composites of thermoplastic polymers with natural fibres as reinforcing agents have in recent years received increasing attention in light of the growing environmental awareness. Especially the combination of the bast fibres of annual fibre crops like flax, hemp or jute with the relatively cheap polypropylene (PP) yields materials with an interesting price/performance ratio, which can be used in for instance interior parts in cars [1].

The mechanical properties of annual fibre/PP composites were studied by several groups [2–5]. By increasing the compatibility between the fibre and the matrix with for instance maleic anhydride modified PP the properties of these materials can reach a satisfying level. The full potential of annual fibres in composite applications, however, has up till now not yet been explored. Since annual fibres have rather intricate structures, the ultimate properties of annual fibre reinforced composites can not be predicted in the same way as for glass fibre reinforced composites.

Table I gives an overview of strength data of several annual fibres presented in literature by different authors. Flax is one of the fibres with potentially the best mechanical properties. Flax can be grown in the temperate climate zones of for instance Europe and the USA. As opposed to man-made fibres, the flax fibre is not a continuous fibre but is in fact a composite by itself. The circa 1 metre long technical bast fibres that are isolated from the flax plant consist of elementary fibres (Fig. 1) with lengths between 2 and 5 cm, and diameters between 10 and 25 μm. The elementary fibres overlap over a considerable length and are glued together by an interphase mainly consisting of pectin and hemicellulose, which is a mixture of different lower molecular weight branched polysaccharides. They are not circular but a polyhedron with usually 5, 6 or 7 angles to improve the packing in the technical fibre. The elementary fibres are single plant cells. They consist of a primary cell wall, a secondary cell wall and a lumen, which is an open channel in the centre of the fibre. The lumen can be as small as 1.5% of the cross section [6]. The primary cell wall is relatively thin, about 0.2 μm [7] and consists of pectin, some lignin and cellulose [8, 9]. The secondary cell wall makes up most of the fibre diameter. It consists of oriented, highly crystalline cellulose microfibrils and amorphous hemicellulose. The microfibrils are packed together in a fibrillar structure, the meso fibrils, with the fibrils oriented spirally at approximately $+10^\circ$ compared to the fibre axis [10, 11]. The fibrils presumably are glued together by a hemicellulose rich phase.

The highly oriented crystalline cellulose structure makes the fibres stiff and strong in tension but also sensitive towards kink band formation under compressive loading, analogous to, for instance, aramid fibres. The compressive strength of aramid fibres is only ca. 20% of their tensile strength due to the formation of kink bands [12]. The compressive strength of flax fibres has up till now, to our knowledge, not been reported.

In view of the intended application of flax fibres in composite materials with either thermoplastic or thermoset resins, it is vital to have a good insight in the mechanical properties of fibres that are available on the market. From Table I it is clear that there is a large scatter in the data given by some of the authors and also there are large differences between data measured on similar fibres by different authors. There are several reasons for this scatter which will be adressed in this paper.

The present methods to extract flax fibres from the plant include mechanical processes like breaking, scutching and hackling. These processes are optimised for the production of fine yarns for the linen industry. However, they are found to induce substantial amounts

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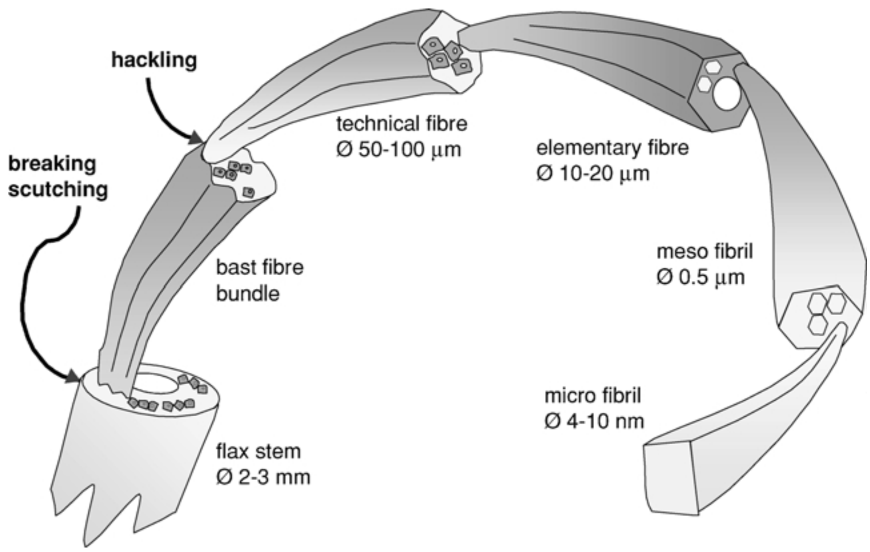


Figure 1 Composition and built of flax fibres.

TABLE I Tensile strength, expressed in MPa, of different natural fibres and of glass fibres, presented by a number of authors

	Kessler *et al.* [13]	Nova *et al.* [14]	Morton *et al.* [15]	Satyanarayana *et al.* [16]
Flax	400–1500	800–930	756	
Hemp		600–1100	658	
Kenaf		930		
Jute		540	434	533
Sisal		855		641
Ramie		585	826	
Glass	900–3500	1625	1913	

of damage in the form of kink bands in the fibres, thereby reducing their strength, not only in compression but also in tension. For the application of flax fibres in composites the presence of these kink bands might pose a serious problem. Apart from the presence of the kink bands, also the composite-like nature of the technical flax fibres will have an influence on their mechanical properties.

In this paper the mechanical properties of flax fibres, both isolated from the plant with the standard techniques, and carefully isolated by hand, are investigated in order to explore the ultimate properties of these fibres in tension as well as in compression.

2. Experimental

2.1. Materials

Flax (JS2-33-1995, Cebeco, NL) was warm water retted on lab scale. Part of the fibres was decorticated using pilot scale breaking, scutching and hackling procedures, part of the fibres was isolated from the plant by hand, taking special care not to damage the fibres unnecessarily.

2.2. Tensile tests

Tensile strength of the technical fibres at span lengths of 10, 25, 50 and 100 mm was measured on a BAC tensile machine, at a strain rate of 0.005 s^{-1}. Fibre tensile strength at 3 mm span length of both the technical and the elementary fibres was measured on a Rheometrics RSA II in tensile mode, using a static strain sweep programme. Individual fibres were glued onto small paper frames with epoxy glue. Specially developed clamps were used to clamp the frames, and prior to the measurement the sides of the frames were cut to allow free straining of the fibre. The strain rate was 0.005 s^{-1}. Diameter of the fibres was determined by investigating the fibre diameter in two perpendicular directions over the fibre length and taking the smallest diameter as a rectangle. Care was taken to select fibres for the test with a relatively homogeneous diameter. All strength measurements have been performed in 25-fold.

