The elaboration of composites from biomaterials is gaining more and more importance in the scope of sustainable development and environmental protection. Thus, more environmental friendly composites are produced today by reinforcing bio-polymers with natural fibres. Among such fibres, flax is known for its remarkable mechanical properties. The industrial production of flax-fibre reinforced composites is however underdeveloped and, to fill this lacuna, it will be necessary to know more about morphological and mechanical properties of the fibres. In a previous study concerning two varieties of flax fibres, a wide dispersion of microstructural parameters (fibre size, porosity) and mechanical properties (Young’s Modulus, strength and ultimate strain) was observed. In the present study, seven varieties of flax fibres were compared in terms of the mean values and the dispersions of their morphological and mechanical properties. It is expected these results will help for choosing the most suitable variety of flax fibre to tailor composite materials with respect to high performances (highest mean values) or to reliability (lowest dispersion).

1 Introduction

The association of natural fibres with polymer matrices offers an opportunity to extend the range of structural materials, which contributes to sustainable development when bio-polymers are used. Among the possible continuous natural fibres, flax is the best candidate because of its high specific mechanical properties [1] [2].

In this study, we have investigated the morphological and mechanical properties of seven varieties of flax fibres, and we have analysed the dispersions associated to these parameters. The goal was to determine which variety of flax, among those commonly grown, is able to impart high properties or good reliability to the derived composite materials.

2 Material and method

2.1 Flax fibres

Seven varieties of flax fibres harvested in the same area in the North-West of France are investigated. The tested varieties are: Agatha, Alizée, Drakkar, Hermes, Marylin, Melina and Suzanne.
Comparison of morphological and mechanical properties of seven varieties of flax fibres

wall, three secondary walls and the lumen. The secondary layers are an arrangement of non-crystalline compounds and crystalline microfibrils. The amorphous phase mainly consists in hemicellulose and lignin, the fibrils of cellulose being dispersed around. The specific angle of the microfibrils depends on the considered layer. The layer referred to as S2 in figure 2 is the thickest one and is the main source of fibre properties.

In this study, the surface of the lumen is disregarded in accordance with the finding of Charlet et al. [3] that there is no significant difference between the mechanical properties if the lumen is included or not in their calculations.

![Fig. 2. Structure of a flax fibre [4].](image)

2.2 Size measurements

Each fibre is glued onto a paper frame with a gauge length of 10 mm in order to measure its size and determine its mechanical properties.

The diameter of a flax fibre is not the same along the length of the fibre. Since we cannot know the exact diameter at the point where the fibre breaks during the tensile test, we calculate an average diameter from three measurements (cf. figure 3). These measurements are taken from three pictures taken with an optical microscope (Olympus BX 41M, magnification: x 20) at regular intervals along the fibre.

![Fig. 3. Three diameters along the same flax fibre.](image)

2.3 Mechanical properties

The ultimate strain and strength) are measured from tensile stress-strain curves of single fibres with a gauge length of 10 mm:

\[ \sigma = \frac{F}{S} \]  

where \( \sigma \) is the stress, \( F \) the applied load, \( S \) the cross-section area of the fibre, \( \varepsilon \) the deformation. The tests are achieved with a tensile test machine (Instron 5566), equipped with a 10 Newtons load cell. At least 40 fibres were tested for each flax variety.

The shapes of the tensile curves vary a lot from one fibre to another. However, as illustrated in figure 4, three distinct domains are commonly observed. These domains are assumed to correspond to the following steps of deformation of fibres during the mechanical tests [5], [6] [7]:

I. Elastic deformation of cell walls (initial linear part of the curve)

II. Progressive alignment of microfibrils

III. Elastic deformation of aligned microfibrils
Comparison of morphological and mechanical properties of seven varieties of flax fibres

Fig. 4. Tensile test of a flax fibre. The Young’s modulus is evaluated by the slope of the stress-strain curve in the third domain.

3 Results

3.1 Statistical representativeness

As the mechanical properties of flax fibres exhibit a wide dispersion, a too small sample will not give representative results. Obviously, the accuracy of the results increases with the sample size. Unfortunately, the testing procedure is tedious and time consuming. It is thus necessary to optimise the number of tested fibres.

Moreover, the classical mechanical properties are determined from tensile tests performed on 40 fibres for each flax variety as previously stated. But these tests are considered to be valid only if the fibre breaks outside the wedges. It implies that almost half of the fibres must be discarded after testing. This study ultimately deals only with some twenty fibres for each flax variety and it is necessary to check whether this number is sufficient to provide meaningful results.

Contrary to mechanical properties, the diameter can be measured on the 40 fibres of each variety. This is the reason why we have chosen to study the sample size on this parameter. For each variety, the deviation, in percentage, between the global average of the 40 fibres and the cumulative average is calculated. In all cases, the deviation from the mean value decreases rapidly with the number of fibres taken into account as can be seen in figure 5. For most varieties, a number of fibres greater than 15 insures a deviation lower than 5%, which results in quite satisfactory accuracy [8].

Fig. 5. Percentage of deviation from the value of the average diameter as a function of the number of fibres considered for each variety.

3.2 Intercomparison among the different varieties

The study compares seven varieties of flax with respect to their morphological and mechanical properties. The results for the fibre diameter, the Young’s modulus, the strength, and the ultimate strain are presented in figures 6 to 9. The mean values and the standard deviations corresponding to each variety are reported in the graphs.

For comparison purposes, the global mean value of each parameter (M) and the global standard deviation (Sd) have been calculated taking into consideration all the varieties together. The upper and lower lines in figures 6 to 9 correspond to (M+Sd) and (M-Sd), respectively.

