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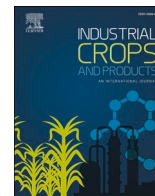
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Towards dynamic in situ observation of flax fibres under varying water contents through environmental SEM and X-ray tomography

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ABSTRACT

Flax fibre represents one of the most promising natural components for the development of new innovative high-performance biobased composite materials. Their properties are closely related to the lumen, the central cavity of the fibre, which was only studied, to date, under unrealistic environments as in dry conditions. The objective of the current study is to characterise, at a micrometre-scale, the surface and volume modifications induced by exposure of fibres to humid atmospheres. Different dynamic characterization strategies have shown a significant opening of the hydrated lumen "channel". Values of this opening rate are discussed in the light of potential biases linked to the investigation technique (SEM versus X-ray tomography), and to the adopted acquisition approach (*in situ* versus *ex situ*). By eSEM, after a 5 min. exposure to the water-saturated environment for instance, the lumen opening value was measured at $36 \pm 2\%$ for *ex-situ* hydration of the bundles with a subsequent delamination of the fibre, while it was only $15 \pm 2\%$ when hydration was progressively conducted into the SEM chamber. By microtomography, the lumen swelling for the single fibre was estimated to be within the same order of magnitude, about 7%, after hydration and measurements performed under realistic atmospheric pressure.

1. Introduction

Biosourced materials have historically been extensively used for centuries in various structural applications (wood, cement, clay...), and are currently widely studied to develop alternatives to petro-sourced products. The field of composite materials is no exception to this ecodesign-driven trend, which explains great efforts to progressively replace traditional composites reinforced with conventional fibres (glass, carbon) with new natural fibre-based composites (Bourmaud et al., 2018). The flax sector is particularly interesting for several reasons. The comparative life cycle assessment between flax and glass fibres shows that for most of the different environmental indicators (climate change, acidification, non-renewable energy consumption...), the production of flax fibres is an ecologically efficient alternative to glass fibres (Le Duigou et al., 2011; Durand et al., 2023). Flax fibres are also hygromorphic, *i.e.* they change their morphology according to the humidity rate (mainly atmospheric). For instance, hygromorph biobased composites (HBC) possess moisture-induced actuation, allowing controlled bending actuation properties of flax/polypropylene

composites over a large range of water contents (Le Duigou et al., 2017a). Those emerging HBC materials can out-of-plane displacement when a moisture gradient is present, and flax fibre elements are key-players (Le Duigou et al., 2019). HBC actuators could have a low environmental footprints but the design of the bilayer configuration (like pine cone) still requires guidelines and enriched database on fibre elements, specially the lumen.

Particular attention is paid to the lumen, which is the central cavity of fibres, corresponding to the remnant of the cell nucleus and the cytoplasm of the fibre (Bourmaud et al., 2018). The lumen plays a major role in the mechanical properties of the fibre and is a preferred path for crack propagation (Beaugrand et al., 2015). Its shape generates stress concentrations that can be the cause of cracks, as already demonstrated in the literature (Richely et al., 2021; Aslan et al., 2011). If the characterization of the flax fibre is quite documented, the lumen remains poorly studied, even though its implication in fracture mechanics of natural fibre-reinforced composites is now well-established (Richely et al., 2021). As a sign of the lumen contribution interest, recently the Influence of the plant fibre geometry, and especially the lumen, has been

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investigated and revealed major role in the apparent transverse elastic fibre property (Govilas et al., 2023). The influence of the water content on the lumen shape and dimension seems therefore a parameter to better decipher. For such very localized studies, different techniques were involved. First, efforts were required to properly prepare samples by microtomy (Richely, 2022; Arnould et al., 2017), or FIB (focused ion beam) milling (Charlet et al., 2008, 2010), before their microscopic (light or electronic) examination. For a global observation, the use of micro-tomography is often reported to visualize the internal structure (Richely et al., 2021; Eve et al., 2012; Abbey et al., 2010) of the fibre and thus to characterize the lumen in its length. Nevertheless, to the best of our knowledge, all papers refer to low water content (about 8% of water content at 50% relative humidity and often less), or 'dry' fibres because of experimental constraints. These ideal conditions are far from the real life *in planta* state, and more realistic conditions have to be tested. In particular, such severe conditions could simulate the behaviour of composites in service, when fibres can absorb water. In that context, an interesting original micrometer-scale *in situ* approach of hydration was proposed by Barbetta et al. using environmental SEM considering wood cell walls (Barbetta et al., 2018). Other more global studies on sorption of flax fibres can be found, based on mass gain measurements, but again at a broader scale for mm-long flax bundles (Lu et al., 2022).

