

Article

Real-Time Evolution of the Mechanical Properties of Flax Stems During Post-Harvest Dew Retting: Towards Objective Retting Assessment

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Abstract

Post-harvest field dew retting is a critical stage in flax fibre production because it facilitates the separation of fibre bundles from the surrounding stem tissues. In current agricultural practice, the optimum retting stop-point is still assessed largely using empirical criteria, motivating the development of quantitative indicators of retting progression. In this study, the mechanical properties of flax stems were investigated throughout a 91-day field dew retting campaign. Bending and lateral compression experiments were combined with a hollow-tube analytical model to determine equivalent flexural and compressive properties from force–deflection measurements. The equivalent flexural modulus and equivalent flexural yield strength showed an overall reduction during prolonged retting, whereas the equivalent compressive properties exhibited much less pronounced systematic variation. Additional tissue-removal experiments showed that the peripheral tissues make a substantial contribution to the whole-stem flexural response, while optical microscopy revealed increasingly evident separation and detachment of these tissues during the later stages of retting. Together, these results identify whole-stem flexural properties as promising quantitative indicators of retting progression and provide a basis for the future development of objective mechanical approaches for field retting assessment.

Keywords: *Linum usitatissimum*; dew retting; retting assessment; flexural properties; mechanical properties; natural fibres



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1. Introduction

Flax fibres are an important natural resource for textile and composite applications [1–3]. An important stage in their production is field dew retting, during which biological processes progressively facilitate the separation of the fibre bundles from the surrounding stem tissues [4–7]. Following retting, the stems undergo mechanical breaking, scutching and hackling to separate the fibres from the woody material [8,9]. The effectiveness of these operations depends strongly on the degree of retting: under-retting can hinder fibre separation, whereas over-retting can adversely affect fibre quality. However, field dew retting is strongly dependent on environmental conditions [10], and the practical decision of when to stop retting continues to rely largely on empirical assessment [9].

Considerable effort has therefore been devoted to identifying objective indicators of retting progression. Biological, chemical, optical and spectroscopic approaches have been investigated, including the assessment of retting microorganisms [11,12], changes

in pectin content [13], and optical or spectroscopic measurements [14–16]. Mechanical approaches have also been explored [17–22], including measurements of the force required to collapse bast-containing stem regions or detach fibres, and the work or energy required to peel fibre bundles from the stem. Such approaches are particularly interesting because the purpose of retting is ultimately to facilitate the mechanical separation of the fibres from the remainder of the stem. Nevertheless, despite these different approaches, no generally adopted quantitative method for determining the optimum retting stop-point is currently available.

Previous studies have demonstrated that mechanical measurements can provide indicators of retting progression [17–22]. Mechanical characterisation has also been widely used to investigate plant stems [23,24], including bending measurements to determine flexural properties [25,26] and compression measurements to determine transverse mechanical properties [27,28]. However, despite extensive studies of flax retting [29,30] and previous mechanically based retting indicators [17–22], the evolution of the whole-stem mechanical properties themselves throughout field dew retting has not, to our knowledge, been systematically established. This distinction is important because the flax stem is a heterogeneous, multilayer biological structure comprising peripheral epidermal/cortical and fibre-containing phloem tissues, cambial tissues and an inner lignified xylem surrounding a central lumen [31]. Consequently, mechanical measurements made on the complete stem represent the response of the overall structure rather than the intrinsic properties of any individual tissue.

In this work, we investigate the evolution of the mechanical properties of flax stems throughout a 91-day field dew retting period. Bending and lateral compression measurements were performed on flax stem samples, and a hollow-tube analytical model was used to determine their equivalent flexural and compressive properties. The objective was to establish whether these whole-stem mechanical properties exhibit systematic changes during retting and, consequently, whether they could provide quantitative indicators of retting progression. Additional tissue-removal experiments and optical microscopy were used to help interpret the observed mechanical behaviour. Figure 1 provides an overview of the experimental methodology.

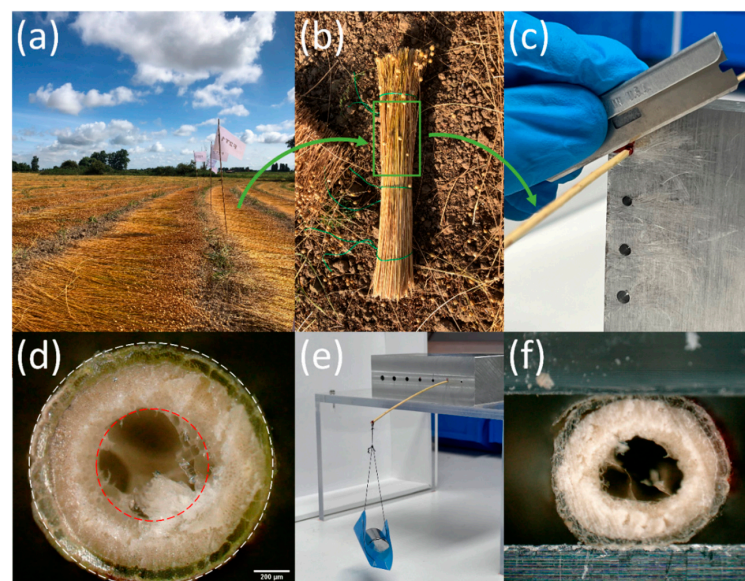


Figure 1. Overview of the study. (a) Flax stem samples undergoing field dew retting during summer 2022. (b) Collection of stem bundles for laboratory analysis. (c) Preparation of flax stem specimens. (d) Measurement of stem dimensions by optical microscopy. (e) Mechanical bending experiment. (f) Lateral compression experiment.

2. Materials and Methods

2.1. Field Retting and Sampling

Flax stem samples (Family: *Linaceae*; Genus: *Linum*; Species: *Linum usitatissimum* L.; cultivar Felice) were collected from a commercial flax production field operated by Van Robaeys Frères (VRF) near Killem, northern France. The flax straw was retted under normal commercial field conditions and was not arranged specifically for the present study. The spatial distribution of the straw therefore corresponded to the field-retting practice used by VRF during the 2022 campaign. Swath density (mass of straw per unit field area) was not independently measured.

Sampling was carried out throughout the 2022 dew-retting season over a period of 91 days, from 6 July to 4 October. Stem bundles were collected approximately twice per week in order to follow the progression of retting under natural field conditions. The campaign extended from the initial post-harvest stage to prolonged retting beyond the practical stop-point identified by VRF.

The retting stop-point was identified by experienced personnel at VRF according to their routine empirical assessment of the flax, approximately 56 days after harvest. The specific criteria used to make this decision during the present campaign were not formally recorded. The VRF-defined stop-point is therefore used throughout this study as an independently determined practical reference rather than as a precisely defined physical or mechanical threshold.

Meteorological data for the local area during the retting campaign, including minimum and maximum air temperature, rainfall, wind speed and duration of direct sunlight, are presented in Supplementary Figure S10. Relative humidity was not available in the meteorological dataset used in this study.

2.2. Sample Preparation and Conditioning

For mechanical characterisation, undamaged sections were selected from the central region of each flax stem to minimise geometric variability associated with the base and apex of the plant. Separate specimens were prepared for the bending and compression experiments. For bending measurements, 12 cm long stem samples were cut using a sharp razor blade and a purpose-built holding fixture to ensure reproducible cutting while minimising local damage. A total of 126 specimens were tested in bending. For compression measurements, 10 mm long stem sections were prepared using the same cutting procedure, and a further 126 specimens were characterised in lateral compression. Following preparation, all samples were examined using digital optical microscopy (VHX-6000, Keyence, Paris, France), and specimens exhibiting visible defects or damage resulting from harvesting or sample preparation were excluded.

Previous work has shown that freshly collected wet flax stems require approximately four hours of drying to approach a stable moisture condition under laboratory conditions [32]. Consequently, all samples were equilibrated under ambient laboratory conditions for at least four hours before mechanical testing. This procedure was used to reduce differences in moisture condition associated with the field conditions at the time of collection and to provide comparable conditions for mechanical characterisation. The absolute moisture content of individual stems was not measured before or after equilibration; residual differences in moisture content between samples therefore cannot be excluded.

