



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



Xuesen Zeng<sup>a,\*</sup>, Sacha J. Mooney<sup>b</sup>, Craig J. Sturrock<sup>b</sup>

<sup>a</sup> Faculty of Engineering – Polymer Composites Group, Division of Materials, Mechanics & Structures, University of Nottingham, University Park, Nottingham NG7 2RD, UK

<sup>b</sup> School of Biosciences, University of Nottingham, Sutton Bonington Campus, Sutton Bonington, Leicestershire LE12 5RD, UK

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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 ( $\mu$ 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 ( $\mu$ 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.

\* Corresponding author.

E-mail address: [Xuesen.zeng@nottingham.ac.uk](mailto:Xuesen.zeng@nottingham.ac.uk) (X. Zeng).

## 2. Materials and methods

### 2.1. Materials

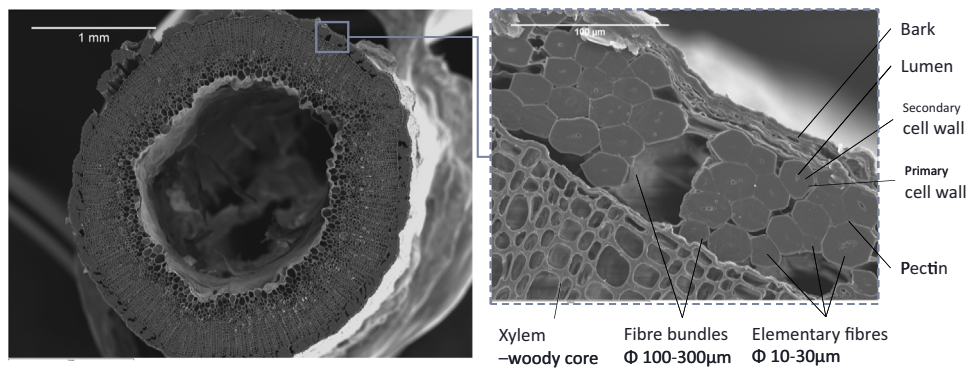
Flax plants were harvested and collected from the farm 'Flaxland' in Gloucestershire, U.K. For the current comparative study, three different Batches I, II and III from the same flax variety were used. Batch I was a yield from Year 2011, completed with the dew retting process. Batch II and III were from 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 $\mu$ CT and uni-axial tensile test

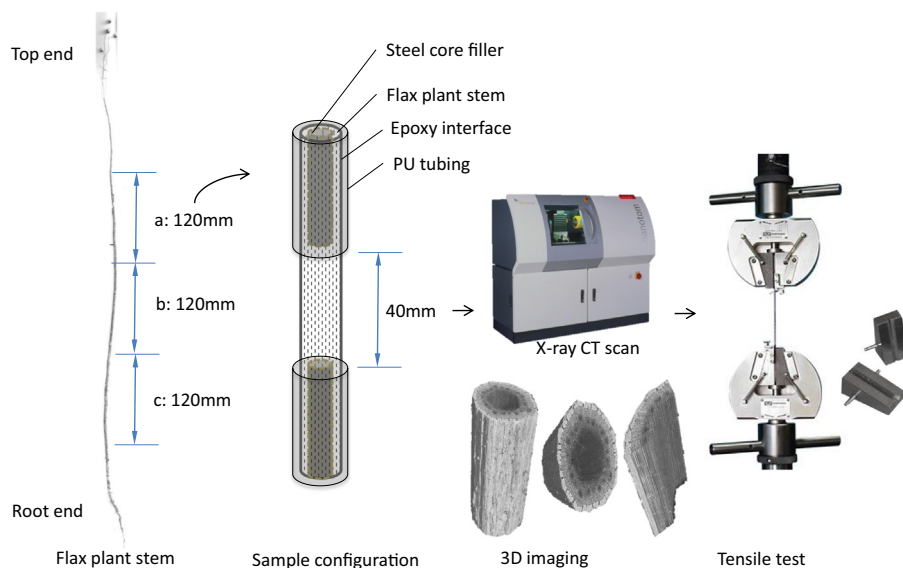
Flax plant stem cross-sections were imaged by SEM (Fig. 1). In order to achieve a flat smooth cross-section for SEM imaging, flax

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<sup>®</sup> 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.)

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