



Assessing the effect of fibre extraction processes on the strength of flax fibre reinforcement



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ABSTRACT

A number of factors impede the direct translation of fibre properties from plant crop species to natural fibre composites. Commercially available fibre extraction processes introduce defects and degrade the mechanical properties of fibres. This study reports on a novel image based approach for investigating the effect of fibre extraction processes on flax fibre bundle strength. X-ray micro Computed Tomography (μ CT) was coupled with uniaxial tensile testing to measure the in-situ fibre bundle cross-section area and tensile strength in flax plant stems. The mean tensile strength result was 50% higher than that of the fibres extracted through the standard commercial process. To minimize fibre damage during fibre extraction, a pre-treatment was proposed via saturating flax plant stems in 35% aqueous ammonia solution. By environmental scanning electron microscopy (ESEM), it was evident that ammonia treatment significantly reduced the extent of damage in flax fibre knots and the optimum treatment parameter was identified.

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1. Introduction

Cellulosic plant fibres, such as flax fibres, offer a sustainable resource as a composite reinforcement with advantages of low embodied energy and bio-degradability compared to man-made glass fibre. Although flax fibre is reported to have a specific stiffness and strength compared to glass fibre [1,2], flax fibre reinforced composites are yet to achieve the consistent properties suitable for industry-scale structural applications. There has been considerable research in aiming to understand and optimize the various production stages of natural fibre reinforced composites. The tensile properties of assembled fibre bundles for composite applications have previously been investigated [3–6]. These studies focused on the optimal fibre twist to balance between yarn stability/processibility and composites strength. To determine the tensile properties of the fibre bundle, it is necessary to quantify their cross-sectional area. In previous studies, bundle cross-sectional area was approximated based on the bundle linear density and the fibre density. Since there was no direct measurement of the cross-sectional area, the reported tensile properties of the fibre bundle were less reliable for consistent comparison [7]. To characterize the tensile properties at the single fibre level, the researchers made the assumption

that fibre cross-section was uniformly circular [2,8], or a rectangular [1] there by simplifying the measurement of fibre diameter. This approximation, demonstrated by Thomason et al. [7], could result in over 100% error from the true fibre cross-section size.

Retting and scutching of flax are the established processes for flax fibre extraction. Both processes introduce certain alterations of the flax fibre. Retting, including water retting and dew retting, introduces enzymes to degrade pectin around flax fibres resulting in separation of flax plant fibres. Overexposure to enzymes during retting can risk degradation of the fibres [9] with the degree of retting influencing the tensile properties of single fibre and short fibre polymer composites [10]. Understanding the impact of these processes on fibre performance may lead to more efficient approaches of fibre extraction. The processes of retting and scutching on flax fibres has been studied by using flax fibre reinforced composites [11]. One of the degradation mechanisms is the formation of kink bands due to fibre buckling [1,2]. The current study applies non-destructive X-ray micro Computed Tomography (μ CT) as a direct experimental approach to quantify fibre bundle strength in flax plant stems and specifically measure irregular fibre cross-sections. Aqueous ammonia solution has shown potential as a biomass pre-treatment agent for biofuels [12] and animal feed [13] where it is capable of altering cellulose crystalline packing and dissolve lignin in the biomass [14]. The present study investigates the ammonia treatment on flax plant stems to reduce damage susceptibility during fibre extraction.

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2. Materials and methods

2.1. Materials

Flax plants were harvested and collected from the farm 'Flaxland' in Gloucestershire, U.K. For the current comparative study, three different Batches I, II and III from the same flax variety were used. Batch I was a yield from Year 2011, completed with the dew retting process. Batch II and III were from Year 2012 following the dew retting with application of Roundup desiccant – an alternative for the cooler and wetter climates [15]. Due to the unfavorable weather in 2012, Batch II and III were 150–200 mm shorter than Batch I (in an average length of 1100 mm) from the previous year. Batch II and III plants were harvested in 5 weeks apart respectively in September and October 2012, resulting in a different retting level. The plants were stored in the laboratory at 18 °C and a relative humidity of 75% for four months before the experiments. For the ammonia pre-treatment experiment, 35% concentrated aqueous ammonia solution was supplied by Fisher Scientific.