Additional specimens were prepared to investigate the contribution of the peripheral fibre-containing tissues to the flexural response of the stems. For these experiments, the peripheral tissues were removed manually by gently rubbing the stem surface along the region subsequently subjected to bending, taking care to avoid mechanically damaging the underlying xylem. Removal was continued until the xylem was visibly exposed over the

test region. The peripheral tissues were more strongly attached to samples collected during the earlier stages of retting and became progressively easier to remove as retting advanced. The tissue-removed and intact measurements were performed on different stem specimens because the bending experiment was destructive and a before-and-after measurement on the same specimen was therefore not possible. The tissue-removed specimens were subsequently characterised using the same bending procedure as the intact stems.

2.3. Stem Dimensions

Accurate knowledge of the external and internal diameters of the stem samples is required for the mechanical modelling and subsequent extraction of their equivalent mechanical properties. Before mechanical testing, the external diameter was measured along the selected stem region to verify that the sample had a reasonably uniform cross-section. Following the bending experiment, the 9 cm tested region of each stem was divided into approximately 1 cm segments to expose a series of cross-sections along its length. The internal diameter could not be determined before testing without destructively sectioning the sample. At each end of every segment, six measurements of both the external and internal diameters were made using a VHX-6000 digital optical microscope (Keyence, France). Representative average external and internal diameters were subsequently calculated for each stem sample and used in the hollow-tube mechanical model.

2.4. Mechanical Testing

Bending and lateral compression experiments were performed using custom-designed experimental setups, illustrated schematically in Figure 2. In both cases, loading was applied quasi-statically using gravity-based methods, and the resulting deformation of the stem samples was recorded optically. Force–deflection relationships were subsequently obtained from the recorded images and used to determine the mechanical response of the stems. Details of the two experimental configurations are given below.

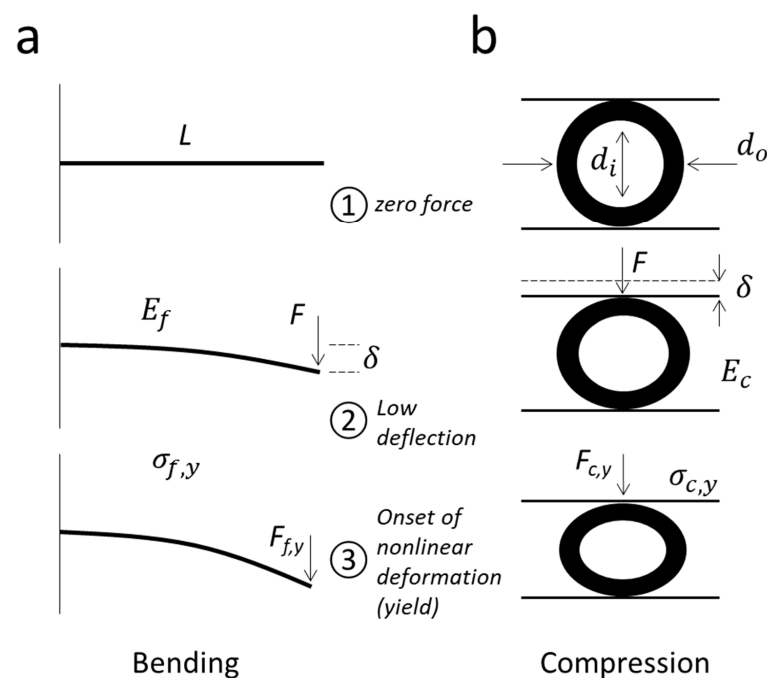


Figure 2. Schematic diagrams illustrating the principles of the mechanical experiments performed on flax stem specimens. (a) Bending of a flax stem configured as a fixed–free cantilever and subjected to a point load F at its free end. (b) Lateral compression of a flax stem specimen subjected to an applied load F .

2.4.1. Bending Measurements

For bending measurements, the 12 cm long stem samples were configured as fixed–free cantilevers, with 3 cm of the stem held in a purpose-built clamp to provide a free length of 9 cm (Figure 2a). The cantilever dimensions were selected specifically for the present experiments rather than from a standard testing protocol. Preliminary estimates based on the expected dimensions and mechanical properties of the flax stems were used to select a free length that would produce readily measurable deflections with the available calibrated loads. The same geometry was used for all samples throughout the retting campaign.

Point loads were applied at the free end of the cantilever using calibrated precision weights ($1\text{--}200\text{ g} \pm 0.1\text{ g}$) suspended from a purpose-built weight carrier, giving applied forces from 9.8 mN upwards. The clamp was slightly chamfered to minimise local damage to the stem at the fixed end. The resulting free-end deflection was recorded photographically using a digital single-lens reflex camera (Canon, Tokyo, Japan) [33], and the images were analysed using ImageJ (Version 1.54d, National Institutes of Health, USA) [34] to obtain the corresponding force–deflection curves.

The initial approximately linear portion of each force–deflection curve was used to determine the bending stiffness. A straight line was fitted to this region, and the flexural yield force was identified as the force at which the experimental response showed a clear and sustained departure from the initial linear behaviour. This force, rather than the force associated with eventual physical fracture of the stem, was used subsequently to calculate the flexural yield strength.

2.4.2. Compression Measurements

For compression measurements, 10 mm long stem sections were subjected to lateral loading using a purpose-built mechanical press (Figure 2b). The applied force was generated gravitationally using a container to which water was added progressively, enabling the compressive load to be increased quasi-statically and avoiding the transient forces associated with adding or changing discrete weights. Applied forces ranged from 294 mN to 57.82 N.

One end of the stem section was observed during loading using a VHX-6000 digital optical microscope (Keyence, France), allowing the deformation of the stem cross-section to be measured as the applied force increased. The recorded images were analysed using ImageJ (National Institutes of Health, USA) to obtain the corresponding force–deflection relationship. The initial approximately linear portion of this relationship was used to determine the lateral compression stiffness. The compressive yield force was identified as the force at which the experimental response showed a clear and sustained departure from this initial linear behaviour.

2.4.3. Tissue-Removal Bending Measurements

Additional bending measurements were performed on the tissue-removed specimens described in Section 2.2 using the same experimental configuration and procedure as for the intact stems. The intact and tissue-removed measurements were performed on different stem specimens because the bending experiment was destructive; the results therefore represent a comparison between independent sample groups rather than paired before-and-after measurements on individual stems. These experiments were intended to investigate the contribution of the peripheral fibre-containing tissues to the whole-stem flexural response rather than to determine the intrinsic mechanical properties of the individual tissues.

2.5. Mechanical Modelling of Flax Stems

As a first-order approximation, the flax stems were modelled as homogeneous hollow tubes of uniform cross-section. In the low-deflection regime, analytical solutions can therefore be used to relate the measured force–deflection response to the mechanical properties of the stem. This representation necessarily idealises the heterogeneous, multilayer biological structure of a flax stem and does not explicitly account for material anisotropy, differences between individual tissues, or local variations in wall thickness. To minimise geometrical deviations from the model, samples were selected from the central region of the stems, where the cross-section was relatively uniform and approximately tubular. The mechanical properties obtained using this model are therefore interpreted as equivalent whole-stem properties rather than intrinsic material properties of the constituent tissues. Local geometrical and structural heterogeneity may contribute to the sample-to-sample variability observed experimentally. All quantities were converted to SI units for calculation.

2.5.1. Bending of a Hollow Tube

For a fixed-free hollow tube cantilever subjected to a point load at the free end, the relationship between the load force F and the deflection δ in the low-deflection regime (Figure 2a) is as follows [35]:

$$F = \frac{3\pi\delta E_f}{64L^3} (d_o^4 - d_i^4) \quad (1)$$

where E_f is the equivalent flexural modulus, L is the length of the cantilever, d_o is the outer diameter of the tube, and d_i is the inner diameter of the tube.

The low-deflection approximation is applicable when the bending angle α is sufficiently small that $\sin \alpha \sim \alpha$. For the present analysis, the experimental data were restricted to $\delta/L \leq 0.2$.

The bending angle is as follows:

$$\alpha = \frac{3\delta}{2L} \quad (2)$$

Rearranging Equation (1) gives

$$E_f = \frac{64k_f L^3}{3\pi (d_o^4 - d_i^4)} \quad (3)$$

The equivalent flexural modulus E_f can therefore be determined from the slope $k_f = F/\delta$ of the approximately linear portion of the experimental force–deflection curve.

The equivalent flexural yield strength $\sigma_{f,y}$, corresponding to the maximum bending stress at the experimentally determined yield force $F_{f,y}$, is as follows [35]:

$$\sigma_{f,y} = \frac{32F_{f,y} L d_o}{\pi (d_o^4 - d_i^4)} \quad (4)$$

For a cantilever subjected to a point load at its free end, the maximum bending stress occurs at the fixed end. The flexural yield force $F_{f,y}$ was experimentally determined as defined in Section 2.4.1 and corresponds to the point at which the experimental force–deflection response shows a clear and sustained departure from the initial approximately linear regime, rather than to subsequent physical fracture of the stem.