The objective of this study is then to determine the dynamic behaviour of the fibre, and especially of the lumen, in terms of both dimension and shape, when the moisture rate is changing. The hypothesis investigated is that the hydration of flax fibres leads to an opening of the lumen, due to the hygroscopic mechanism. The lumen hygroscopic mechanism is a phenomenon observed in certain plant cells, particularly in the xylem vessels of woody plants. It involves the movement of water through the plant's vascular system due to the presence of hydrophilic (water-attracting) substances within the cell walls of cells (Tyree et al., 2002; Sperry et al., 2004). A two-step mechanism is described in the case of flax fibres, with a first absorption of moisture from air, causing then the cell walls to expand (Pickering et al., 2016; Müssig, 2010). In this mechanism, the role played by the lumen needs to be better understood, the objective of this paper. A specific non-contact preparation protocol is first developed allowing a submicrometric observation of the fibre networks section under contrasted Relative Humidity (RH) conditions, as well as in a fully saturated state. A semi-quantitative complementary three-dimensional characterization will also be carried out by X-ray microtomography to assess the behaviour of single fibres.

2. Materials and methods

2.1. Materials

Scutched flax fibres and bundles (*Linum usitatissimum* L.) from the Bolchoi variety were provided by Groupe Depeste/Teillage Vandecandelaère (Bourguebus, France). Plants were cultivated in 2017 in Normandy, France, and dew-retted in the field. Fibre extraction was achieved by industrial scutching. The fibre batches were then stored in a controlled environment and protected from light (Richely et al., 2021).

2.2. Electron microscopy in environmental mode

Before their observation, flax bundles were prepared according to a specific non-contact protocol, preserving their morphological characteristics. Flax bundles were thus first immersed for a couple of minutes in liquid nitrogen (-196°C), then cut manually under slight tension. Finally, the resulting sections were finished by ion polishing at low temperature (Ilion II+ Gatan (5 keV, 3 RPM, 90° wedge, -5°C, 2h00)) to reveal the internal microstructure.

An environmental scanning electron microscope (eSEM Quattro S Thermo Fisher) was used in environmental mode to observe microstructural changes of the sample with the RH conditions. In this mode,

both the pressure in the SEM chamber and the temperature of the sample (via a Peltier stage) are imposed. Therefore, the sample is exposed to a well-controlled humidity rate, from 0 (high vacuum) to 100% (condensed water) (Faustini et al., 2018; Chatre et al., 2023). Images were acquired using an acceleration voltage of 20 kV, for a beam current of about 0.05 nA, ensuring that no significant beam effect was induced by the electron irradiation. The control of the environment, in terms of both RH and treatment time, is described in Fig. 1. The hydration step is carried out either in the eSEM (Fig. 1a,b) or outside, using a chamber (crystallization dish) where the atmosphere is saturated in water vapour (Fig. 1c,d). To better determine the microstructural evolution of the sample, the same regions of interest were systematically followed throughout the test. Images corresponding to each experimental condition were interpreted with the ImageJ software. From the calculation viewpoint, the lumen opening rate, expressed in %, does correspond to the relative variation of the lumen area with the hydration time. For reproducibility reasons, the lumen area measurements were repeated independently 5 times per fibre, and for at least 4 different fibres.

