Influence of the Absorbed Water on the Tensile Strength of Flax Fibers

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Summary: The complex structure of flax fibres involves many chemical biomolecules located in an amorphous matrix in which cellulose micrifibrils are embbeded. The drying of flax fibres influences significantly their tensile strength. This result can be explained by the creation of damages within the fibre and by the modification of the chemical composition of the matrix components. This loss of water involves a modification of the adhesion between the cellulose microfibrils and the matrix. This modification is due to the evolution of the components ensuring the transfer of load between the microfibrils and thus conditioning the strength of the cellular wall.

Keywords: cellulosic fibre; fibrils adhesion; tensile strength; water absorption

Introduction

Ecological concern has resulted in a renewed interest in natural materials. Cellulosic fibres such as flax, are an interesting, environmentally friendly alternative to the use of glass fibres as reinforcement in engineering composites. Flax fibres are very specialised cells, multinucleate but without septum, having extreme length (2-5cm) and thin diameter (15-25μm). When mature plant are pulled, most of the fibres are dead cells with very thick multilayer cell-walls. The most external layer consists of a primary wall (0.1-0.5 μm thick) coated with a polymer matrix (designated as middle lamellae and intercellular junctions) that insure the intercellular cohesion in a fibre bundle. The secondary wall is composed of 3 layers, S1 (0.5-2 μm thick), S2 (5-10μm) and S3 (0.5-1μm) whose main constituents are cellulose microfibrils with particular orientations [1]. Ultimate tensile strength, elastic modulus and ultimate elongation do mainly depend on both the cellulose content and the angle between microfibril and fibre axis. The mechanical properties of fibres are essentially due to the cellulose microfibrils locked into an almost axial direction (angle of 10° according to Wang[2]) in the layer S2.

The main components of primary wall and cell junctions consist of pectins, i.e. a complex polysaccharide family whose two of them principally respond to growth conditions that is 1)

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homogalacturonans and 2) rhamnogalacturonan I (RG-I). Pectins are polyanions and hence are responsible for the main part of the adsorbed water in the cell wall, especially homogalacturonans which have a high charge density. In the cell walls, pectins organise water molecules in some kind of network of micropores and macropores [3,4]. The micropores correspond to the Donnan space where the number of water molecules depends on the nature of cations and also on the charge density while the macropores constitute a water free space where counter ions are expelled.

In contrast with the lignocellulosic fibres of wood, flax fibres almost lack lignin but cellulose microfibrils are embedded in 5-15% of non cellulosic polymers (NCPs), and are designated as cellulosic fibres. Long chains of pectic β1-4-D-galactan (DP 10-30) have been clearly identified as the main encrusting component of ultimate fibres [5-11]. They are branched on a rhamnogalacturonan backbone whose charge density is about half that of homogalacturonan. Again, these pectic components are the main components interacting with water in the secondary wall. Hemicelluloses, present in few amount, interacting strongly with the cellulose molecules at the surface of the microfibrils contribute only partly to the water adsorption. Thus, cristalline cellulose-microfibrils, coated by hemicellulosic cross-linking glucans and (galacto)glucomanans, are embedded in a pectic galactan matrix. Acidic proteins such as arabinogalactan proteins (AGPS) can also take place within the matrix of the secondary wall and contribute to water adsorption.

The interactions of water (hydrogen bonds) with fibres occur mainly via the hydroxyl groups of polysaccharides [12], at least those that are not involved in cristallites. According to Chauban [13] the adsorption of water can be divided into 2 parts : water at the surface of the fibre and ii) water within the cell walls. As described above, the level of adsoption at the fibre surface would be high and due to the large amount of pectins. It will depend on the degree of retting of flax, a microbial process that progressively degrades cell wall polysaccharides, and also of the subsequent mechanical/chemical treatments which strongly influence the surface composition [14-15]. On the other hand the adsorption of water in the secondary wall is multicomponents although essentially due to the pectic –proteoglycan matrix. Thus, the percentage of water linked to each cell wall components greatly depends on process [16] but also on variety and ecophysiology. According to the literature, the moisture of flax fibres is within a range of 6 - 10 %.

For the polymer reinforcement, the water presence in fibres is synonymous of volume variation (swelling / shrinkage), degradation, poor interfacial bonding between fibre and

matrix, and water vaporisation during the process (with voids development). For the manufacture of composite material the influence of water is still an open question. In this text we are interested in the influence of a drying, therefore with a loss of water absorbed, on tensile strength of flax fibres.