2.5.2. Compression of a Hollow Tube

For a hollow tube of uniform cross-section subjected to lateral compression, the relationship between the applied force F and the resulting deflection δ (Figure 2b) is as follows [36,37]:

$$F = \frac{\delta L_c t^3 E_c}{24 (1 - \nu^2) r_o^3 \left(\frac{\pi}{8} - \frac{1}{\pi} \right)} \quad (5)$$

where L_c is the length of the compressed stem section, t is the tube-wall thickness, r_o is the external radius, and ν is Poisson's ratio [38]. The wall thickness is given by $t = (d_o - d_i)/2$. Rearranging Equation (5) gives

$$E_c = \frac{24 k_c (1 - \nu^2) r_o^3}{L_c t^3} \left(\frac{\pi}{8} - \frac{1}{\pi} \right) \quad (6)$$

The equivalent compressive modulus E_c can therefore be determined from the slope $k_c = F/\delta$ of the approximately linear portion of the experimental force–deflection curve.

The equivalent compressive yield strength $\sigma_{c,y}$ of the hollow tube is as follows:

$$\sigma_{c,y} = \frac{6 F_{c,y} r_o}{\pi L_c t^2} \quad (7)$$

where $F_{c,y}$ is the experimentally determined compressive yield force defined in Section 2.4.2. As for the bending measurements, this force is identified as the point at which the experimental force–deflection response shows a clear and sustained departure from the initial approximately linear regime, corresponding to the onset of nonlinear deformation rather than to subsequent physical collapse or fracture of the stem.

2.6. Data Analysis and Model Validation

Unless otherwise stated, mechanical-property data are presented as the arithmetic mean $\pm$ sample standard deviation (SD). For the principal bending and compression experiments, $n = 9$ independent specimens were measured at each sampling date; for the tissue-removal experiments, $n = 3$ independent specimens per group were measured at each sampling date. Sample SD was calculated using the $n - 1$ denominator. These statistics are used descriptively; no inferential comparisons between individual sampling dates were performed.

The analytical procedures used to extract the mechanical properties were validated experimentally using homogeneous hollow tubes of known material properties. For the bending model, measurements performed on 304 stainless-steel and polypropylene tubes yielded flexural moduli of 195.0 GPa and 1.336 GPa, respectively, in close agreement with the expected material values. The compression model was independently validated using the same polypropylene tube, yielding an equivalent compressive modulus of 1.310 GPa, compared with 1.336 GPa obtained from the bending experiment. The close agreement between the independently determined bending and compression moduli for the homogeneous polypropylene tube supports the validity of the analytical procedures used to extract equivalent mechanical properties from the flax stem measurements. Further details of the validation experiments, including tube dimensions and force–deflection data, are provided in the Supplementary Materials [34,39].

3. Results

3.1. Stem Dimensions and Geometrical Validation

Figure 3 shows representative optical microscope images of flax stem cross-sections taken at approximately 1 cm intervals along the tested region. The dashed white and

red circles indicate the external and internal diameters, respectively, used in the hollow-tube mechanical model. The cross-sections show that the selected central regions of the stems were approximately tubular and sufficiently close to circular to permit the first-order geometrical approximation adopted in the model.

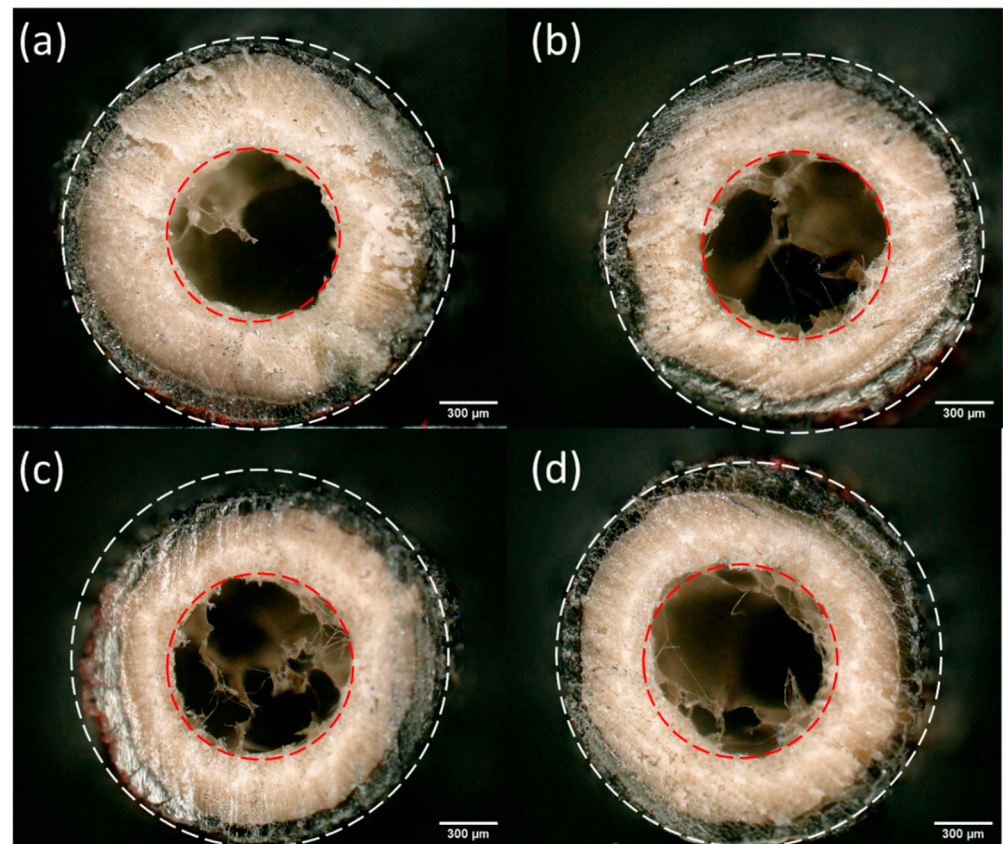


Figure 3. Typical optical microscope images of flax stem cross-sections obtained at positions of (a) 1 cm, (b) 3 cm, (c) 5 cm, and (d) 8 cm along a stem specimen. The white dashed and red circles indicate the outer and inner diameters, respectively.

Determination of the internal diameter was less straightforward than that of the external diameter because the boundary of the central lumen was not always sharply defined in the optical images. The internal diameter was therefore determined by fitting a circle to the visible, in-focus boundary of the xylem surrounding the lumen, as illustrated particularly clearly in Figure 3c.

Figure 4 shows a representative example of the external and internal diameter measurements obtained along the 9 cm tested region of a stem used in the bending experiments. As described in Section 2.3, measurements were made from cross-sections distributed along the tested region, with six measurements of both the external and internal diameters made at each cross-section. For the example shown, the average external and internal diameters were $1430.5 \pm 31.77 \mu\text{m}$ and $622.31 \pm 76.04 \mu\text{m}$, respectively. Across all samples, the mean relative SDs of the external and internal diameters were $2.56 \pm 1.05\%$ and $5.76 \pm 2.41\%$, respectively, while the mean ratio of internal to external diameter was $d_i/d_o = 0.47 \pm 0.07$ (Table 1). These measurements indicate that the selected stem regions exhibited sufficiently limited geometrical variation along their length for application of the hollow-tube approximation.