2.2. Coupled μ CT and uni-axial tensile test

Flax plant stem cross-sections were imaged by SEM (Fig. 1). In order to achieve a flat smooth cross-section for SEM imaging, flax

plant stems were sectioned using a cryostat at $-21\text{ }^{\circ}\text{C}$ to give $10\text{ }\mu\text{m}$ slices. To rigidify flax plant tissues for slicing, the specimens were soaked in water for 48 h and embedded in an Optimal Cutting Temperature (OCT) matrix in the cryostat. After cutting, the specimens were defrosted and washed with water. The specimens were dried at ambient atmospheric conditions for one week before coating a 22 nm thick layer of gold for SEM. A beam current of 10 kV with a magnification of $\times 2500$ was used for the SEM scans. The field of view for each scan was shifted swiftly to avoid overheating of the sample by the electron beam.

Fig. 2 illustrates the experimental procedure for measuring fibre bundle strength in flax plant stems. Three Sections (a, b and c) were cut into 120 mm lengths from each plant stem. Bark and fibre bundles were removed carefully by hand from Section b to only retain the woody core. Fixed at a gauge length of 40 mm, each specimen was prepared with the insertion of steel cores and polyurethane tubing at both clamp ends. Steel cores with diameters of 0.5 mm, 1 mm, and 1.5 mm were used to fit the various sizes of flat plant samples. The presence of steel cores was only at the clamp length, with no interference of the tensile tests. Araldite[®] epoxy resin acted as binder between polyurethane tubing and flax plant at the clamp ends. This configuration proved effective during the tensile test as no premature fibre damage at the clamped areas was observed with the majority of fibre bundle breakage located away from the clamps.

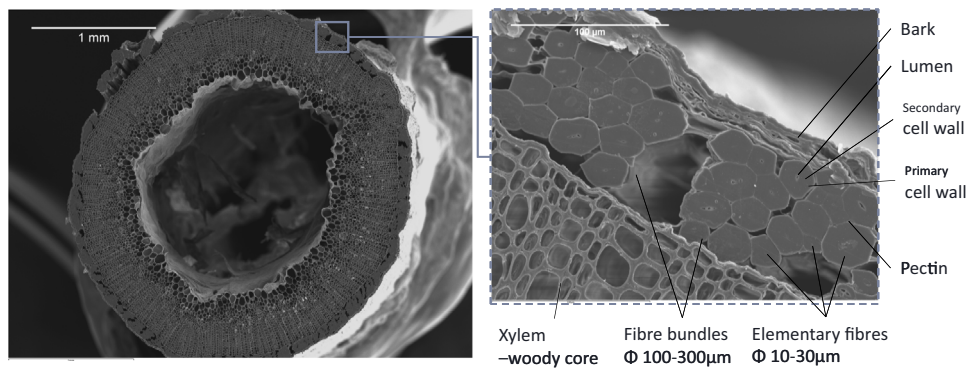


Fig. 1. Composition of the flax plant stem in cross-section revealed by SEM images. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