2.3. Compression tests

The compressive strength of the fibres was measured using an elastica loop test as developed by Sinclair [17]. Elementary fibres were placed in a loop under an optical microscope (with magnification 200×) and their ends were strained slowly. The creation and growth of kink bands was monitored and the dimensions of the loop during straining were measured. The elastica loop test was performed on 14 elementary fibres containing small kink bands induced by the decortication process and 8 elementary fibres free of kink bands.

2.4. Microscopy

Scanning electron microscopy was performed on a Philips 515 SEM. Environmental scanning electron microscopy was performed on an Electroscan ESEM 2010 at Cavendish laboratory, University of Cambridge, UK. Confocal scanning laser microscopy was performed using a Biorad MRC600 microscope from the University of Wageningen, NL.

3. Results and discussion

3.1. Tensile properties

Fig. 2 shows two typical stress-strain curves, for technical fibres clamped at 100 mm and 3 mm respectively. It is apparent that there is no great difference

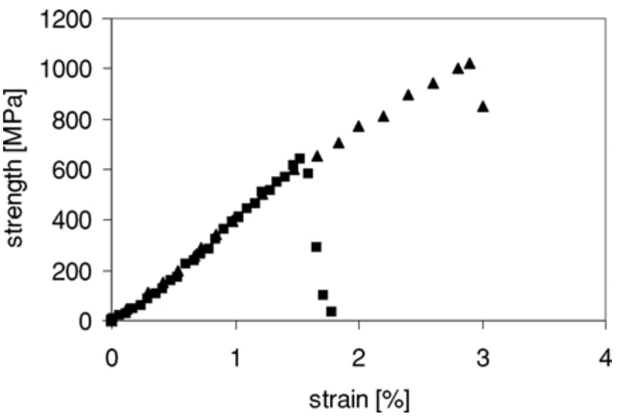


Figure 2 Typical stress strain curves for technical flax fibres. ▲ 3 mm clamping length; ■ 100 mm clamping length.

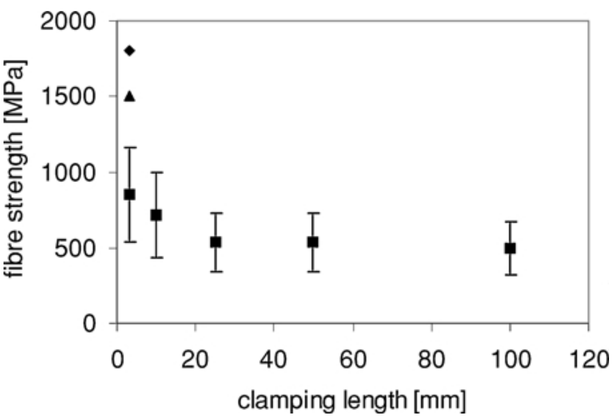


Figure 3 Fibre tensile strength versus clamping length. ■ Technical fibres; ▲ elementary fibres, standard decortication; ◆ elementary fibres, hand decortication.

in stress-strain behaviour between both fibres. However, there is a large difference in fibre strength between both fibres. Fig. 3 shows the fibre strength as a function of clamping length. The technical fibre strength is constant, approximately 500 MPa, down to a clamping length of 25 mm. Below 25 mm the fibre strength begins to increase towards a value of about 850 MPa at a clamping length of 3 mm.

Even though it is known that the strength of fibres generally increases with decreasing fibre length due to the reducing chance of the presence of critical flaws, it is unlikely that the specific dependency of strength on clamping length found in this case is caused by just this effect. Since the technical flax fibres are composed of shorter elementary fibres, it is likely that at large clamping lengths fibre failure takes place through the relatively weak pectin interphase that bonds the elementary fibres together. This gives rise to the plateau value in tensile strength of 500 MPa found for larger clamping lengths. Since the pectin interphase is oriented predominantly in the length direction of the fibre, it must break by shear failure. From the sharp fall in the curve in Fig. 2, it can be concluded that the failure in the pectin interphase is not a process involving large scale plastic flow, but happens rather instantaneously. The rise in tensile strength of the technical fibres at shorter clamping lengths is caused by a change in failure mechanism. At clamping lengths below the elementary fibre length, failure can no longer take place through the pectin interphase, but the crack must now run through the, stronger, cellulosic cell wall of the elementary fibres. The climb in strength is obviously gradual, due to the distribution in elementary fibre lengths and to the decreasing influence of critical flaws. The fact that the climb in strength starts around a clamping length of 25 mm supports this picture: elementary fibre lengths of flax fibres are usually quoted to lie between 20 and 50 mm, with the mean value around 30 mm.