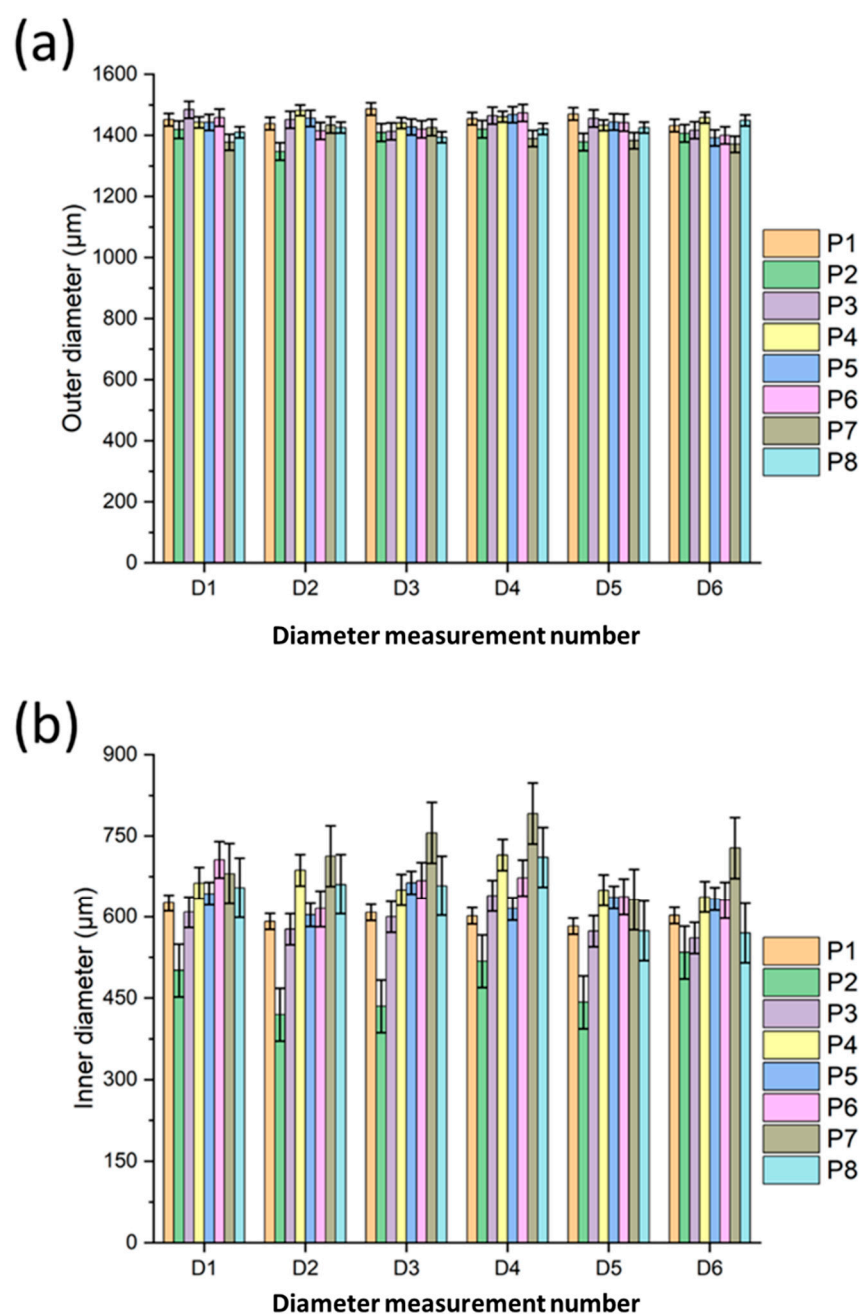


Figure 4. External and internal diameter measurements obtained along a representative flax stem specimen using the six-diameter measurement procedure. (a) External diameter and (b) internal diameter as a function of position along the specimen. Data are presented as mean $\pm$ SD from six diameter measurements at each position.

Table 1. Mean inner-to-outer diameter ratio and relative standard deviations of the external and internal diameters for all stem samples tested.

d_i/d_o	Relative SD of d_o	Relative SD of d_i
0.47 ± 0.07	$2.56 \pm 1.05\%$	$5.76 \pm 2.41\%$

The limited geometrical variation observed along the selected stem regions therefore supports the use of the hollow-tube approximation for the subsequent mechanical analysis.

3.2. Equivalent Flexural Properties of Retted Flax Stems

Figure 5 shows representative force–deflection curves obtained from nine stem samples collected after 35 days of retting. The complete curves (Figure 5a) exhibit an initial approximately linear elastic regime followed by nonlinear deformation and, ultimately, mechanical failure. Figure 5b shows the initial linear region used to determine the bending stiffness of each stem. Only data satisfying both the low-deflection criterion defined in Section 2.5.1 and the approximately linear force–deflection response were used for this analysis. The measured stiffness and individual stem dimensions were subsequently used with Equation (3) to determine the equivalent flexural modulus. The departure from the initial linear response was used to identify the flexural yield force and, using Equation (4), the corresponding equivalent flexural yield strength.

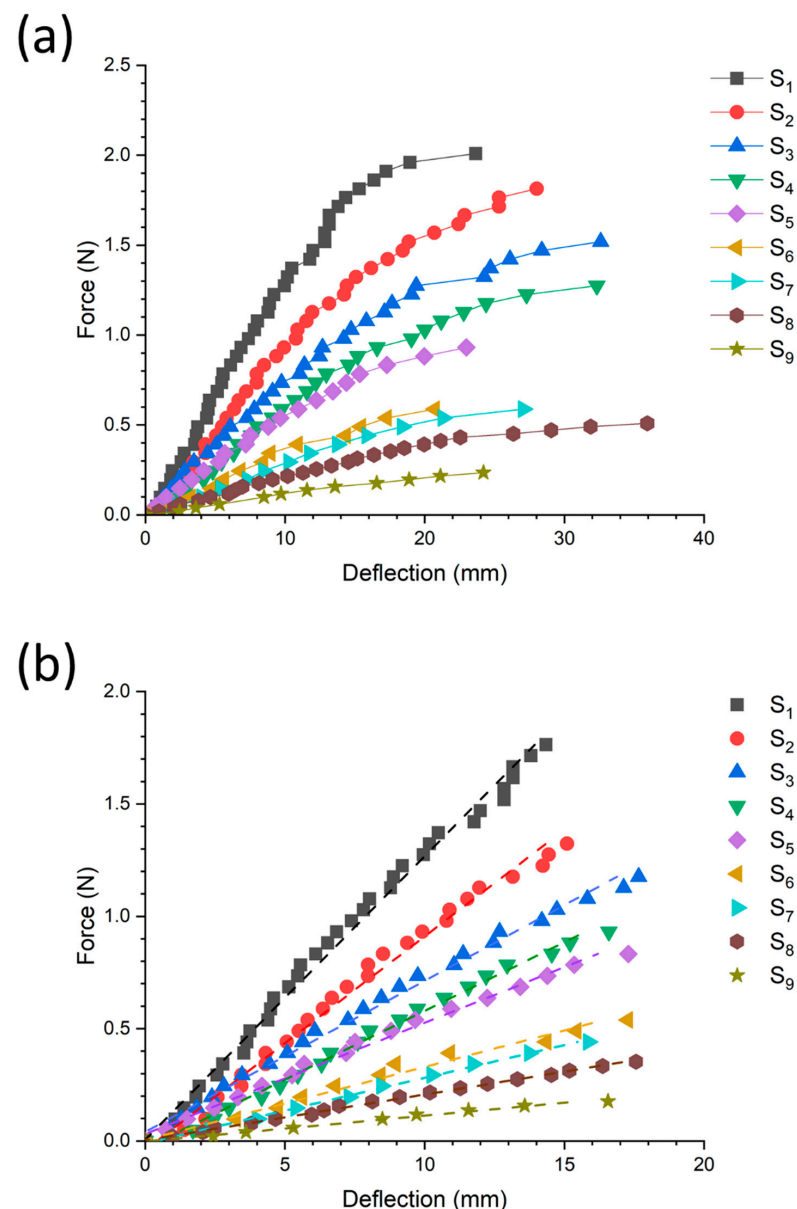


Figure 5. Representative force–deflection curves obtained from bending experiments on flax stem specimens collected after 35 days of retting. (a) Complete force–deflection curves showing the initial approximately linear regime, subsequent nonlinear deformation, and eventual mechanical failure. (b) Initial approximately linear region used to determine the flexural stiffness of the stem specimens. Dashed lines represent linear fits; the average coefficient of determination, R^2 is 0.991.

Figure 6 shows the evolution of the equivalent flexural modulus throughout the 91-day retting period. The data were obtained from 126 independent stem samples collected at 14 sampling dates, with each point representing the mean $\pm$ SD of nine samples ($n = 9$). Considerable variability is observed between specimens throughout the campaign. Nevertheless, the data show an overall reduction in flexural modulus during prolonged retting, with the lowest mean values occurring during the latter part of the campaign. The independently determined VRF stop-point at approximately day 56 is shown as a practical reference; the mechanical data do not indicate an abrupt transition at this point.