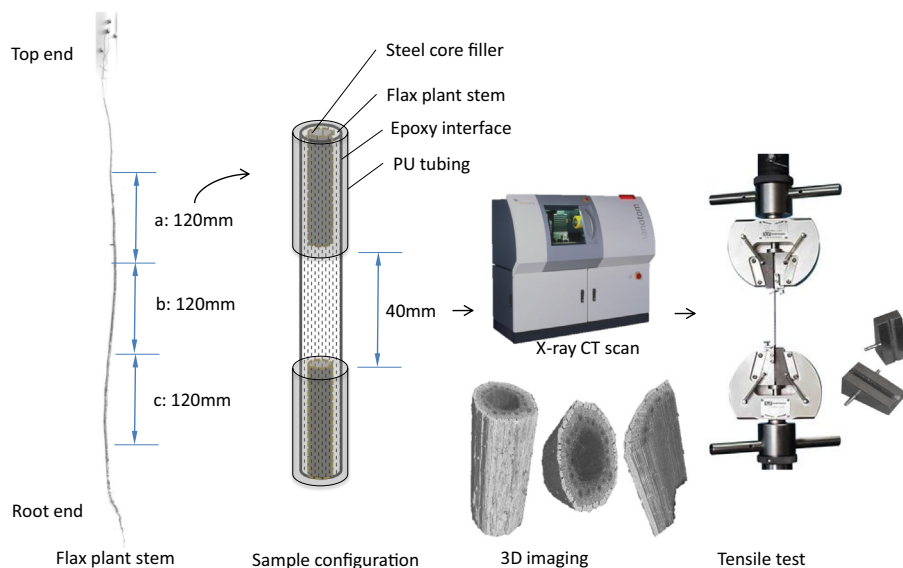


Fig. 2. Experimental procedure for characterizing fibre bundle strength in flax plant stems. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

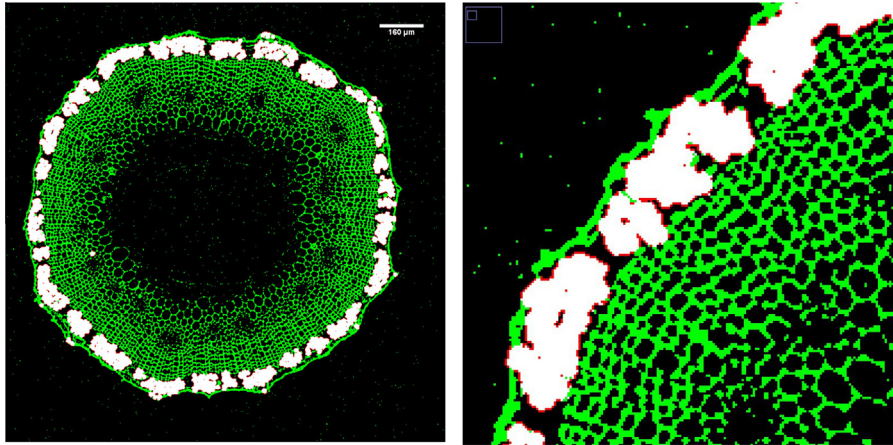


Fig. 3. An X-ray μ CT image of flax plant stem; the image was segmented in Fiji to isolate the fibre bundles for accurate measurement of fibre cross-section area. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Before the tensile tests, 15 stems from Sections a and c of Batch I were scanned using a GE Phoenix Nanotom 180NF X-ray CT system (GE Inspection Technologies GmbH, Wunstorf, Germany). Scans were conducted with an X-ray acceleration energy of 50 kV and 180 μ A current, collecting 1200 projection images over a 360° rotation of the sample which resulted in a scan time of 20 min. The spatial resolution of the scan was 4 μ m/voxel, – the diameter of flax fibre ranged from 10 μ m to 30 μ m as shown in Fig. 1.

X-ray μ CT scan images were used to quantify the cross-sectional area of fibre bundles in plant stems over the gauge length of each tensile test sample. The image volumes of flax plant stems, as

shown in Fig. 2, were processed in Fiji [16], an open source image processing package. The procedures of grey scale thresholding and local thickness mapping in Fiji were applied to isolate the fibre bundle from the woody core and bark, enabling the cross-sectional measurement of the isolated fibre bundles. Fig. 3 demonstrates the imaging process with the thresholded area in white representing the fibre bundles.

The μ CT image based measurements were calibrated by geometric features with known dimensions, such as the diameter or thickness of the sample. Error arising from the applied image processing was estimated by evaluating the sensitivity of the