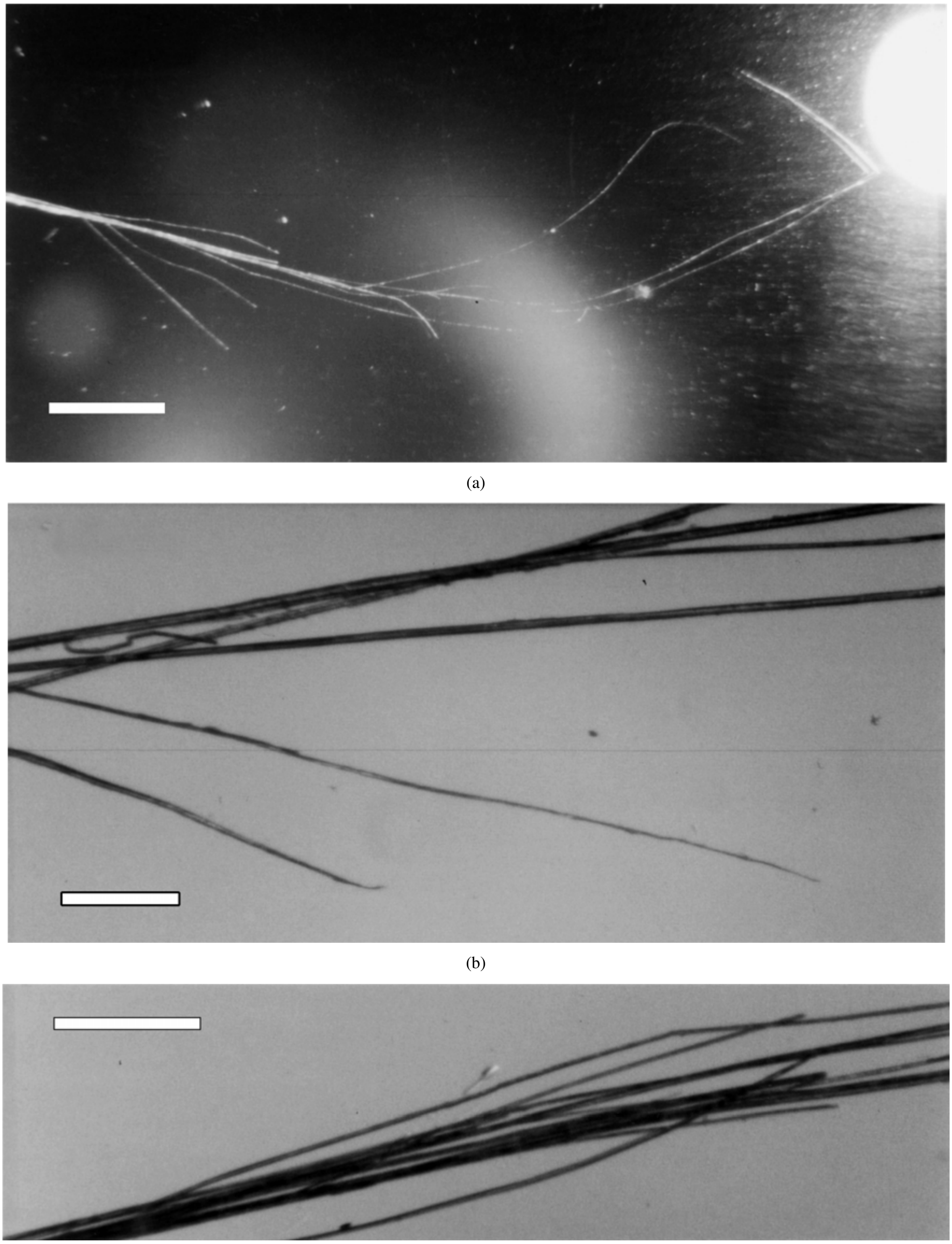
Also a closer look at the failed fibres partly supports this view. Fig. 4a shows the point of failure of a fibre tested at 50 mm clamping length. The fibre has split into elementary fibres and bundles of a few elementary fibres. Fig. 4b shows that indeed part of the elementary fibres is intact over the entire length up to the pointed fibre ends, and have separated completely through the pectin interphase. Some of the other elementary fibres, however, have broken halfway as can be concluded from the blunt elementary fibre ends visible in Fig. 4c. It is remarkable that approximately 6 elementary fibres have broken at the same spot, probably at this spot a kinkband was present over a large part of the fibre diameter.

The length over which the fibre splitting can take place is depending on the clamping length. For fibres which are tested at long clamping length the splitting usually is visible over a length up to circa 2.5 centimetres, similar to the length of the elementary fibres, giving the fibre the possibility to fail fully through the pectin interphase.

The triangular point in Fig. 3 gives the average tensile strength as measured on single elementary fibres, 1522 ± 400 MPa. This value is similar to the upper limit in tensile strength as reported by Kessler *et al.* [13] for the tensile strength of steam exploded flax. Sotton *et al.* [18] report a value of 2000 MPa for the strength of steam exploded elementary flax fibres.

The strength values discussed up to now were all measured on scutched and subsequently hackled fibres. To investigate the influence of the presence of kinkbands on the fibre strength, the strength of elementary fibres isolated by hand was determined as well. These fibres were found to be virtually free of kink bands. The mean fibre strength of the hand decorticated fibres is higher, 1834 ± 900 MPa, as compared to the value of 1522 ± 400 MPa found for standard decorticated fibres (Fig. 3), but also has a considerably higher scatter on the data.

The tensile strength of single elementary fibres is clearly substantially higher than the tensile strength of technical fibres at the same clamping length; the technical fibre strength is found to be 57% of the elementary fibre strength. This strength difference is most likely due to the bundle effect. Van der Zwaag [19] gives a method to derive the strength of a bundle consisting of a large number of filaments from a Weibull plot. A Weibull plot [20] of the fibre strength data, measured both on standard decorticated and hand isolated elementary fibres, is given in Fig. 5 (each point represents a single measurement). The Weibull modulus, m, is the slope of the line. For standard decorticated flax



(a)

(b)

(c)

Figure 4 Flax fibre tested at 50 mm clamping length. (a) Overview, scale bar indicates 2.5 mm, (b) Elementary fibres separated through the pectin interphase, scale bar indicates 20.7 cm, (c) Elementary fibres separated through the pectin interphase and broken halfway, scale bar indicates 0.5 mm.

fibres a Weibull modulus of 4.0 is found and for hand decorticated fibres a Weibull modulus of 2.2 is found. Following van der Zwaag [19] the bundle efficiency, ε, can now be calculated as:

$$\varepsilon = (em)^{1/m} \Big/ \Gamma(1 + 1/m) \qquad (1)$$

with e the base of the natural logarithm, Γ the gamma function and m the Weibull modulus. For the kinked, standard decorticated, fibres with a Weibull modulus of 4.0 this leads to a bundle efficiency of 60%. Even though in principle Equation 1 is only valid when there is no filament interaction in the bundle and when the bundle contains over 100 filaments—generally a technical fibre encloses up to 40 elementary fibres in its cross section—, the result is remarkably close to the efficiency of 57% found experimentally. For a hand decorticated bundle with a Weibull modulus of 2.2, the calculated bundle efficiency is 50%. Given a mean elementary fibre strength of hand decorticated

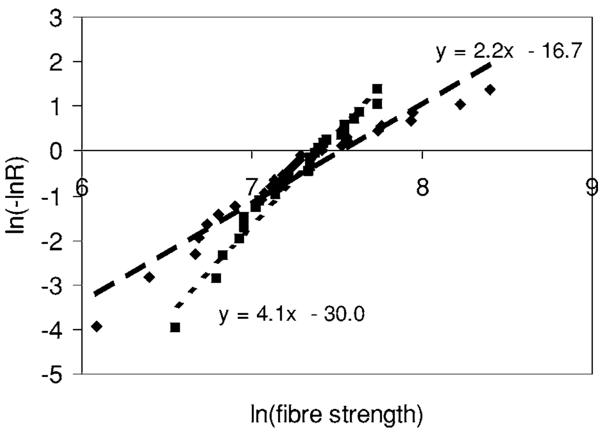


Figure 5 Weibull plot of elementary fibre strengths. ◆ Hand decorticated fibres; ▲ standard decorticated fibres.

fibres of circa 1800 MPa, the bundle strength would be approximately 900 MPa, similar as for standard decorticated fibres. This indicates that although the standard decortication process reduces the strength of individual elementary fibres, the strength of the technical fibre is hardly affected. It can now be understood why, for the linen industry, where the strength, fineness and homogeneity of the technical fibre are the major quality parameters, the fact that the standard decortication process damages the elementary fibres is of minor importance. For applications of the fibres in composites, however, the strength of the elementary fibre is an extremely important parameter.