2.3. Tomography

In addition to the micrometre-scale characterization of the fibres' surfaces, a laboratory tomograph (EasyTom tomograph - RX Solutions, France) was used to determine and quantify the volumic internal structure of single fibres at high resolution (0.3 μm^3 voxel size). The tomograph is equipped with a LaB6 emission tip for the X-ray source (Hamamatsu) operating at a voltage of 80 kV. Continuous rotation was used, and the number of projections was reduced according to the stability of the fibre: between 180 and 720 projections were collected over 360° of rotation with an exposure time of 3 seconds per radiograph. These parameters resulted in measurement periods of 10–35 minutes for a complete scan. The sample was first tomographed under ambient conditions (25°C, atmospheric pressure), and then in hydrated environments using the ex-situ hydration protocol involving the saturation chamber (Fig. 1d) for a total of 5, 15 and 35 minutes. All data were reconstructed by a Feldkamp algorithm in filtered back-projection, and resulting volumes were processed with ImageJ and ParaViews dedicated software. From a quantitative viewpoint, uncertainty associated to the reconstructed volumes were estimated considering two extreme threshold levels corresponding to deliberately under/over-estimated values. Adopting that conventional approach, precision of the reconstructed volume values are 0.5% and 0.7% for the lumen and the fibre, respectively.

2.4. Hydration procedure

Two different procedures were indeed used to highlight a potential artefact linked to the low pressure inherent to the SEM use. They differ from the fact that the hydration step was conducted either inside or outside the SEM chamber (*in situ* or *ex-situ* respectively). Fig. 1 illustrates these two ways of hydration, adopted for eSEM (a-d) and tomography (e, f) characterizations. First, bundles were subjected to a water-saturated environment (20°C, 20 torr, 100% Relative Humidity) for 5, 10, 20 and 90 minutes inside the microscope chamber, and then observed with optimal imaging conditions (1°C, 4.4 torr, 90% RH). For the second *ex-situ* protocol, the bundles were subjected to a water-saturated environment at the atmospheric pressure outside the microscope chamber in a vapour-saturated chamber for 1, 2, 3, 5 and 10 minutes, and then observed under the same identified imaging parameters.

3. Results and discussion

3.1. Qualitative proof of lumen opening

Fig. 2 compares the evolution of the lumen size of flax fibre bundles, observed by environmental electron microscopy, after the two hydration