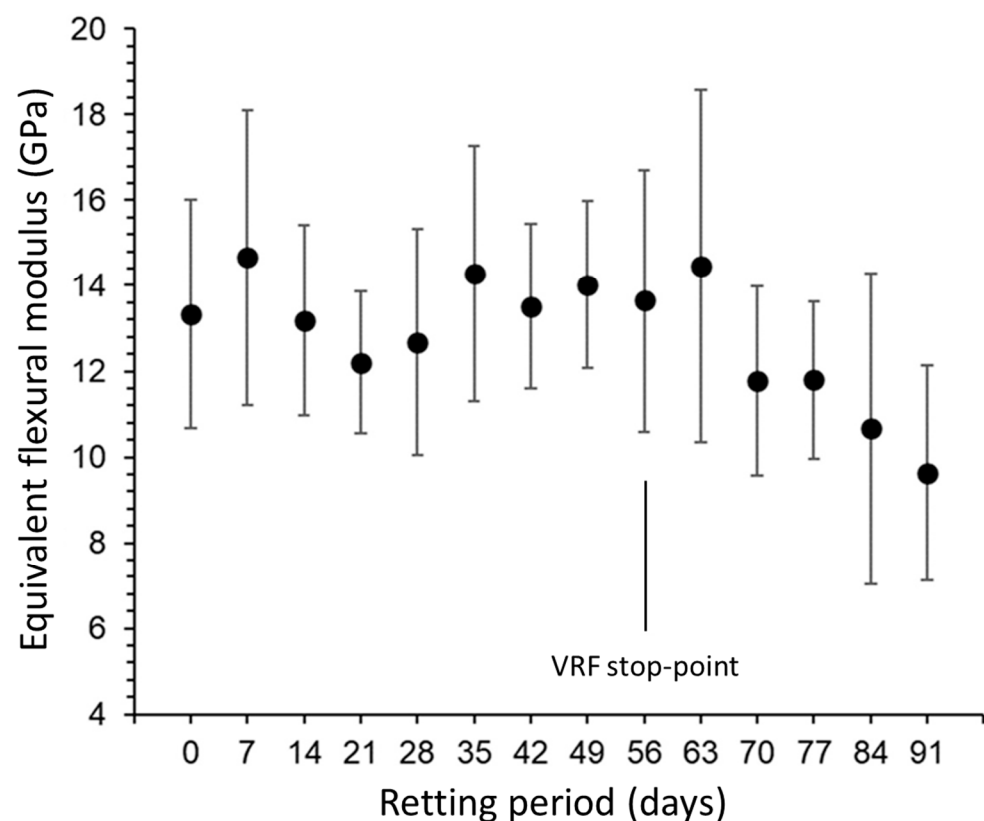


Figure 6. Equivalent flexural modulus of flax stems as a function of field dew retting time. Each data point represents the mean $\pm$ SD of nine independent stem specimens ($n = 9$). A total of 126 specimens were tested at 14 sampling dates throughout the 91-day retting period. The VRF-defined retting stop-point at day 56 was determined independently by an experienced retting specialist and is shown as a practical reference.

Figure 7 shows the corresponding evolution of the equivalent flexural yield strength. As for the flexural modulus, appreciable variability is observed between specimens, but lower mean values occur during the later stages of the retting campaign. The evolution is again gradual, with no evidence from these measurements of a sharply defined mechanical transition at the VRF stop-point. Taken together, Figures 6 and 7 indicate that prolonged field retting is associated with a reduction in both the equivalent flexural modulus and flexural yield strength of the flax stems.

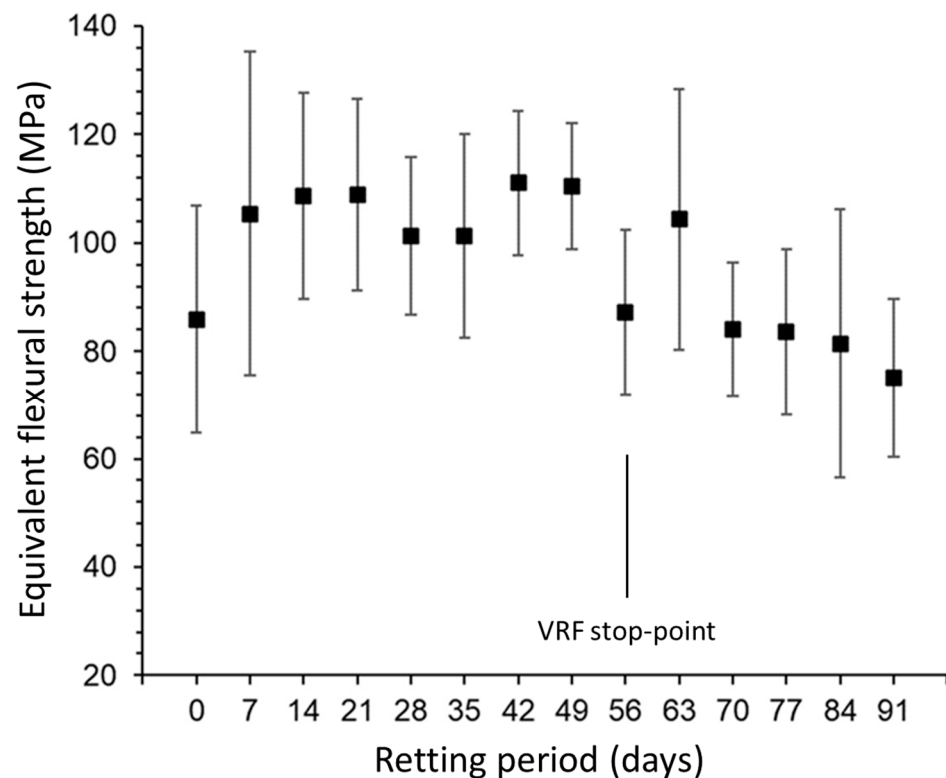


Figure 7. Equivalent flexural yield strength of flax stems as a function of field dew retting time. Each data point represents the mean $\pm$ SD of nine independent stem specimens ($n = 9$). A total of 126 specimens were tested at 14 sampling dates throughout the 91-day retting period. The VRF-defined retting stop-point at day 56 was determined independently by an experienced retting specialist and is shown as a practical reference.

3.3. Equivalent Compressive Properties of Retted Flax Stems

Figure 8 shows representative force–deflection curves obtained from nine stem samples collected after 35 days of retting. The complete curves (Figure 8a) exhibit an initial approximately linear regime followed by nonlinear deformation and, ultimately, collapse of the stem cross-section. Figure 8b shows the initial linear region used to determine the lateral compression stiffness of each stem. The measured stiffness and individual stem dimensions were used with Equation (6) to determine the equivalent compressive modulus. The departure from the initial linear response was used to identify the compressive yield force and, using Equation (7), the corresponding equivalent compressive yield strength.

Figure 9 shows the evolution of the equivalent compressive modulus throughout the 91-day retting period. In contrast to the flexural modulus, no pronounced overall reduction is apparent with advancing retting. Although the mean values vary throughout the campaign, the equivalent compressive modulus remains within a relatively limited range, with an overall mean value of 125.64 ± 25.13 MPa.

Figure 10 shows the corresponding evolution of the equivalent compressive yield strength. Again, variation is observed throughout the retting campaign, but no pronounced systematic reduction comparable to that observed for the flexural properties is apparent. The overall mean equivalent compressive yield strength was 42.33 ± 5.91 MPa.