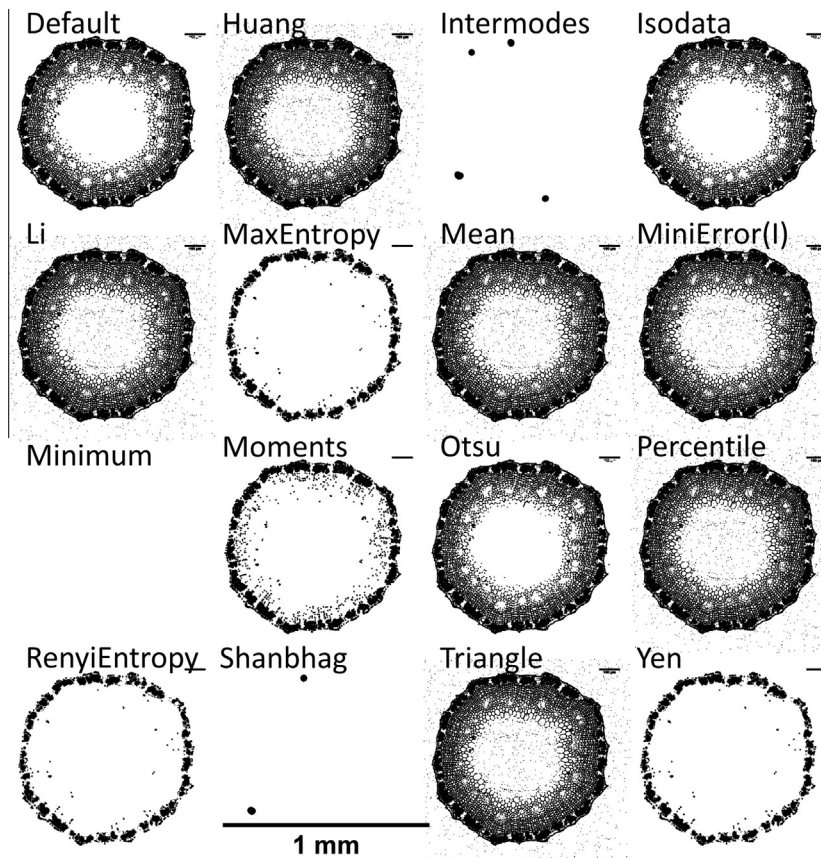


Fig. 4. Segmented X-ray μ CT images of flax plant cross-section with the results from various automatic threshold algorithms in Fiji.

threshold algorithms in Fiji. An automated threshold algorithm was preferred in order to avoid user dependent bias. A montage of segmentations based on various threshold algorithms is shown in Fig. 4. Nine out of sixteen algorithms gave visually similar results by different threshold grey values ranging from 65 to 68. As it is hard to judge optimum segmentation visually, this narrow range of threshold values from the automated algorithm is treated as a source of uncertainty. By applying the upper and lower bound of the threshold range, the standard deviation in the cross-section area measurement was quantified as $\pm 7\%$.

The tensile test was conducted on an Instron 5900 Series Universal Testing machine with a set of 2716 Series wedge action grips and a load cell capacity of 5 kN. Both ends of the specimens, as configured in Fig. 2, were held in place by V-serrated faces during the tensile test. The tensile forces were measured in accuracy ± 0.025 N. No attempt was made to obtain a strain measurement, because of difficulties in applying an extensor metre or digital image correlation on the flaky bark surface of flax plant stem. Hence the comparison would focus on the ultimate strength of flax fibre bundles. Sections a, b, and c of fifteen stems from each batch were tested by this method. The measured force in a sample of Sections a and c were carried by both fibre bundles and the woody core. To obtain the load solely acting on fibre bundles, the peak force measured in Section b was deducted from that from Sections a and c. This was based on the assumption that the same woody core existed in neighbouring Sections a and c. This assumption was verified by the tensile tests of the woody segments of Sections a, b, and c from three plant samples. The measured peak force of the woody core within the same plant had the standard deviation of $\pm 2\%$ of the peak force of the plant stems.

2.3. Coupled ESEM and knot tying fibre test

During the fibre extraction process, the most critical load case on fibre degradation can be simplified as kinking band formation in the bending mode [17]. The knot tying test is a qualitative yet effective experiment on fibre flexural strength and failure mechanisms. In order to study the effects of ammonia pre-treatment on damage tolerance of flax fibre, the experiments were set to capture the damage mechanisms of fibre knots via ESEM imaging. Five flax plant stems from the 2012 harvest were used. Each stem was divided into four specimen groups undergoing the pre-treatments, as described in Table 1. The saturation time was chosen on the following basis: Group B follows the reported processes for natural fibre treatment in liquid ammonia [18–20]; Group C refers to the saturated infusion time of flax fibre with ammonia aqueous solution [21]; and Group D refers to the estimated time of lignin being partially dissolved in aqueous ammonia solution [12].