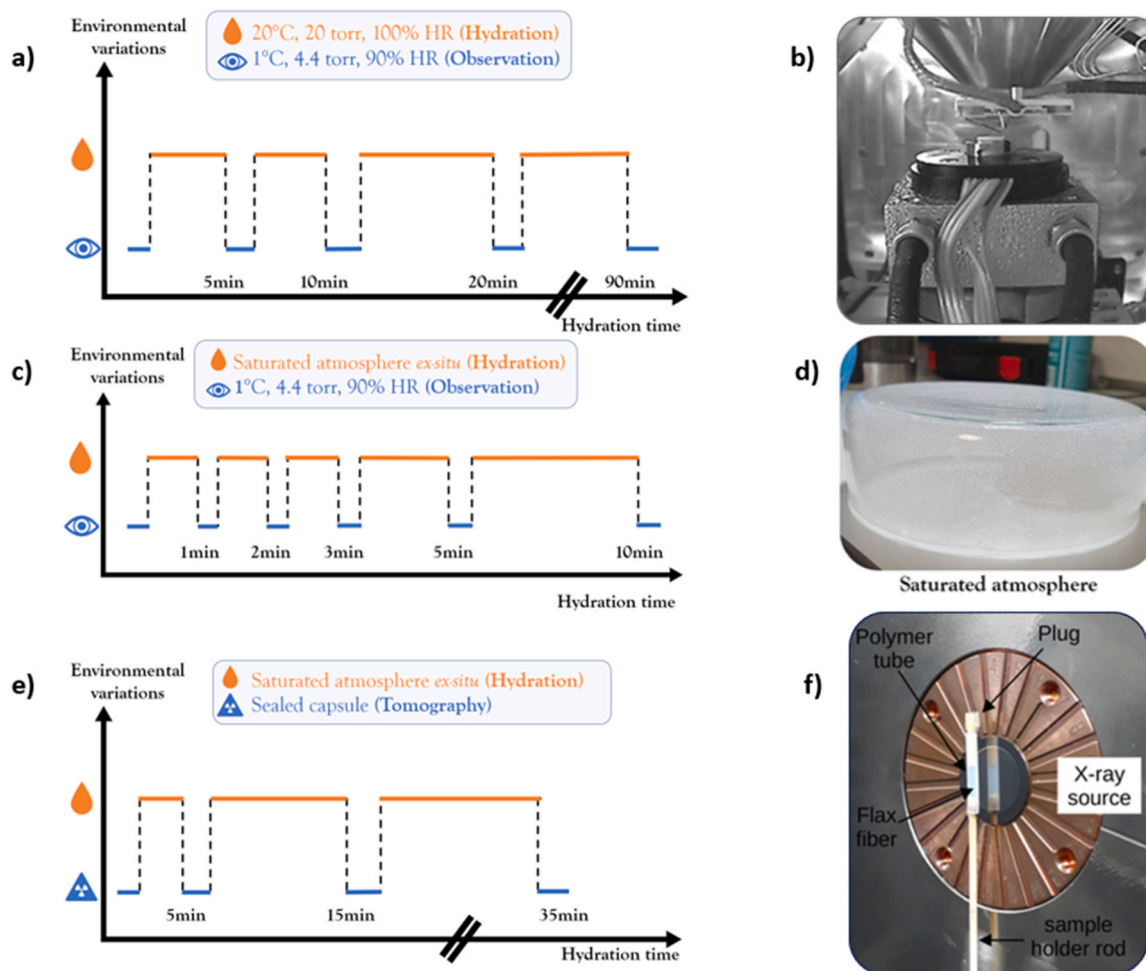


Fig. 1. In situ (a) and ex situ (c) hydration protocol for the eSEM characterization, Peltier stage into the eSEM chamber (b), crystallization dish used for the ex situ water saturated hydration (d), ex situ protocol (e) and experimental setup (f) involved in the tomography characterization (f).

protocols. As expected, the hydration of flax fibres reveals their hygromorphic behaviour, with a global volume increase which is undoubtedly confirmed. More interestingly, the hygromorphic behaviour manifests itself by a significant opening of the lumen, which has not been documented to date. Moreover, the kinetics of lumen opening vary according to the protocol adopted and are much faster for *ex-situ* hydration. Indeed, for a 5 min. exposure to the water-saturated environment for instance, the lumen opening rate is estimated to be $36 \pm 2\%$ for *ex-situ* hydration of the bundles, while it is only $5 \pm 2\%$ when hydration is conducted in the SEM chamber. The ability of the lumen to open also varies greatly according to the hydration procedure: while the opening parameter seems to be capped at $17 \pm 2\%$ for the *in situ* hydration protocol after 90 minutes of hydration, this opening rate is reached in only one minute for the *ex-situ* hydration. Furthermore, the lumen opening rate reaches $49 \pm 3\%$ at the end of the experiment, after only 10 minutes of hydration. Moreover, no plateau is observed, so the lumen might have opened more if the *ex-situ* hydration study had been pursued. Nevertheless, it is worth mentioning that the SEM observation of *ex-situ* hydrated fibres reveals a strong structural modification. The recorded damage, shown for several fibres in the insert of Fig. 2a and Fig. 2b, does correspond to a concentric delamination of the fibre, that is not observed if hydration is conducted into the SEM chamber. This irreversible degradation by sublayer decohesion may be explained via biopolymers induced modifications, by a potential dissolution of hydrophilic cell wall polymers like hemicellulose and pectins at those inter sublayer interfaces. The fibre damage may also result in a cellulose structural organization modification in the distance of their microfibrils in the

sublayer cores, as reported recently (Gautreau et al., 2022). One could also hypothesise that this irreversible delamination happens through physicochemical considerations, due to the degassing of waterlogged fibres during the under-vacuum setting of SEM. Interestingly, the observed delamination creates voids in the structure, which was recently proven to participate in the damage mechanism in flax fibre, especially in the kink-band region (Morgillo et al., 2023). In this sense, characterization of the bundle hydrated progressively into the SEM throughout the test would be then less aggressive for the sample, and this *in situ* protocol would be more representative of natural conditions.

3.2. Toward a realistic estimation of the lumen opening

The previous part dedicated to SEM analysis gave information on the cross-section microstructure of bundles. It clearly showed an opening of the lumen structure when hydrated, but its measurement could be affected by a potential effect of pressure. Moreover, observed fibres constitute free surfaces, opened to the atmosphere, which may affect their behaviour towards water. That's why another complementary characterization technique was used: the X-ray tomography. By this technique, we can access a more global 3-dimensional examination of samples on the one hand, and try to determine an assessment of the lumen volume variation in realistic conditions (atmospheric pressure) on the other hand.