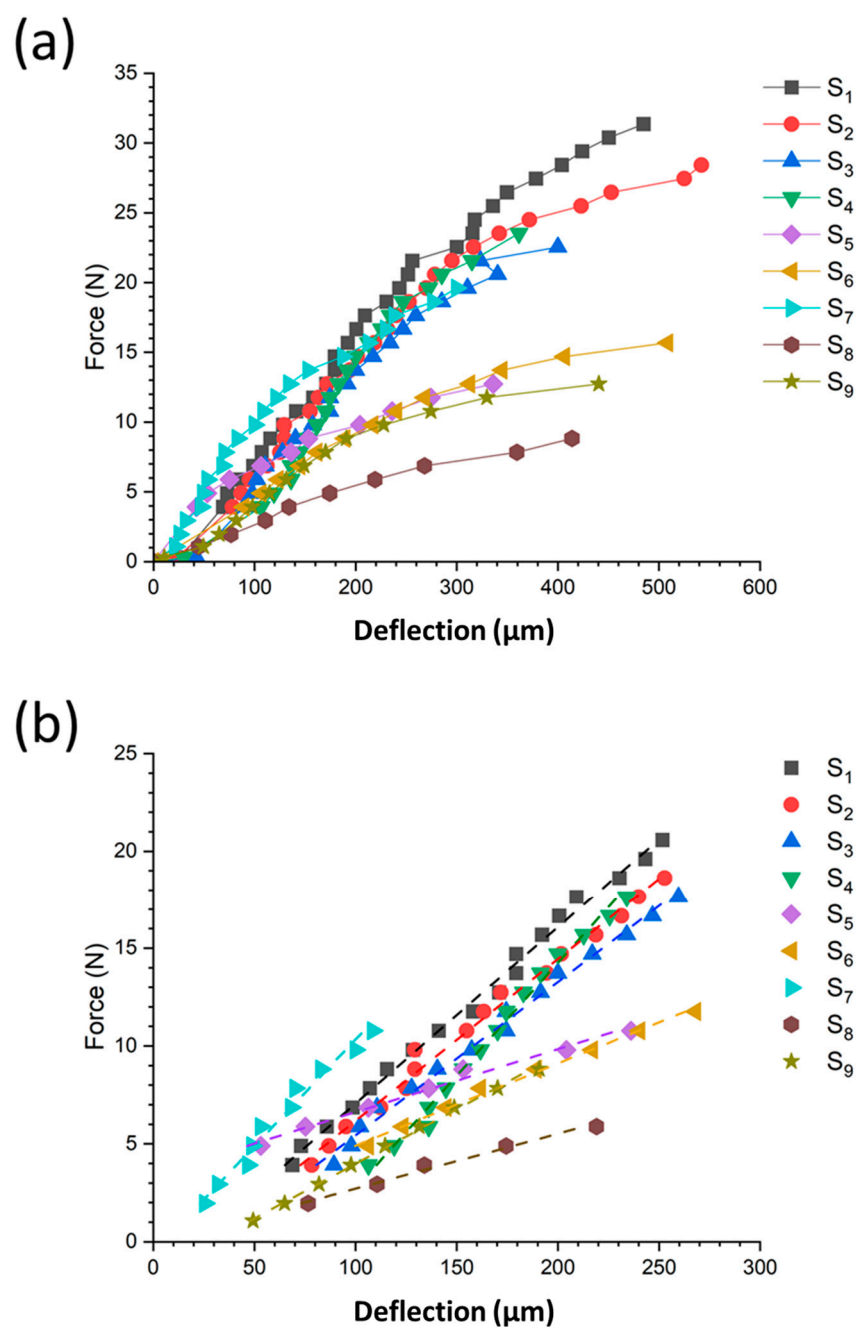


Figure 8. Representative force–deflection curves obtained from lateral compression experiments on flax stem specimens collected after 35 days of retting. (a) Complete force–deflection curves showing the initial approximately linear regime, subsequent nonlinear deformation, and eventual mechanical collapse. (b) Initial approximately linear region used to determine the lateral compression stiffness of the stem specimens. Dashed lines represent linear fits; the average coefficient of determination, R^2 is 0.99.

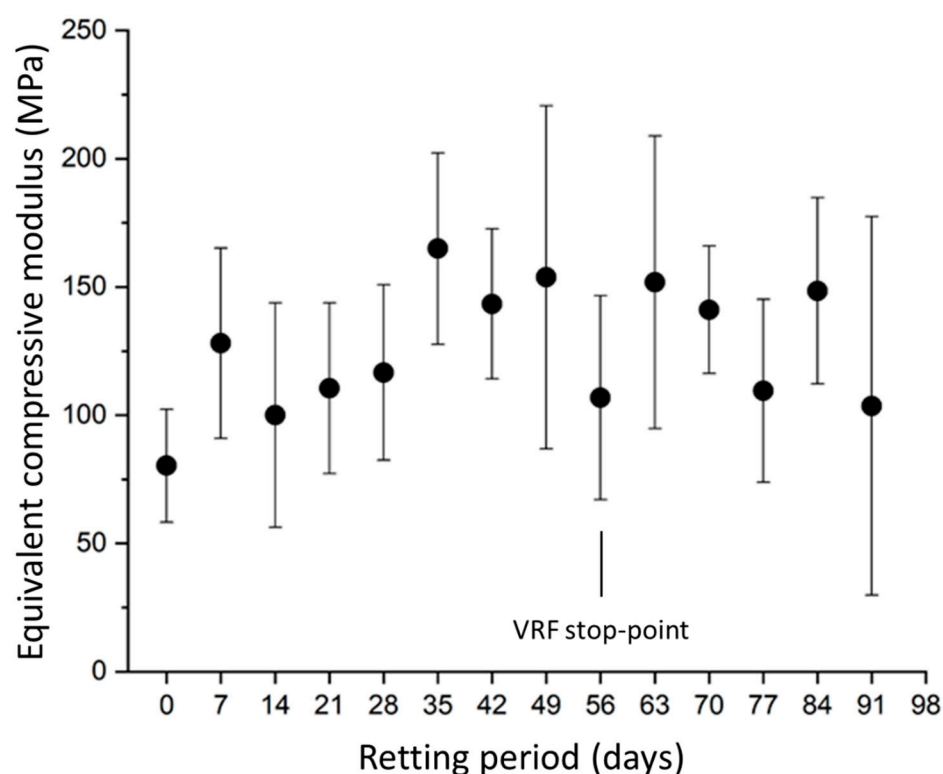


Figure 9. Equivalent compressive modulus of flax stems as a function of field dew retting time. Each data point represents the mean $\pm$ SD of nine independent stem specimens ($n = 9$). A total of 126 specimens were tested at 14 sampling dates throughout the 91-day retting period.

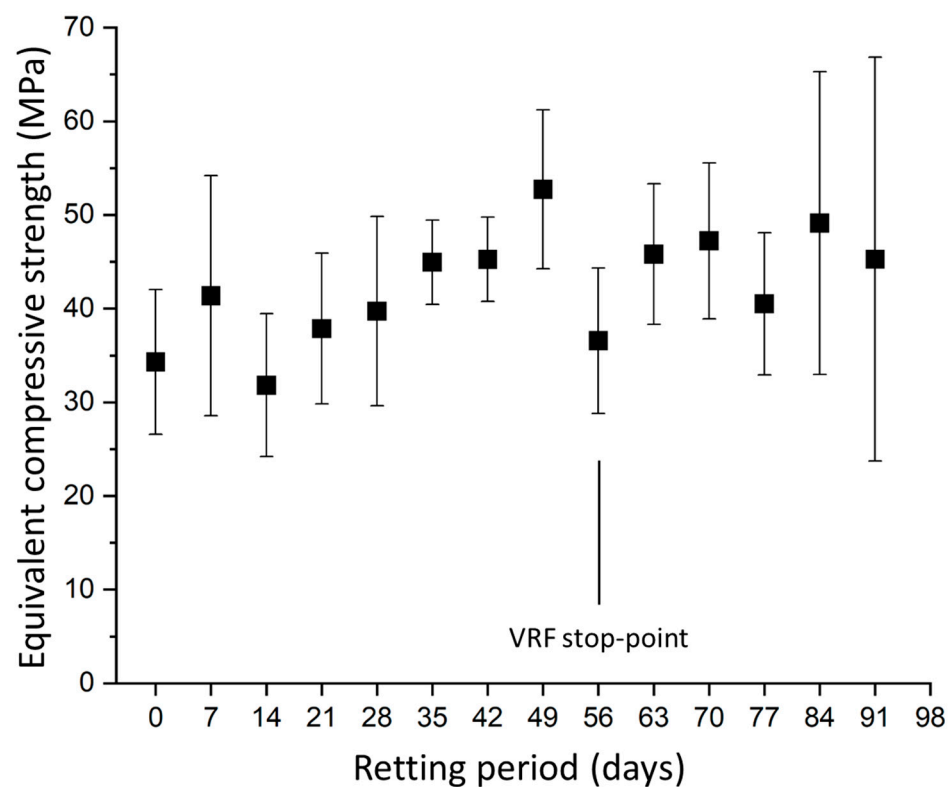


Figure 10. Equivalent compressive yield strength of flax stems as a function of field dew retting time. Each data point represents the mean $\pm$ SD of nine independent stem specimens ($n = 9$). A total of 126 specimens were tested at 14 sampling dates throughout the 91-day retting period.

3.4. Effect of Peripheral-Tissue Removal on the Flexural Response

Additional bending measurements were performed on intact and tissue-removed flax stem specimens throughout the retting campaign. Figure 11 shows the equivalent flexural modulus obtained for the two independent sample groups as a function of retting time. Each data point represents the mean $\pm$ SD of three independent specimens ($n = 3$), giving a total of 42 intact and 42 tissue-removed specimens.