The scatter in strength is much larger for the hand decorticated elementary fibres than for the standard decorticated elementary fibres. Thus, apart from lowering the mean fibre strength, due to the kink bands it induces, the standard decortication process also removes the weakest links, thereby reducing the scatter in fibre strength. It is interesting to note that nearly 50% of the fibres come off as short fibre waste, during the standard production process of long fibre yarns. One could further assume that the standard decortication process especially reduces the strength of the strongest fibres, since, when there is already a weaker spot in the fibre present, the introduction of extra kink bands will probably only lead to a limited extra drop in strength. When there is no weak spot present, any kink band that is introduced in the fibre will lead to a serious drop in strength. Worth mentioning in this respect is the fact that from the hand isolated fibres, some were found to be stronger than 2500 MPa with one measured value of even 4200 MPa. The scatter in tensile strength, however, poses a serious problem for the use of these fibres in composite materials. With the development of alternative decortication processes, especially aimed at producing fibres for the composite industry, care should be taken that the weakest fibres are removed during the process, without damaging the stronger ones.

3.2. Compressive properties

Measuring compressive strength of fibres is not a trivial problem. A possible approach is the use of the elastica loop test. The elastica loop test was originally devel-

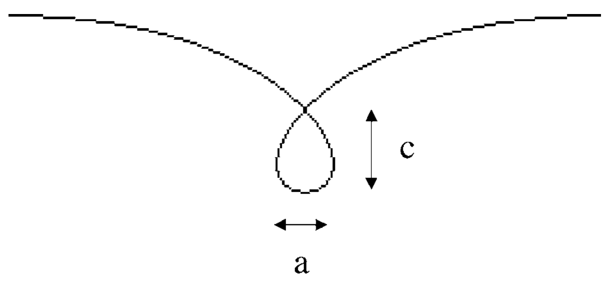


Figure 6 The geometry of the loop test.

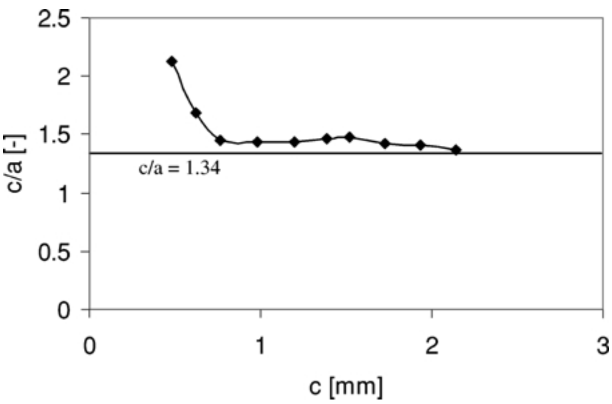


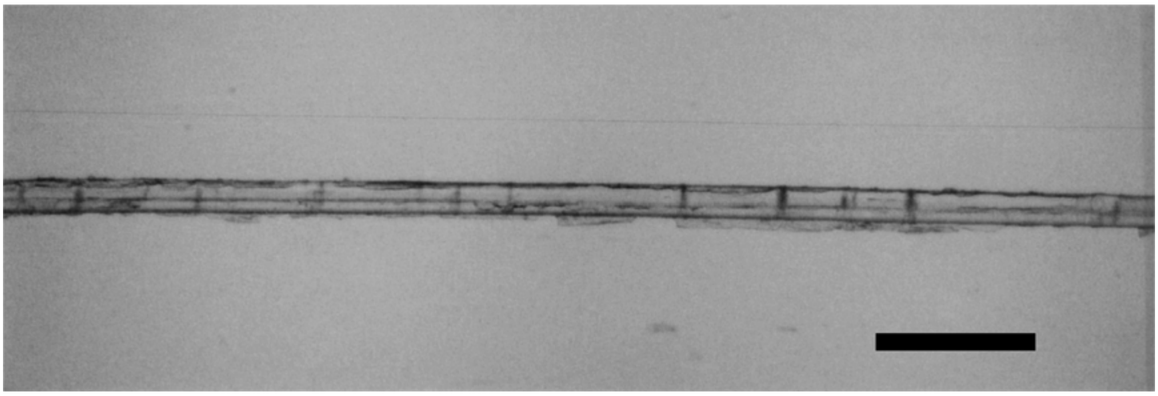
Figure 7 A typical plot of c/a versus c of an elastica loop test; c_{crit} is the c at which c/a starts to rise.

oped by Sinclair [17], to measure the tensile strength of glass fibres. Greenwood and Rose [12] and Peijs *et al.* [21] used the test to measure the compressive strength of man made polymeric fibres, like aramid and PVOH, which fail in compression rather than tension. In the loop test, due to the geometry of the loop (Fig. 6) the highest stress is found in the top of the loop. Homogeneous fibres therefore usually show either tension or compression failure in the top of the loop. Upon tightening the loop, the relative dimensions of the loop (c/a) will remain constant, around 1.34, until a non-linear deformation takes place in the top of the loop. The loop test subsequently takes the point of non-linear deformation as the point of failure. At that point c/a will increase and from plotting c/a against c (Fig. 7), c_{crit} can be determined which allows the stress at the moment of failure to be calculated as:

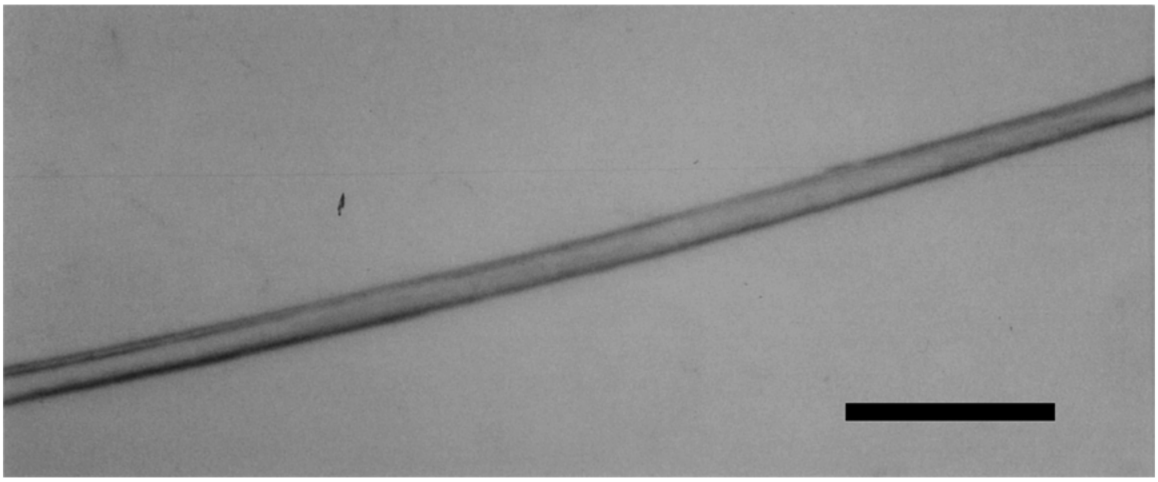
$$\sigma_{\text{fc}} = \frac{1.34 E_{\text{fc}} d}{c_{\text{crit}}} \tag{2}$$

with E_{fc} the compressive modulus of the fibre, usually assumed to be equal to the tensile modulus (and taken as 50 GPa, a typical value found for the modulus of technical fibres at 10 cm clamping length), and with d the fibre diameter.

A problem with standard decorticated flax fibres is the large number of kink bands they already contain, distributed more or less evenly over the fibre length (Fig. 8a). Hand isolated fibres are virtually free of kink bands (Fig. 8b), indicating that kink bands are not caused during growing, but are a result of the isolation process. A test on fourteen different single fibres led to nine fibres failing in the top of the loop giving a value for the compressive strength of 1200 ± 370 MPa.



(a)



(b)

Figure 8 Optical micrographs of elementary flax fibres. (a) Fibre containing kink bands. Scale bar is 16.3 cm. (b) Fibre without kink bands. Scale bar is 21.1 cm.

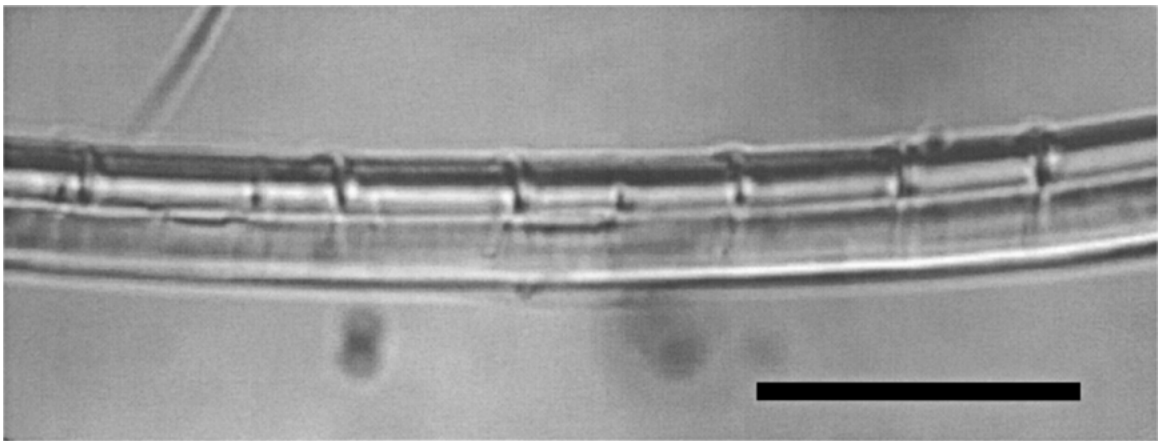


Figure 9 Optical micrograph of an elementary flax fibre in a loop with local fibre thickening at the compression side of the fibre. Scale bar indicates 50 μm.

Five fibres failed at arbitrary points along the loop at lower stresses. Since all fourteen fibres are damaged by the decortication process, the compressive strength values measured on these fibres can be assumed to form a lower limit.

In the loop test, irrespective of the presence of the kink bands, the loop often has the expected shape with (c/a) circa 1.34 at the start of the test. The point at which in the loop test failure is determined corresponds to the point at which one of the smaller kink bands in the top of the loop has grown so far that it extends over the whole fibre diameter.

In order to more closely examine the fibre deformation under compression, a few hand isolated fibres were tested in the loop test as well. The first deformation during the test which is visible in the optical microscope, are small black dots in the centre of the fibre axis in the top of the loop, starting at rather low stresses. Using Equation 2 and substituting c_{crit} by c, it can be calculated that these dots become visible at around 300 MPa. The black dots gradually grow in the direction of the compressive side of the fibre until around 600 MPa local thickening on the compressive side begins to occur (Fig. 9). Further tightening of the loop causes the