Experimental

Flax plants, variety Ariane, were cultivated in Normandie (France) and retted on the field (dew retting). After harvest, the stems were scuched and the fibres combed. The percentage of absorbed water in flax fibres (n samples of x g) was calculated [as (Ww-Wd)/Wd] by weight difference before (Ww) and after drying at 105°C for 12 hours (Wd). The longitudinal tensile behaviour of flax fibres was measured using the standard method for single material (NFT 25-704, ASTM D 3379-75), by taking into account the compliance of the system. The range of the load cell is 0-2N and the precision of measure of displacement is one micron. Test conditions are: speed 1 mm/mn, and fibre length 10 mm.The tensile tests are carried out on raw fibres, drying fibres (14 hours at 105 °C) and drying fibres having reabsorbed water in contact with the ambient air (storage 8 days at 20 °C under the same conditions as raw fibres).

Results and discussion

After drying the average lost weight is 8.3%, and in contact with the ambient air the fibre reabsorbs water (Figure 1). The mass increase is rather fast and the fibres have recovered their initial mass after 5 hours.

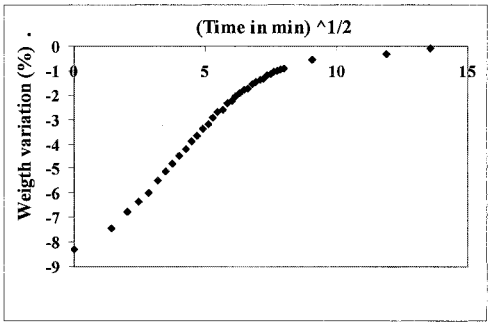
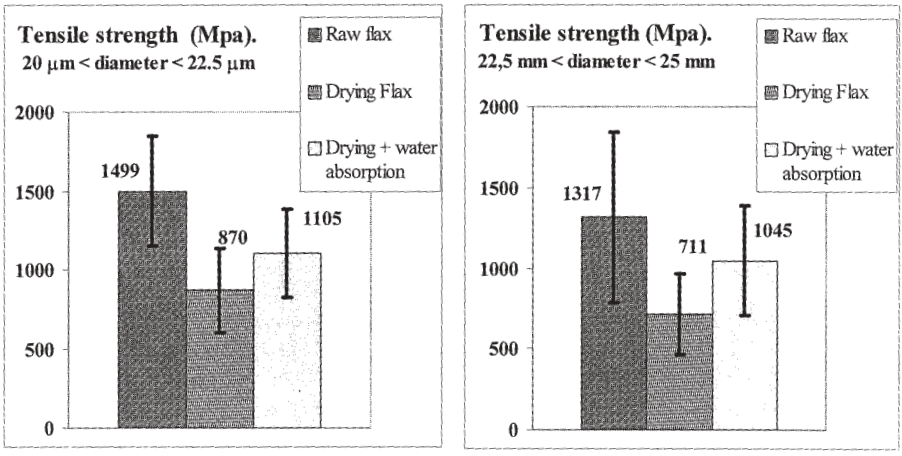


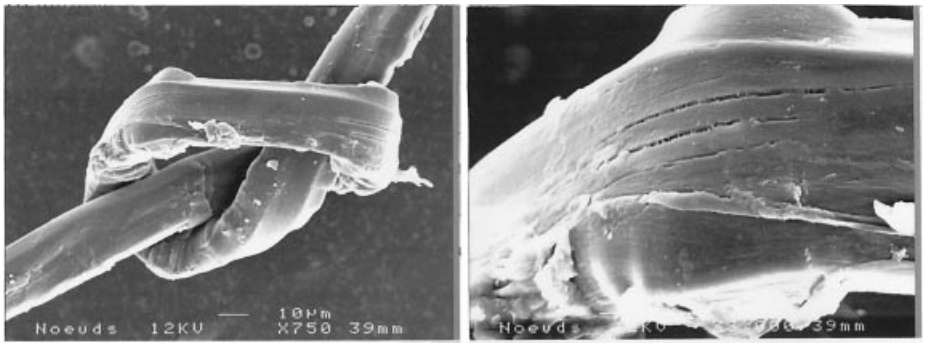
Figure 1. Evolution of the flax fibres mass in contact with the ambient air after drying (14 hours at 105°C).

Mechanical properties of raw flax fibres were presented in a preceding article [17], we notice that Young's modulus and tensile strength are a function of fibre diameter. The tensile strength are indicated for two classes of fibres diameter (20-22.5 μm and 22.5-25 μm) according to the treatments undergone (Figures 2 and 3). One notices, for the two classes of fibres, the influence of drying on tensile strength which results in a the fall of the mechanical properties. After water absorption, the fibres do not recover their initial properties. This result can be explained by the creation of damage within fibre and by modification of the chemical components location in the fibres. In addition the results are more dispersed than before drying. Dispersions of fibre properties are due to two main factors: 1) the fibre development in the stem and 2) the transformation process.