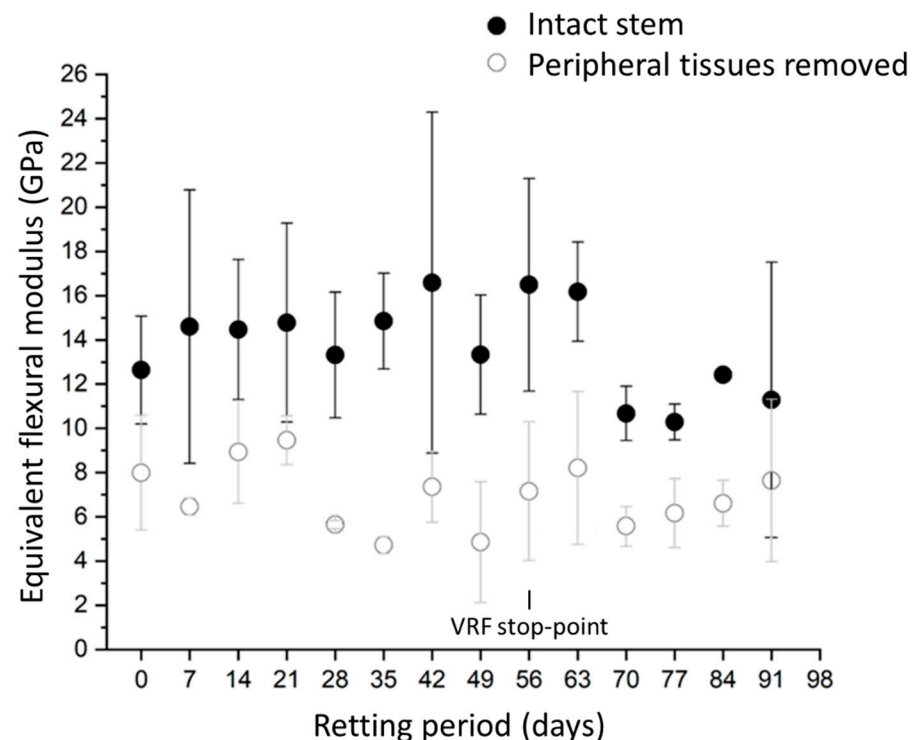


Figure 11. Equivalent flexural modulus of intact flax stem specimens (filled circles) and specimens with the peripheral tissues removed (open circles), as a function of field dew retting time. Each data point represents the mean $\pm$ SD of three independent stem specimens ($n = 3$). A total of 42 independent specimens were characterised for each experimental group.

The equivalent flexural modulus of the tissue-removed specimens was consistently lower than that of the intact specimens throughout the retting campaign. Across the 42 intact specimens, the mean equivalent flexural modulus was 13.70 ± 2.07 GPa, comparable to the value of 12.84 ± 1.48 GPa obtained from the larger dataset of 126 intact specimens shown in Figure 6. For the 42 tissue-removed specimens, the corresponding mean equivalent flexural modulus was approximately 6.95 ± 2.26 GPa. Thus, removal of the peripheral tissues was associated with a substantial reduction in the equivalent flexural modulus of the stem specimens.

Since the intact and tissue-removed measurements were performed on different specimens, these data represent a comparison between independent sample groups rather than paired before-and-after measurements on individual stems.

3.5. Optical Microscopy of Flax Stem Cross-Sections During Retting

Figure 12 shows representative optical microscope images of flax stem cross-sections obtained at four stages of the 91-day retting campaign. At day 1 (Figure 12a), the peripheral stem tissues appear predominantly green and remain closely associated with the underlying stem structure. At the VRF-defined retting stop-point at approximately day 56 (Figure 12b), the peripheral tissues appear brown and some local separation from the underlying tissues is visible. At day 70 (Figure 12c), the peripheral tissues appear darker and more extensive

separation and detachment are visible. These features are particularly evident at day 91 (Figure 12d), where substantial detachment of the peripheral tissues can be observed.

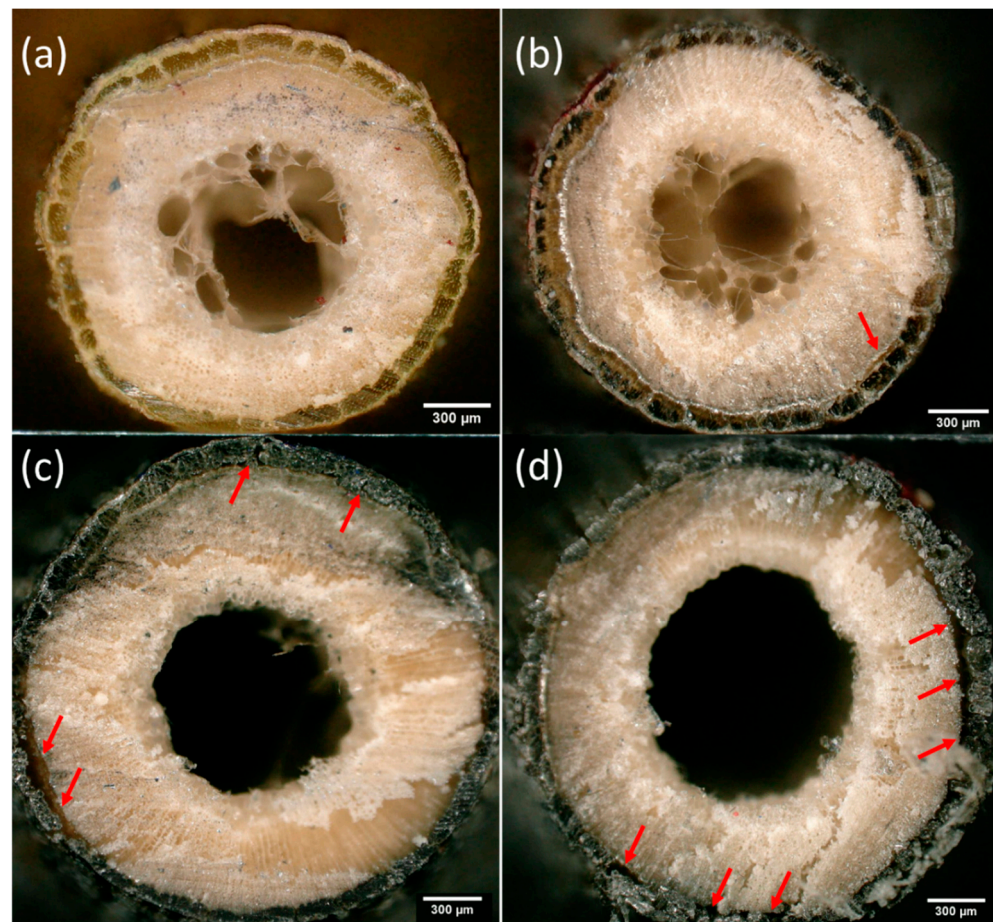


Figure 12. Representative optical microscope images of flax stem cross-sections obtained at different times during field dew retting: (a) day 1, (b) day 56, corresponding to the VRF-defined retting stop-point, (c) day 70, and (d) day 91. Red arrows indicate regions of visible separation and detachment of the peripheral stem tissues.

Overall, the representative cross-sections show increasingly evident separation and detachment of the peripheral stem tissues during the later stages of the retting campaign. Other morphological differences are also visible between the samples; however, the present optical images do not allow changes within individual tissue layers, or at the interfaces between them, to be identified or quantified unambiguously.

4. Discussion

The principal finding of this study is that the whole-stem mechanical response of flax evolves during field dew retting, but that this evolution depends strongly on the mode of mechanical loading. The equivalent flexural modulus and flexural yield strength show an overall reduction during prolonged retting, whereas the equivalent compressive modulus and compressive yield strength exhibit much less pronounced systematic variation. The VRF-defined retting stop-point at approximately day 56 provides a useful industrial reference for these observations, but the mechanical data do not indicate an abrupt transition at this point. Rather, the evolution of the flexural properties appears to occur progressively during the retting campaign.

The contrasting behaviour observed in bending and lateral compression is physically interesting because the two loading configurations interrogate the heterogeneous stem

structure differently. The equivalent compressive modulus is approximately one order of magnitude lower than the equivalent flexural modulus. This difference is unlikely to result simply from the use of different analytical models: control measurements on homogeneous polypropylene tubes gave closely comparable moduli in bending (1.336 GPa) and lateral compression (1.310 GPa). In flax stems, by contrast, the longitudinally oriented fibre-containing peripheral tissues and the cellular xylem contribute differently to deformation under the two loading configurations. Bending therefore probes a different effective combination of the constituent tissues and their geometrical arrangement from lateral compression [40]. This provides a plausible explanation for both the large difference between the equivalent flexural and compressive moduli and their different evolution during retting. The present measurements do not, however, resolve the individual contributions of the different stem tissues to either loading mode.