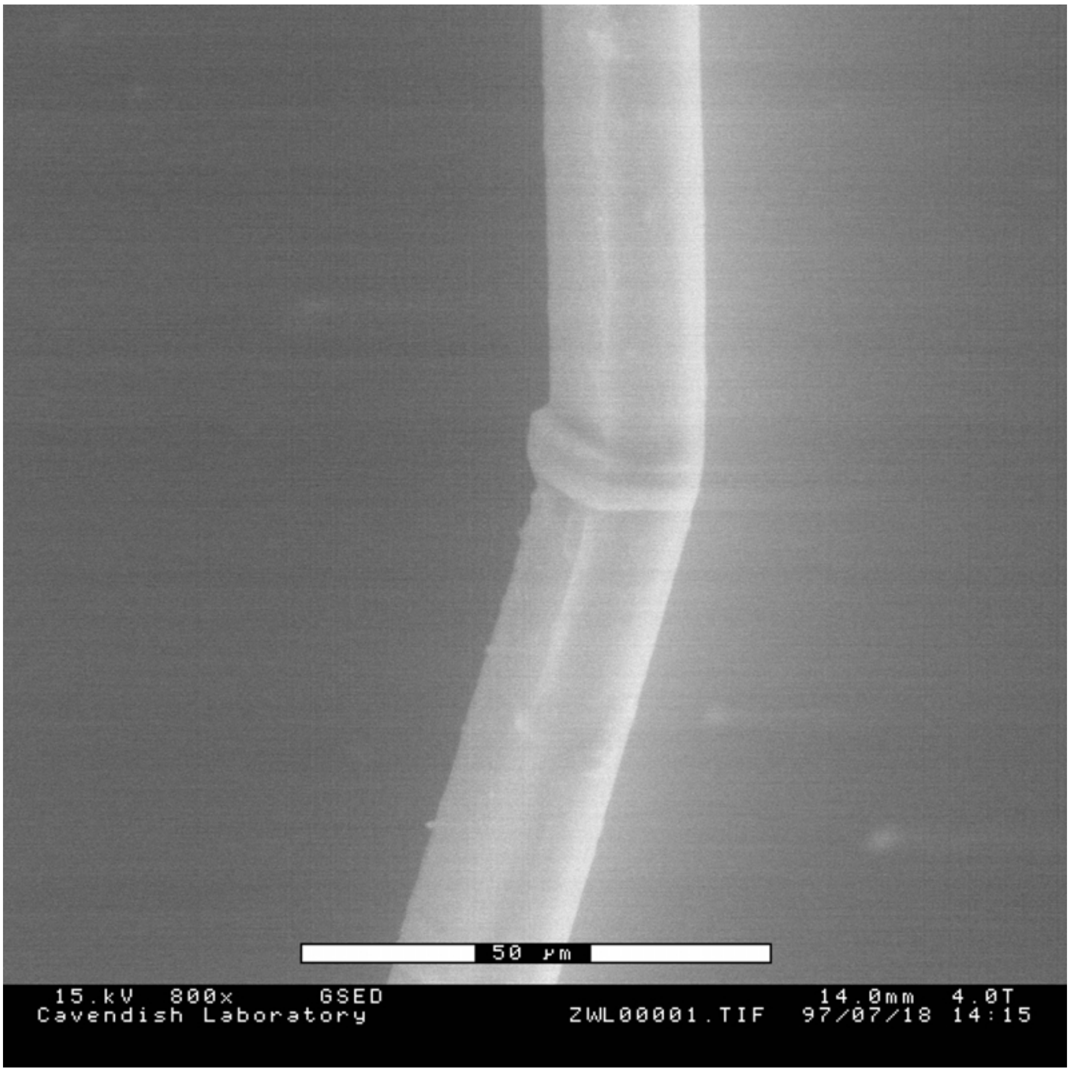


Figure 10 ESEM micrograph of a flax fibre which has just developed a full kink band. Scale bar indicates 50 μm.

fibre to undergo compressive failure, similar to the standard decorticated fibres and at similar c_{crit}. The few tests performed on hand isolated fibres do not indicate that there is a difference in ultimate compressive failure strength between standard decorticated fibres that fail in the top of the loop and hand isolated fibres. This can be expected when it is assumed that the process of kink band formation is similar during both the decortication process and the loop test, and the point of failure is determined as merely the point at which the kinking process has occurred over the full diameter of the fibre. It does however pose a question to the validity of the loop test for determining the compressive strength of fibres, since some small scale failure probably already occurs long before the loop shows large scale non-linear deformation.

An ESEM micrograph of an area which has just developed a full kink band in bending is shown in Fig. 10. The primary cell wall is still intact, although it has buckled outwards. The deformation in the primary cell wall has grown over the entire fibre diameter. The damage in the secondary cell wall obviously is not visible in this micrograph. A view of what happens during compressive failure in the secondary cell wall is shown in a CSLM micrograph in Fig. 11. The CSLM micrograph shows a section of the secondary cell wall of a hand isolated fibre, underneath the primary cell wall, which has deformed in a loop test and was subsequently re-straightened. The deformation in the fibre is localised in a number of narrow bands. The deformation bands form an angle of circa 80° with the fibre axis, i.e. they are perpendicular to the direction of the fibrils in the secondary cell wall. Furthermore, interfibrillar failure clearly has taken place within these deformed bands, the bands resemble a crack bridged by fibrils. The thickness of the bridging fibrils is circa 0.1–0.3 μm.

As discussed in the introduction, the secondary cell wall of elementary flax fibres has a composite-like structure. The SEM micrograph in Fig. 12 shows the cross section of a broken elementary fibre. It can be seen that the fibre has a more or less concentric layer-like structure. Furthermore, the thin primary cell wall—being the outer skin of the fibre—can be seen in this picture. The dimensions of the fibril conglomerates, by the authors called meso fibrils, is typically in between 0.2 and 1 μm. Fig. 13 shows a SEM micrograph from a technical fibre which has been buried in the soil during a number of days. The primary cell wall of this fibre has disappeared and the secondary cell wall of the elementary fibres with the fibrillar structure can be seen. The fibrils have the expected angle of 10° with the main fibre axis. Some longitudinal cracks are running