After the plant stems were air dried and fibres were extracted manually with care, single fibres and fibre bundles were tied into simple knots using a pair of thin straight ended forceps under a 10 \times magnifying LED lamp. Five samples were prepared in three knot types, i.e. single fibre loose knot, single fibre tight knot and fibre bundle tight knot for each of Groups A, B C and D. In total 60 specimens were examined under ESEM (the model Philips XL30 FEG).

Table 1 Specimen groups of flax plant stems for ammonia pre-treatment.

Group A	Group B	Group C	Group D
Non-treatment	Dip in 40 ml 35% ammonia solution for 10 s at 18 °C	Dip in 40 ml 35% ammonia solution for 41 min at 18 °C	Dip in 40 ml 35% ammonia solution for 24 h at 18 °C

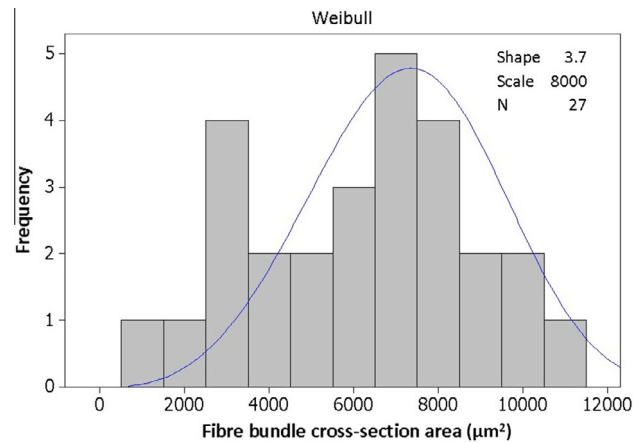


Fig. 5. Measurements of fibre bundle cross-sectional area fitted by Weibull distribution. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

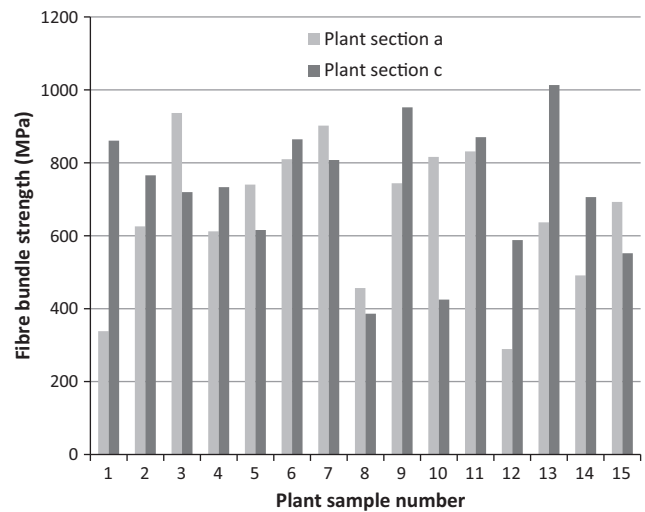


Fig. 6. Measurement of fibre bundle strength for thirty specimens following X-ray μ CT scanning.

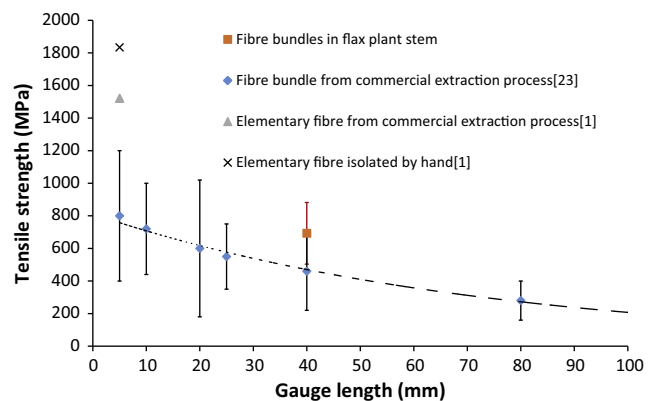


Fig. 7. Tensile strength of flax fibre bundle from the current test of flax plant compared with reported tensile strengths of flax fibres. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

3. Results and discussion

3.1. Tensile strength of flax fibre

By counting individual fibres from the high resolution SEM images, such as the example in Fig. 1, there were approximately 1200–2000 fibres in each plant stem cross-section. Following the μ CT image processing procedure, the measured cross-sectional areas of fibre bundles were obtained for 2×15 samples from Batch I. It was observed that the cross-sectional area of fibre bundles

seemed to follow a Weibull distribution in a single flax plant stem, as seen in Fig. 5.