A single fibre was first observed in its as-received state, *i.e.* without artificial hydration (Fig. 3). The tomogram carried out in these initial conditions shows a lumen present all along the quasi-continuity of the

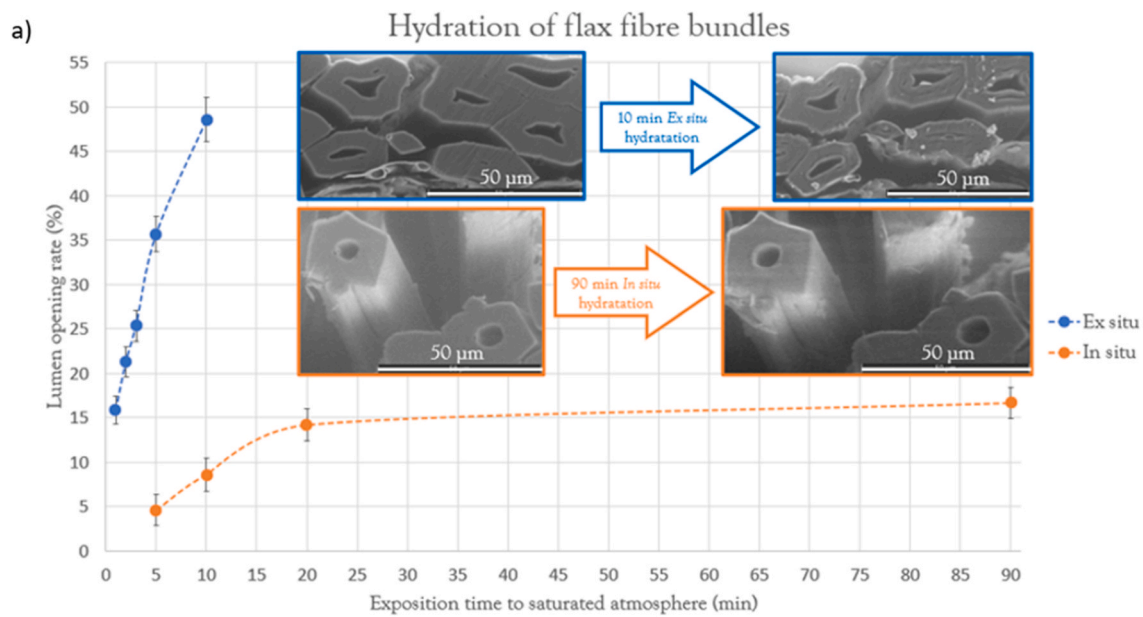


Fig. 2. eSEM observation of flax fibre bundles, and comparison of lumen opening depending on the hydration mode (a), and lumen evolution as a function of hydration time in *ex-situ* configuration (b).

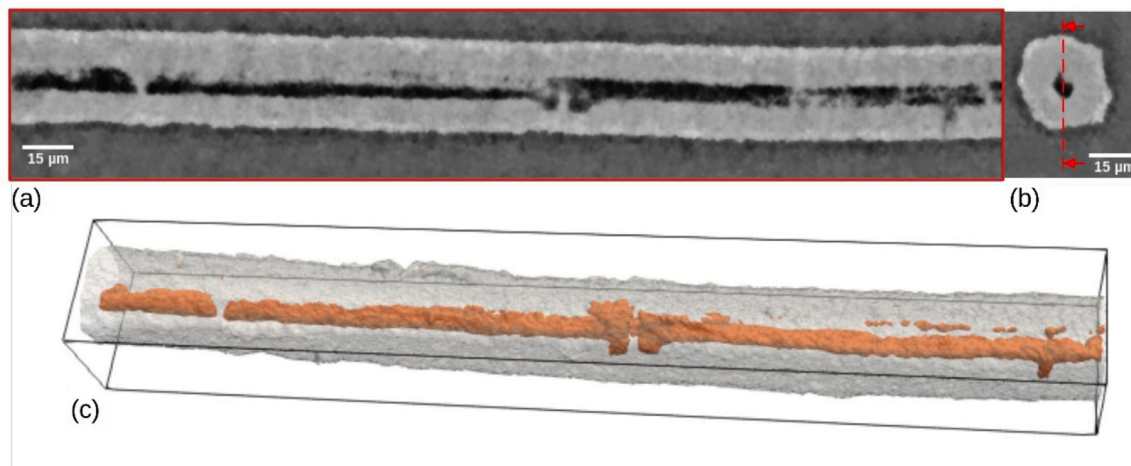


Fig. 3. X-ray microtomograms of a longitudinal (a), and cross-section (b) of a unitary fibre recorded at ambient state (T_{amb} , atmospheric pressure), and its corresponding 3D reconstruction (c).