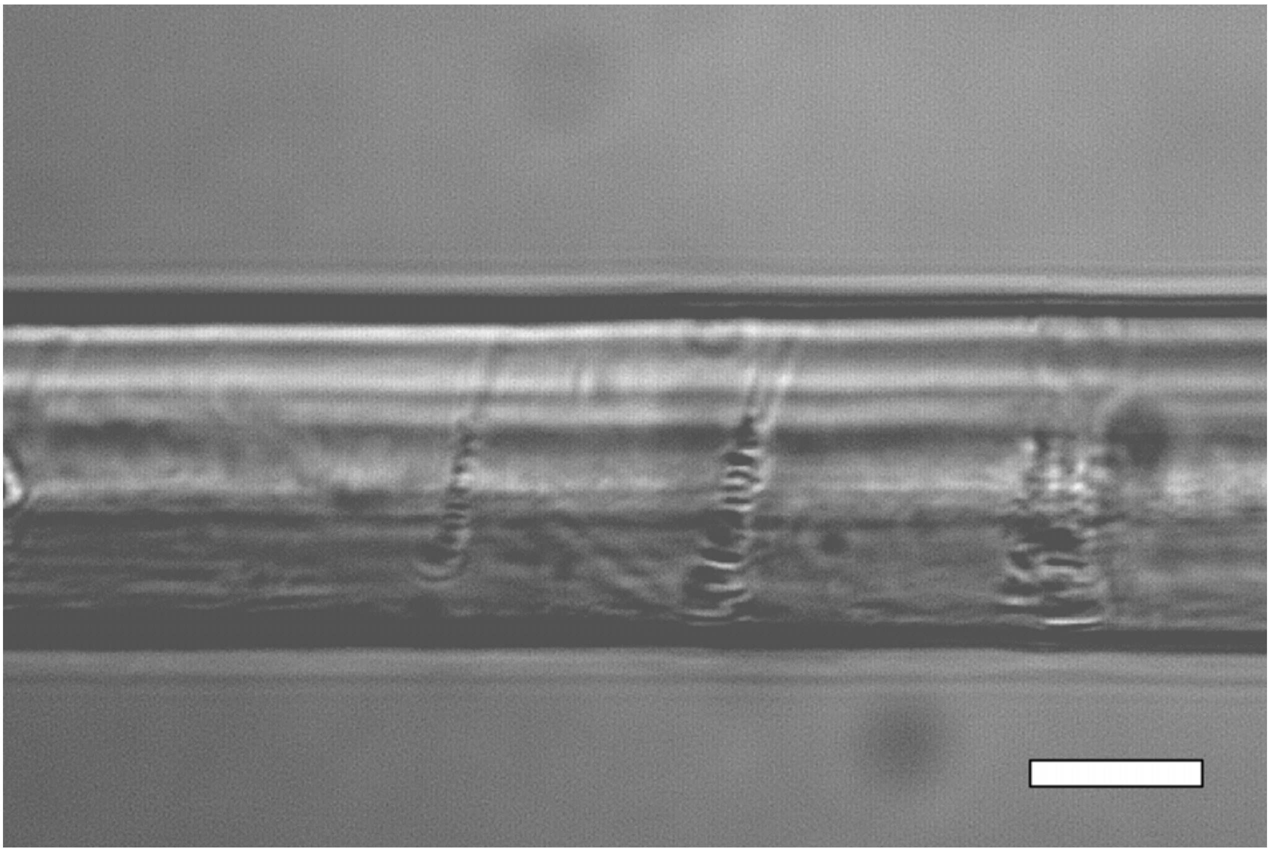


Figure 11 CSLM micrograph of kink bands formed during a loop test after re-straightening of the fibre. Scale bar indicates 21.7 cm.

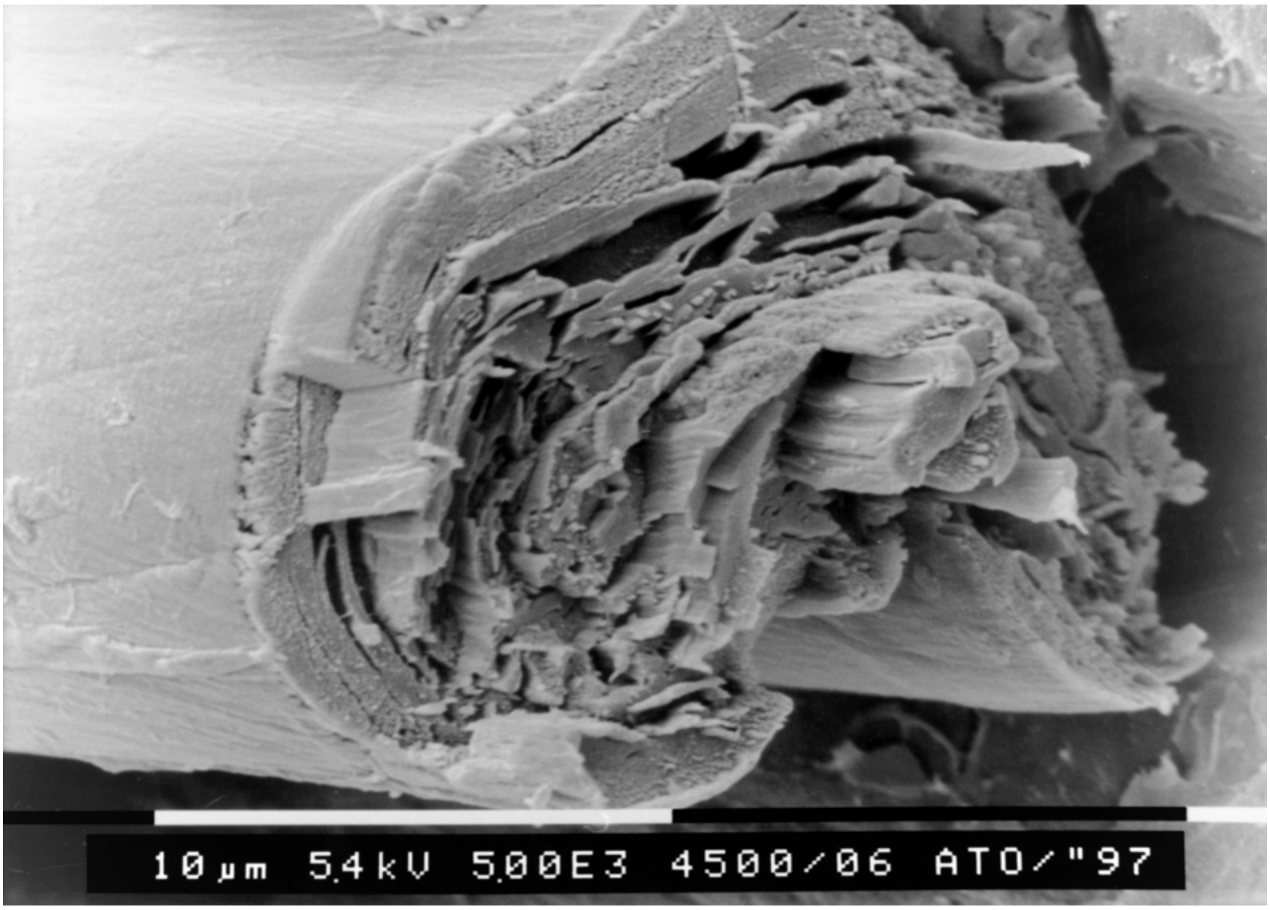


Figure 12 SEM micrograph of a flax fibre fracture surface. Scale bar indicates 10 μm.