The variation in the quantity of fibre count and the cross-sectional area along the flax stem was reported previously [22]. Yet there was no direct mechanical measurement to correlate the fibre bundle strength with the different locations along the flax stem. In the present study, the total cross-sectional area, together with the measured peak force isolated from the tensile test, was used to calculate fibre bundle strength. The woody core Section b had an average peak force of 32 N. The intact flax stem samples had the

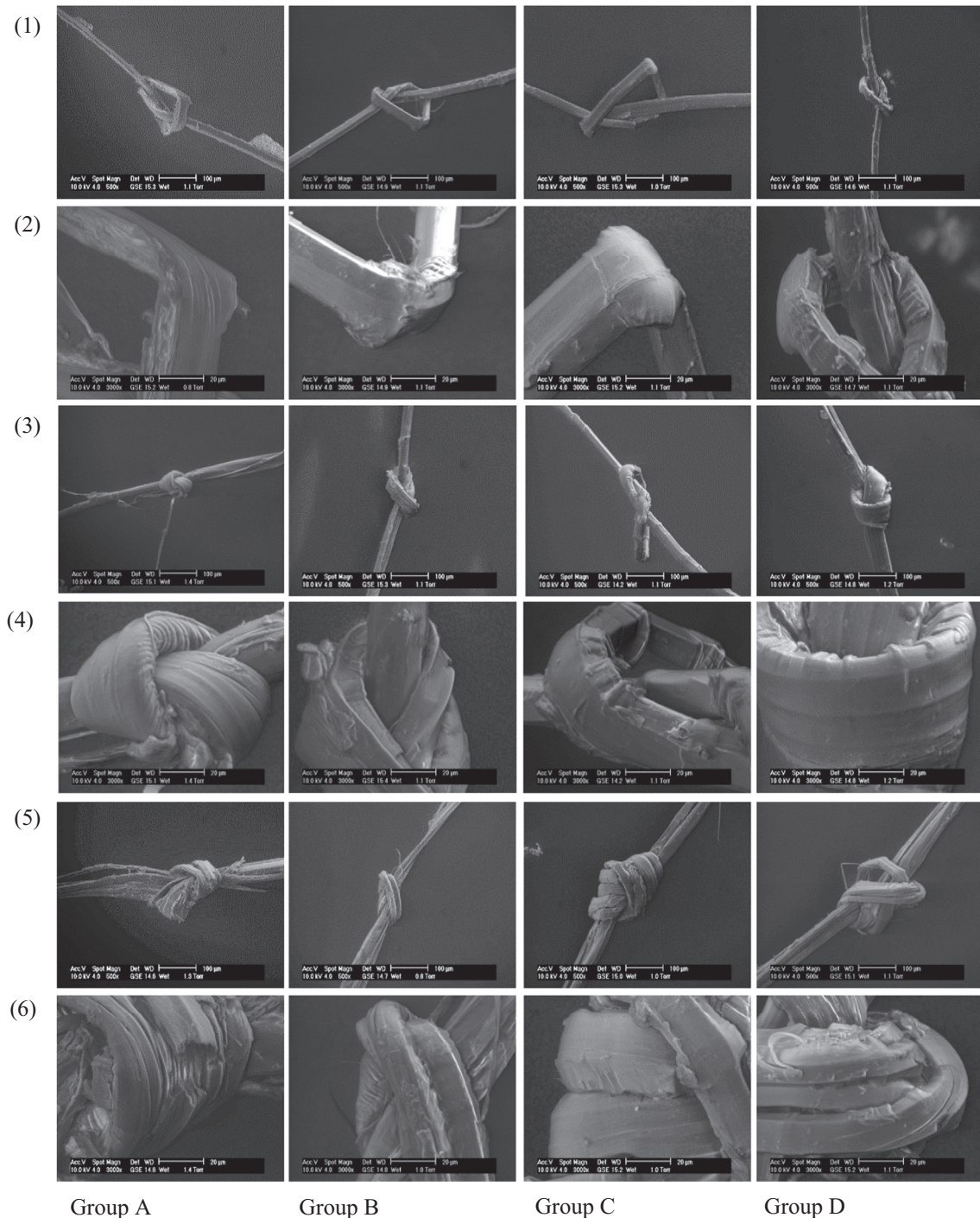


Fig. 8. ESEM images of flax fibre knots of Groups A, B, C and D under the pre-treatments described in Table 1; Rows (1) and (2) single fibre loose knots, (3) and (4) single fibre tight knots, (5) and (6) fibre bundle knots. The image scale bar is 100 µm for (1), (3) and (5), while 20 µm for (2), (4) and (6).

average peak forces of 187 N in Section a and 243 N in Section c. Fig. 6 plots the strength of fibre bundles for Sections a and c in each of 15 stem samples from Batch I. It was clear that fibre bundle strength differs by its location along the same plant stem and varies from plant to plant. Overall the measured mean strength was higher in Section c ($699.9 \text{ MPa} \pm 182.8 \text{ MPa}$) than Section a ($661.5 \text{ MPa} \pm 196.5 \text{ MPa}$). The previous study [23] found: (1) flax fibre diameter decreased from the bottom to the top of the plant stem; (2) elementary fibre strength increased from the bottom to the top of the plant stem. This was explained by the smaller the fibre diameter, the lower likelihood of defects and higher strength of elementary fibre [22]. Our results suggests an opposite trend of decreasing fibre bundle strength from the bottom to the top of the stem. This may well be explained by the distribution of fibre length along the flax stem however further investigation is required to confirm whether the average fibre length decreases from the bottom to the top of the plant.

As shown in Fig. 7, the measured tensile strength of fibres on average was 50% higher than the fibre bundles from the commercial fibre extraction processes [24], by comparison at the same gauge length of 40 mm. The large standard deviation implies strong variability in flax fibre. It is worth noting that the coupled μCT and uni-axial tensile test was equivalent to measurement of around 50,000 fibres with high fidelity in determining fibre cross-sectional