studied unit fibre, whose width varies a bit along the studied length (300 μm), confirming previously reported observations (Beaugrand et al., 2015). Figs. 3a and 3b show longitudinal and perpendicular slices of the fibre. Based on volume quantifications, the average equivalent diameter of the fibre is around 30 μm , while the equivalent diameter of the lumen is estimated at 6 μm . The ratio between lumen and fibre volume is of the order of 4%, which is consistent with the literature data (Thuault, 2011).

Tomograms gathered Fig. 4 shows volume variations of the lumen,

for which hydration was done sequentially, and with an acquisition conducted at atmospheric pressure. Due to the specific setup adopted for such non-conventional measurements (Fig. 1e-f), a compromise was required between the image quality and the sample stability, leading to a slightly affected resolution. Four characteristic hydration times (0, 5, 15, 35 min.) of the fibre were considered in both longitudinal and transversal directions (Fig. 4a). Whatever the humidity level, the continuous character of the lumen channel along the fibre is preserved. The visual comparison of the fibre volumic changes is not obvious so data

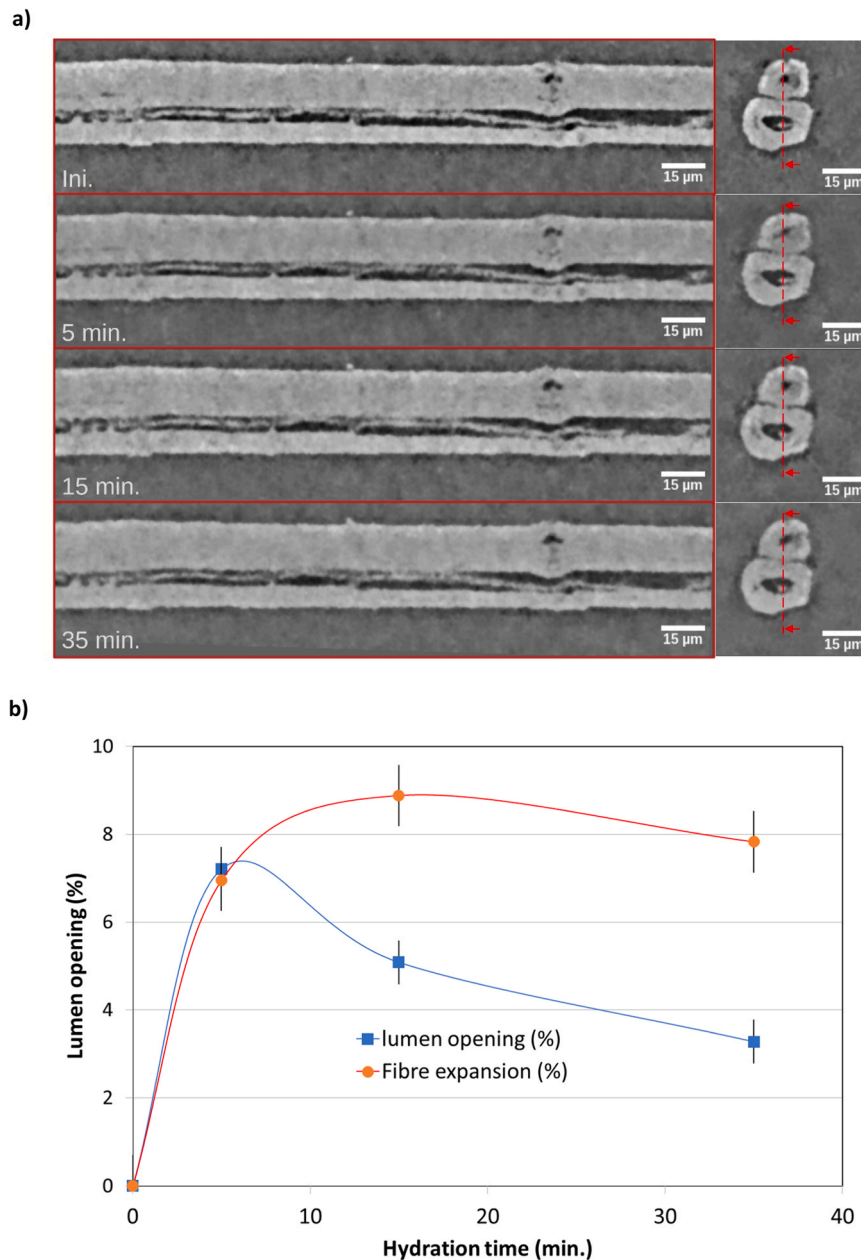


Fig. 4. X-ray micro-tomography of a unitary flax fibre in longitudinal and cross-sectional views recorded at ambient state after a variable exposition to a water saturated atmosphere (a) and the corresponding quantitative estimation of the volume variation for the lumen and the whole fibre (b).

processing was required. Fig. 4b indicates values of the lumen opening (blue squares) and of the fibre expansion (red circles) as a function of hydration time. In humid conditions, lumen opening is confirmed. Nevertheless, its volume expansion, when measured by tomography at atmospheric pressure is more limited, around 10%, in comparison to about 15% when estimated by SEM analysis (for *in situ* hydration). This would corroborate a slight overestimation of the opening rate by SEM means, potentially attributed to the low pressure in the SEM chamber. Similarly, the present values are a bit lower than that published in the study of flax fibres sorption, proposed by Lu et al (Lu et al., 2022), reporting swelling values higher than 20%. However, in that case, their samples were selected among technical bundles, which can over-estimate their sorption ability. Slightly higher moisture contents are also reported by Barbetta et al. for wood samples, but using electrolytes (i.e. not water) for which a further osmotic contribution had to be considered (Barbetta et al., 2017). If similar single flax fibres are considered, it is worth mentioning that the volumes variations found are