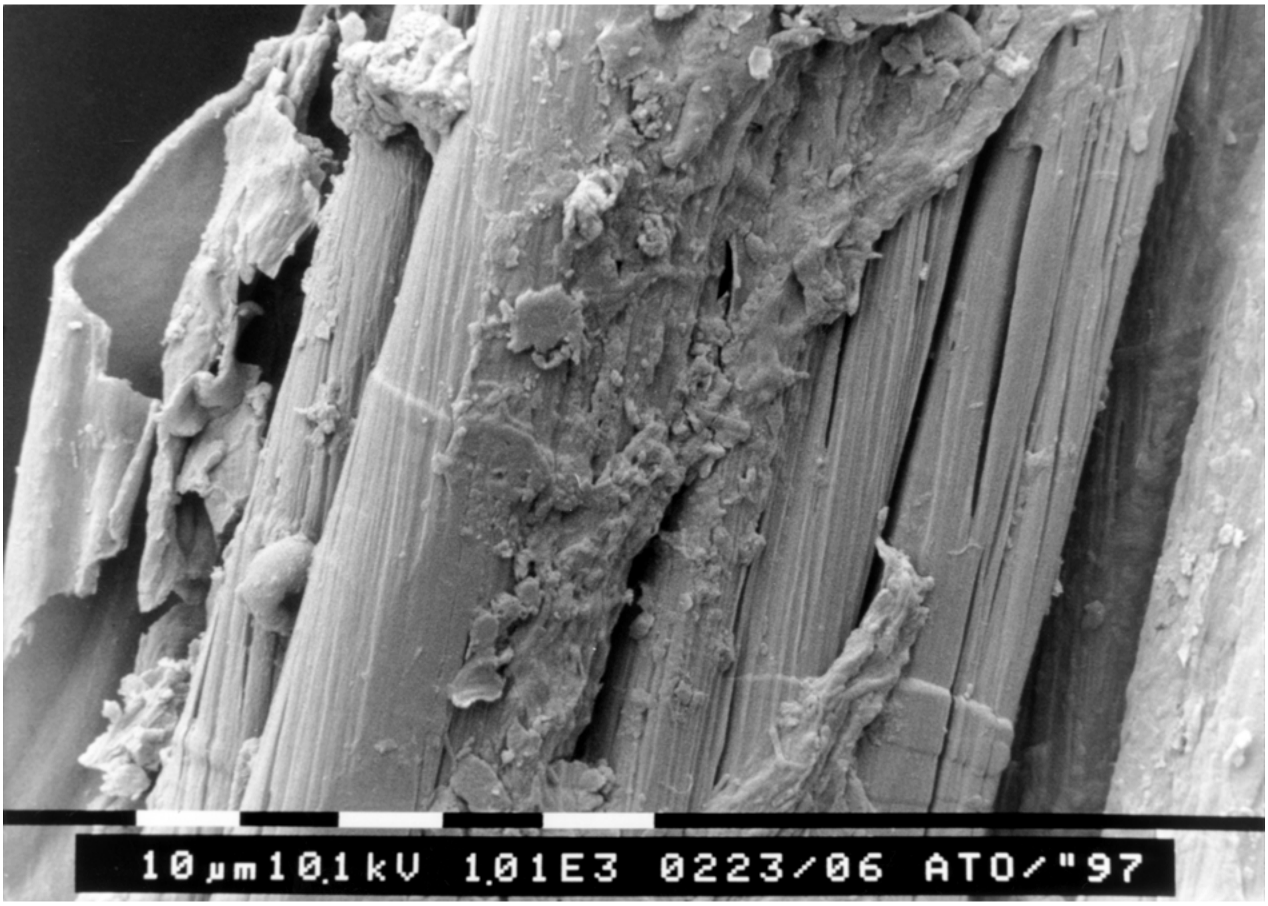


Figure 13 SEM micrograph of a technical fibre after removal of the outer primary cell wall during a short soil burial period. Scale bar indicates 10 μm.

between the fibrils. The fibril thickness seen in the secondary cell wall is circa 0.5–1 μm, somewhat larger than the thickness of the fibrils bridging the crack-like area in the CSLM micrograph (Fig. 11) and similar to the size of the meso fibrils in Fig. 12.

Williams *et al.* [22] give a model for the compressive failure of carbon fibres which also seems applicable to a first approximation to the mechanism of flax fibre failure under compressive loading. They depict the fibre as a stranded wire, in which the strands are slightly twisted. Since the wires have little lateral connection they buckle outwards under compressive strain. In flax fibres, the hemicelluloses that keep the fibrillar structure of the secondary cell wall together form relatively weak interphases. Under compressive loading the fibrillar structure of the secondary cell wall starts to buckle outwards, eventually causing the hemicellulose interphase to split. Apparently this is a very locally occurring process, the split zones appear to be restricted to small bands. Furthermore, the primary cell wall is not broken by this process.

A closer look at Fig. 11 gives more insight in the way the kink bands grow. It is clearly visible that the crack-like areas are wider at the compression side of the sample and slowly diminish in width towards the tensile side. During bending of the fibre, all kink bands slowly grow penetrating into the width of the fibre. Once one of the kink band has grown over the full width of the fibre, the stiffness of the fibre locally drops to a large extent, leading to the non-linear deformation in the loop test.

It can be expected that in flax which has undergone the standard decortication, kink bands in a wide range of growth stage are present. It is possible that, although there are many kink bands present, none of them have grown over the full width of the fibre. This could explain why these fibres in the loop test have the expected shape with (c/a) of 1.34 and why the loop test gives little difference in the compressive strength for standard and hand decorticated fibres. It does however pose the question how standard decorticated fibres are going to behave in pure uniaxial compression, probably the fibres will have little resistance against uniaxial compressive deformation due to the damage they contain.

Even though the results of the loop test may not give a good indication of the behaviour of the fibres under uniaxial compression, the value of ca. 1200 MPa for compressive strength can be compared with data from other authors using the same test for other fibres. Peijs *et al.* [21] have measured compressive strength values with the loop test on various highly crystalline polymers. They find a compressive strength of 180 MPa for gel-spun polyvinylalcohol (PVOH), which is ca. 10% of the fibre's tensile strength. For high-performance polyethylene fibres (HP-PE) they quote a value for the compressive strength of ca. 60 MPa, which is only 2% of the fibre's tensile strength. And finally, for aramid fibre (Twaron^{TM} HM, Akzo) they find a compressive strength of 600 MPa, which is about 20% of the fibre's tensile strength.

It is clear that compared to these high performance polymer fibres flax fibres have a very high compressive strength. Furthermore, the ratio between tensile and compressive strength for flax—80% for the standard decorticated fibres—is much higher than for the high performance polymer fibres. In this light, the extremely high values we sometimes measure for the hand decorticated fibre tensile strength (up to 4200 MPa), maybe is the strength the fibres can reach, given that they do not contain any damage. Therefore, in case one succeeds to develop a decortication process that is less damaging, flax fibres might possibly be considered as truly high performance fibres.

4. Conclusions

Tensile properties of flax fibres depend bi-linearly on the clamping length, due to the composite-like structure of technical flax fibres. This could be one of the underlying reasons for the enormous scatter in flax fibre tensile strengths reported in literature. Single elementary flax fibres have considerably higher strengths than technical fibres. The elementary fibre strength was found to depend also on the decortication method used. Compressive strength of elementary flax fibres as measured in the loop test is approximately 80% of their tensile strength. Due to the fibrillar structure of the secondary cell wall of the elementary fibres they fail in compression due to kink band formation. Since the primary cell wall of the fibres is only slightly affected by the kink band formation, it is difficult to predict the extent of fibre damage by just examining the fibre surface.

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