area, confirming both approaches. This suggests the current fibre extraction technique significantly degrades fibre performance which is coherent with the degradation by 23% of elementary fibre due to the extraction process [1] (Fig. 7). This evidence shows that there is a potential to improve fibre strength via novel fibre processes, such as the ammonia pre-treatment being investigated in the present study.

We also focused on the effect of retting by comparing Batch II and III samples. The tensile tests measured average peak force as $244 \text{ N} \pm 57 \text{ N}$ for Batch II and $215 \text{ N} \pm 40 \text{ N}$ for Batch III. Fibre strength decreased by 10% from Batch II to III when flax plants were left retting for a further 5 weeks indicating the deleterious effect of over-retting. Over the period of 5 weeks, enzymes released by fungi start to degrade flax fibres, after breaking the surrounding barrier of pectins [25]. Optimization of the retting process to permit improved fibre separation whilst maintaining fibre strength would be worth future investigation as it has potential applications in current industrial practices.

3.2. Flexural strength of flax fibre

The collation of ESEM images in Fig. 8 illustrates the comparative flexural strength and failure modes of flax fibres with the varied ammonia pre-treatments. Row (1) in Fig. 8 shows the images of simple loose knots with a sharp bend observed for fibres from Groups A, B and C where as Group D displayed a smoother loop. Row (2) shows a higher resolution view of these bends with $5\times$ magnification (the scale bar $20 \mu\text{m}$). Similarly Rows (4) and (6) are the magnified views of the specimens in Rows (3) and (5). For the loose knots in Rows (1) and (2), the images reveal fibre failure modes in great detail. Untreated fibre knots (Group A) tended to collapse by axial splitting and extensive collateral fibrillation. Following a 10 s submersion in the ammonia solution (Group B), fibre fibrillation was significant reduced and the damage was more localized. After 41 min in the ammonia solution (Group C), the pre-treated fibre experienced no splitting and no fibrillation around the sharp bend and after 24 h in the ammonia solution (Group D), the fibre did not buckle while forming a smooth loop. This observation indicates that the fibre becomes more flexible and transversely tougher to withstand local buckling after the ammonia pre-treatment.