strictly in line with published data (Le Duigou et al., 2017b).

Another important difference of tomography results with respect to eSEM ones is highlighted in Fig. 4b. It shows the fact that the lumen swelling seems to decrease with time, while it continuously increases when recorded by SEM. Our interpretation relies on the different nature of the considered sample. Indeed, tomography measurements were carried out on single fibres to get a resolution high enough to allow a subsequent quantification. In this configuration, the fibre is free, and its behaviour is not dependent on the other surrounding fibres, as is the case in the real configuration of a compact bundle, as characterized by SEM. In this sense, we can see that the fibre itself is also subjected to a volume gain slightly higher than that of the lumen. Therefore, the weak "closure" of the lumen with hydration time could be interpreted as an indirect consequence of the fibre thickening.

4. Conclusion

An original investigation was performed to determine, at the micrometre scale, the hygromorphic behaviour of the lumen of flax under the form of unitary fibres and bundles. It involved two complementary experimental approaches using environmental SEM and X-ray micro-tomography to access the microstructure of the specimen in 2 and 3 dimensions. An opening of the lumen was evidenced and quantified by both characterization techniques and specific hydration protocols. Under *ex-situ* configuration, hydration conditions are too severe and led to an unexpected irreversible degradation of the fibre, by delamination. A softer procedure for which hydration was conducted progressively into the eSEM chamber was then proposed, giving then a more realistic value of lumen opening of about 15%. Besides, further complementary micro-tomography images were performed on unitary flax fibres. The "lumen channel" appeared continuous along the fibre. For the micro-tomography configuration, both fibre hydration and acquisition conditions are close to those met in nature (atmospheric pressure), and the lumen opening value due to humidity is again demonstrated, estimated at around 7%, even if this value can be also influenced by the concomitant swelling of the fibre itself.

CRedit authorship contribution statement

Paul Chatron-Michaud: Writing – original draft, Investigation. **Philippe Steyer:** Writing – review & editing, Supervision, Project administration, Methodology, Conceptualization. **Johnny Beaugrand:** Writing – review & editing, Validation, Methodology, Conceptualization. **Jérôme Adrien:** Investigation. **Bérangère Lesaint:** Investigation. **Victor Trillaud:** Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

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