The tissue-removal experiments provide further insight into the flexural response. Removal of the peripheral tissues reduced the mean equivalent flexural modulus from 13.70 ± 2.07 GPa for intact specimens to approximately 6.95 ± 2.26 GPa for tissue-removed specimens. This substantial reduction demonstrates that the peripheral regions make an important contribution to the whole-stem bending response. Comparable observations have been reported by Réquillé et al. [41], who obtained an apparent modulus of 22.2 ± 2.5 GPa for intact flax stems and 9.2 ± 1.7 GPa following removal of the outer fibre-containing tissues. Nuez et al. [42] reported values of approximately 6.2–7.1 GPa for flax stem xylem. Although the absolute values differ between studies, which may reflect differences in cultivar, stem geometry, tissue structure and experimental methodology, the substantial reduction in effective flexural modulus following removal of the peripheral tissues is consistent between the different measurements. It should nevertheless be emphasised that the intact and tissue-removed specimens in the present study were independent samples and that the tissue-removal experiment does not provide intrinsic mechanical properties for individual anatomical tissues.

The optical microscopy observations provide complementary morphological context for these mechanical measurements. Increasing separation and detachment of the peripheral tissues became apparent during the later stages of the retting campaign. Considered together with the tissue-removal experiments, these observations are consistent with structural changes involving the peripheral tissues and their interfaces contributing to the evolution of the whole-stem flexural response. Dew retting is known to modify the pectin-rich components involved in cohesion and separation of the fibre-containing tissues [13]. However, the flax stem comprises epidermal, cortical, phloem, cambial and xylem tissues and the interfaces between them, and these cannot be treated as two homogeneous mechanical layers. The present experiments therefore do not establish whether the observed mechanical evolution arises predominantly from changes within particular peripheral tissues, at tissue interfaces, or from a combination of these processes. Tissue-resolved mechanical and structural measurements would be required to distinguish these mechanisms.

Although the reduction in whole-stem flexural properties during the later stages of the campaign is consistent with retting-induced structural changes, other factors cannot be excluded. Field retting occurred under naturally varying environmental conditions, and differences in stem moisture content, temperature, solar exposure or other uncontrolled field variables may have contributed to the observed mechanical evolution. All specimens were equilibrated under laboratory conditions for at least four hours before testing, but their individual moisture contents were not measured. Nevertheless, complementary evidence was obtained in our previous study using flax stems collected during the same retting campaign [22]. In that study, mechanically induced damage of the peripheral stem tissues under torsional loading was limited in under-retted samples but became increasingly apparent

around the optimum retting stage and during extended retting. These complementary mechanical observations are consistent with increasing susceptibility of the peripheral stem structure to mechanically induced damage as retting advances. They therefore support, although do not by themselves establish, an association between the reduction in whole-stem flexural properties observed here and retting-related structural changes.

An interesting comparison can be made with our previous measurements on individual flax fibres obtained during the same field retting campaign [43]. The flexural modulus and strength of manually extracted, damage-free single fibres showed comparatively little change with retting, even at advanced retting times. This contrasts with the reduction in the whole-stem flexural properties observed here. Taken together, these observations suggest that the evolution of the stem response need not result from a corresponding reduction in the intrinsic mechanical properties of the individual fibres, but may instead reflect changes in the surrounding tissues, tissue cohesion and/or mechanical coupling within the stem. This distinction is relevant to industrial processing, where efficient fibre extraction depends not only on fibre properties but also on the mechanical separation of the fibre-containing tissues from the remainder of the stem.

These results also provide a basis for considering whole-stem flexural properties as potential quantitative indicators of retting progression. Previous mechanically based approaches have investigated quantities associated with fibre separation, collapse or peeling [17–22], whereas the present approach follows equivalent mechanical properties of the complete stem during field retting. A practical indicator does not necessarily require the intrinsic mechanical properties of every constituent tissue to be known; rather, it requires a measurable parameter that responds reproducibly to changes relevant to the retting process. In the present campaign, flexural properties were more sensitive to prolonged retting than lateral compressive properties and therefore appear to be the more promising candidates for such an approach. However, simultaneous measurements of established retting indicators, fibre-extraction efficiency and final fibre quality were not performed on the present samples. The results should therefore be regarded as demonstrating the potential of whole-stem flexural measurements as quantitative indicators rather than establishing a validated retting stop-point or complete assessment method.

Several limitations should be considered when interpreting these findings. The experiments correspond to a single cultivar and a single field-retting campaign conducted under naturally varying environmental conditions. The summer 2022 campaign included periods of high temperature and limited rainfall (Supplementary Figure S10), conditions that may have contributed to its relatively long duration. Individual stem moisture contents were not measured after laboratory equilibration, and residual moisture differences may therefore have contributed to specimen-to-specimen variability. In addition, the hollow-tube model deliberately represents the heterogeneous stem by equivalent whole-stem properties, the tissue-removal measurements involved independent rather than paired specimens, and the microscopy observations were qualitative rather than quantitative. Future studies across different cultivars, growing conditions and retting seasons, together with simultaneous measurements of established retting indicators and fibre-extraction performance, will be required to determine the robustness and practical sensitivity of the mechanical indicators identified here.

5. Conclusions

This study investigated the evolution of the whole-stem mechanical properties of flax during a 91-day field dew retting campaign. Bending and lateral compression experiments, combined with a hollow-tube analytical model, were used to determine equivalent flexural and compressive properties throughout the retting period. The equivalent flexural modu-

lus and equivalent flexural yield strength showed an overall reduction during prolonged retting, whereas the equivalent compressive properties exhibited much less pronounced systematic variation. The mechanical evolution was gradual, with no evidence of an abrupt transition at the VRF-defined retting stop-point. Removal of the peripheral tissues substantially reduced the equivalent flexural modulus of the stem specimens. Optical microscopy additionally revealed increasingly evident separation and detachment of the peripheral tissues during the later stages of retting. Together, these observations suggest that structural changes involving the peripheral tissues and/or their interfaces may contribute to the evolution of the whole-stem flexural response. However, the present experiments do not resolve the respective contributions of individual tissues or tissue interfaces. The sensitivity of the flexural properties to retting progression suggests that whole-stem bending measurements have potential as quantitative indicators for objective assessment of field dew retting. The present study was, however, conducted during a single field campaign using one flax cultivar, and the mechanical measurements were not correlated directly with fibre extraction efficiency or final fibre quality. Further studies across different cultivars, growing conditions and retting seasons, together with such correlations, will therefore be required to establish the robustness of the observed trends and determine whether flexural measurements can ultimately provide a practical criterion for identifying the optimum retting stage.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/agriculture16181940/s1>. Figure S1: Examples of photographs of a fixed-free flax stem cantilever subjected to increasing point loads, from the undeflected state to eventual mechanical failure; Figure S2: Digital optical microscope images of an example of a flax stem under compression using the stem compression tool developed for the study; Figure S3: Schematic representation of the dimensions of steel and polypropylene pipes used for the model validation experiment; Figure S4: Accurate determination of the inner and outer diameters of the tubes; Figure S5: Accurate determination of the inner and outer diameters of the tubes; Figure S6: Experimental setup for the fixed-free cantilever bending experiments on steel and polypropylene pipes; Figure S7: The experimentally-determined deflection of the end of the cantilever as a function of the applied force for a steel pipe and a polypropylene pipe; Figure S8: Plots of cantilever deflection versus applied load force in the low deflection portion of the experiments; Figure S9: Deflection of the polypropylene pipe as a function of load; Figure S10: shows the weather data for Killem, France during the period of 6th July to the 1st October; Table S1: Summary of diameter measurements for a steel pipe; Table S2: Summary of diameter measurements for a polypropylene pipe; Table S3: Results of the deflection of the steel pipe; Table S4: Results of the deflection of the polypropylene pipe; Table S5: The low deflection regime of the fixed-free cantilevers (steel and polypropylene); Table S6: Calculation of the elastic modulus of the pipes (stainless steel 304 and polypropylene) using the experimentally-determined parameters: pipe dimensions and stiffness; Table S7: Experimentally-determined values used for the determination of the polypropylene tube's elastic modulus.

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