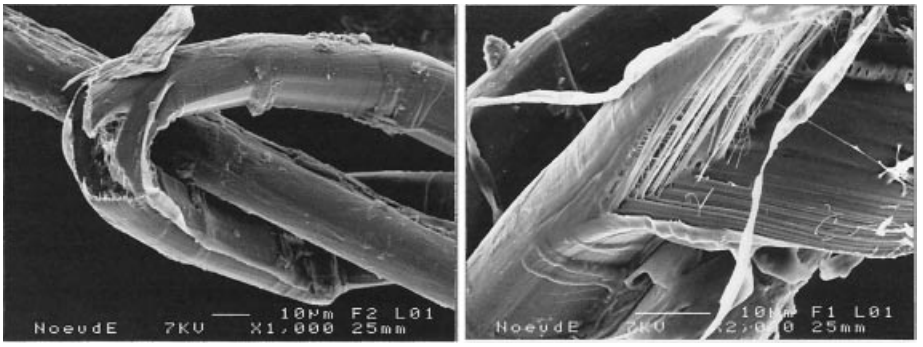


Figures 2 and 3. Drying influence on tensile strength of flax fibres.

To illustrate the influence of the absorbed water by a flax fibre on the damage mechanisms, a node is made on fibre, then observed under SEM. The operation is carried out on a raw fibre and on a fibre after drying (14 hours with 105 °C). For a fibre with moisture content of 8.3 % (raw fibre), we notice two damage mechanisms (Figures 4 and 5): buckling in the compression area and cracking in the tensile area (these cracks are explained simply by the Poisson's ratio of fibre). After drying, we notice a loss of cohesion between the matrix and the microfibrils which seems to be mainly influenced by the absorbed water (Figures 6 and 7).



Figures 4 and 5. Damages in a flax fibre with a moisture percentage of 8.3 %.



Figures 6 and 7. Damages in a flax fibre after drying.

Astley and Donald [18] studied the effect of the hydration on the distribution of cellulose microfibrils of the S2 layer (secondary wall) of a flax fibre using small angles x-rays scattering. Water penetrates in the amorphous zones and enters in competition for the sites of potential hydrogen bonds, involving a loss of the interactions between crystalline areas (cellulose microfibrils) and the amorphous matrix. This effect involves an increase in the strain at failure as reported by [19] and Joffe [20] who considers that the absorption of water has a plasticizing effect on a flax fibre In addition, Netravali [21] claimed, without indicating experimental results, that repeated moisture absorption/desorption cycles can significantly lower the fibres strength. The percentage of water absorptive in mass by a flax fibre is general information because this percentage is not homogeneously distributed in the thickness of fibre, the exchanges with the air being done on the surface [22]. Moreover, its structure is microporous which influences the retention of water [23].

The thermal stability of flax fibres is a significant parameter for the processing of composite materials and it is generally admitted that the low thermal stability of vegetable natural fibres presents a limit at their use [24]. This is particularly true for the thermoplastic matrices whose process temperatures are high. Therefore, during the temperature increase one notes:

- a vaporisation of absorbed water,
- the development of internal mechanical strains. The thermal expansion coefficients of the components of the cellular walls are different. An increase in temperature will create irreversible damages and cracks affecting the physical properties of the fibres [25],
- Physicochemical modifications of the components, and possibly a degradation beyond a certain temperature.The loss of water starts at 60°C and at 120°C there is no more water and there is a degradation of waxes, at 180°C there is a decomposition of pectins and at 230°C a degradation of hemicelluloses and cellulose is claimed [26].

The definition of the acceptable temperatures of transformation of vegetable fibres is studied by several authors. The evolution of the fibres properties is a function of the temperature and the time. 200°C mustn't be exceed for more than 5 min (current data) for vegetable fibres [20,27] and the ideal is to not exceed 160°C which is a low temperature for thermoplastic processing.

Conclusions

The drying of flax fibres influence significantly the tensile strength and after water desorption/absorption cycle the fibres do not recover their initial properties. This result can be explained by the creation of damages within the fibre and by the modification of the chemical composition of the matrix components. This loss of water involves a modification of cohesion between the cellulose microfibrils, modification due to the evolution of the components ensuring the transfer of load between the microfibrils and thus conditioning the strength of the cellular wall. For the processing of composite materials, the limited thermal stability of flax fibres is a key parameter to be taken into account for the choice of polymer and the thermal cycle. It is worth noting that such mild and specific treatments could keep to fibres the maximum of their native properties.

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