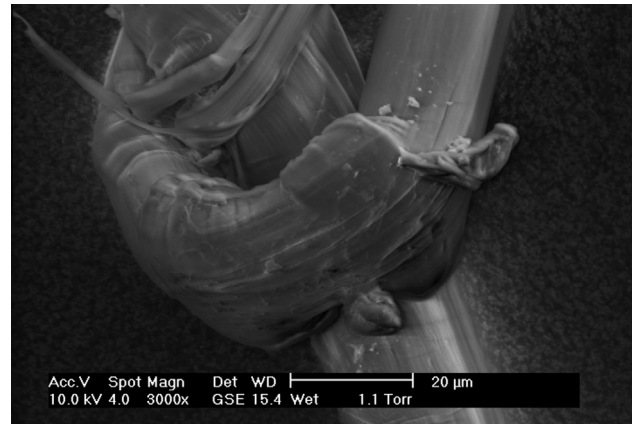


Fig. 9. ESEM image of a 24-h ammonia pre-treated fibre knot.

The evidence from single fibre tight knots (Rows 3 and 4) and fibre bundle tight knots (Rows 5 and 6) reinforce this observed trend. For the untreated fibre (Row 4, Group A), alongside with the axial splitting on the outer surface, there were dense kink bands/crease formation (5 crease per $10 \mu\text{m}$) around the inner surface of a tight knot. There was a reduced number of kink bands after the ammonia treatment, for example, 1 kink band per $15 \mu\text{m}$ (Row 4, Group D). In the case of fibre bundles with tight knots, fibres had severe cell wall fracture and splitting for untreated fibres (Row 6, Group A), while the damages were superficial for the ammonia treated fibres (Row 6, Groups B–D). Overall, the observation from Fig. 8 suggests that Group C had the most pliable fibres with the least damage in the knot tests.

Fig. 9 shows an ESEM image indicating the detrimental effect of a prolonged exposure time to the ammonium solution. In this case, the primary cell wall of the 24-h pre-treated fibre from Group D was completely degraded.

Cellulose is the main constituent of flax fibre cell walls. The interchangeable crystalline packing from native or natural Cellulose I to Cellulose III, using ammonia is well documented [18,26]. Liquid ammonia has been applied as an industrial process to improve crease resistance in easy care natural fibre garments [20,27]. The alteration of cellulose intermolecular hydrogen bonding by ammonia may explain the improved flexural strength of the treated flax fibre. Another important aspect of the ammonia treatment is the dissolution of lignin within flax cell wall. Lignin acts as rigid binder in close association with cellulose microfibrils. Aqueous ammonia solution has been extensively investigated for lignin removal in order to improve biomass digestibility [14]. It was not yet confirmed in the present study that the modification of lignin within flax fibre contributes to higher flexural strength. However the lignin was expected to be partially removed in Group D after soaking for 24 h [12]. The ESEM images (Fig. 9) suggest the treatment may cause fibre cell wall debonding and deteriorate fibre properties.

4. Conclusion

This study demonstrates a novel and effective approach to characterise tensile and flexural strength of bast fibre from flax plants. By using X-ray μCT coupled with mechanical measurements of tensile strength, the findings provide quantitative evidence that the current practice and technology in the flax fibre industry may not be adequate to maximize fibre properties. To address the issue of fibre degradation in the extraction process, a unique ammonia pre-treatment was investigated for its effectiveness to improve fibre flexural strength. The results provide strong

evidence that flexural toughness increases after the ammonia treatment. This offers part of the solution for minimizing the degradation of natural fibre, not only during fibre extraction, but also during subsequent fibre conversion and composites preforming.

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