Firstly, we see in all figures that there is a wide dispersion of values for each variety. In all the graphs, the vertical bar associated to each mean value indicates the magnitude of this dispersion. Note that the height of the vertical bars represents the dispersion of the raw data and not that of the mean values.

Yet we chose varieties coming from the same geographical area and harvested by the same producer, so as to keep a traceability of the species. Moreover, the storage of the fibres and the experimental conditions have been standardized in order to minimize as much as possible the dispersion factors. But even taking all these precautions, we obtain a wide dispersion of the properties for the same variety.
Comparison of morphological and mechanical properties of seven varieties of flax fibres

This dispersion can be attributed to several factors such as:
- climate and weather hazards during the growth of the plant
- chemical composition of the fibres: amount of cellulose, hemicellulose, lignin…
- conditions of retting and scutching
- conditions and time of storage
- environmental conditions during the experiments (more particularly humidity and temperature).

The variations may also be explained by some properties inherent to a given fibre like its amount of crystallinity or amorphousness, the orientation of its molecular chains, the number and the position of defects and cracks or the degree of polymerization [9]. This can explain the variation within a same variety of flax even if we tried to minimize at best the influence of these parameters.

Secondly, for all the parameters (figures 6 to 9), we can see noticeable differences in the results from one variety to another. These variations could have been explained by a biocomposition peculiar to each species. However, Thuault et al. [7] showed that no clear distinction between varieties can be made on the basis of their biocompositions. This observation is coherent with the fact that no systematic trend can be identified from the present results. All the mean values and most of the vertical bars are inside the
Comparison of morphological and mechanical properties of seven varieties of flax fibres

interval represented by the two horizontal bars (M+Sd) and (M-Sd).

However, we can try to make a classification of the different varieties of flax on the basis of their mechanical properties. We can imagine two kinds of classification according to either the mean values or the dispersion of the properties. Therefore, considering a given mechanical property, the difference between the maximum mean value obtained for all the varieties and the minimum one is divided arbitrarily into five equal classes. Each variety is then numbered according to the class to which its results belong. Summing these numbers for the three mechanical properties examined allows us to make a ranking of varieties in a simple way. This classification is presented in table 1 in which the highest numbers correspond to the best mechanical properties. It can be seen in this table that the variety Agatha presents clearly the best combination of Young’s modulus, strength and ultimate strain in terms of performances.

<table>
<thead>
<tr>
<th></th>
<th>Marylin</th>
<th>Alizee</th>
<th>Agatha</th>
<th>Drakkar</th>
<th>Hermes</th>
<th>Melina</th>
<th>Suzanne</th>
</tr>
</thead>
<tbody>
<tr>
<td>Young's modulus</td>
<td>1</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>2</td>
<td>5</td>
<td>3</td>
</tr>
<tr>
<td>Ultimate strain</td>
<td>1</td>
<td>2</td>
<td>2</td>
<td>1</td>
<td>5</td>
<td>3</td>
<td>5</td>
</tr>
<tr>
<td>Strength</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>4</td>
<td>2</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Total</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>8</td>
<td>9</td>
<td>13</td>
<td>13</td>
</tr>
</tbody>
</table>

Tab. 1. Classification of the seven varieties of flax as a function of the mean values of their mechanical properties.

The same procedure is repeated using the standard deviations of the mechanical properties instead of mean values. It must be noted that the relative standard deviation, commonly called “coefficient of variation”, is preferred here in order to eliminate the influence of the magnitude of the mean value. The classification of the relative standard deviations is presented in table 2. In this table, the lowest numbers correspond to the less dispersed values of mechanical properties. One observes that the best results correspond to the Marylin and Alizee varieties while the Melina and Suzanne varieties lead to much more dispersed results.

<table>
<thead>
<tr>
<th></th>
<th>Marylin</th>
<th>Alizee</th>
<th>Agatha</th>
<th>Drakkar</th>
<th>Hermes</th>
<th>Melina</th>
<th>Suzanne</th>
</tr>
</thead>
<tbody>
<tr>
<td>Young’s modulus</td>
<td>4</td>
<td>5</td>
<td>2</td>
<td>2</td>
<td>1</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>Ultimate strain</td>
<td>4</td>
<td>1</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Strength</td>
<td>5</td>
<td>3</td>
<td>4</td>
<td>2</td>
<td>2</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Total</td>
<td>13</td>
<td>9</td>
<td>9</td>
<td>8</td>
<td>8</td>
<td>7</td>
<td>6</td>
</tr>
</tbody>
</table>

Tab. 2. Classification of the seven varieties of flax as a function of the value of their relative standard deviations.

If we combine the results of tables 1 and 2, we can conclude that the Agatha variety presents the best compromise between high values and low dispersion of the properties.

4 Conclusions

The purpose of this study has been to identify the variety of flax fibres that would allow for the most efficient reinforcement of a composite.

The experimental results confirm that, for a given variety of flax, there is a huge deviation of properties from one fibre to another (the average reduced standard deviation for the values of the mechanical properties is roughly 40%).

Moreover, when a comparison is made between seven varieties of flax, it may be concluded that the dispersion of the results does not allow for a statistically significant distinction between all the varieties.

Nevertheless, a classification of the varieties can be made on the basis of the mean value or the dispersion of the mechanical properties. This comparison makes it possible to choose the most suitable variety of flax to reinforce a composite with respect to its performances (highest mean values) or to its reliability (lowest dispersion).

Acknowledgements

The authors are grateful to the “Réseau Inter-Régional “Matériaux Polymères, Plasturgie” du Grand Bassin Sud-Parisien” for its PhD fellowship financial support and its financial participation to the experimental set-up used in this work.
Comparison of morphological and mechanical properties of seven varieties of flax fibres

References


