Deliverable n°1.2.1

CATALOGUE OF PROPERTIES OF SELECTED RAW MATERIALS

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PP LEADER INRA

European Regional Development Fund

Partners

PP Leader : INRA

Partners involved : UBS

Content

- Material :

Two different batches of flax tows and one batch of flax fibers grown in Normandy were provided by our partner DEPESTELE (Figure 1). The first batch is a Bolchoï variety considered of good quality with a low quantity of shives (<5%). The second one is also a Bolchoï variety with a lot of shives (>5%) considered as a lower quality. The last batch (n°8072) is a Bolchoï variety of flax fibers which are scutched.

Figure 1 : Pictures of the material provided by DEPESTELE : a) High quality tows b) Tows with shives c) High quality fibers 8072es

PLA fibres have also been studied. PLA is the second component of non-woven preforms for POP products. The objective of this WEB is to quantify their morphology, mechanical performances and rheological behaviour.

- Biochemical analysis :

Biochemical analysis was performed at INRA Nantes in order to determine the monosaccharides composition of the different batches using Gas Phase Chromatography (GPC). The results are presented in Figure 2.

The majority monosaccharide in the three lots of flax is glucose, the results are expressed as a percentage of the dry mass used. Glucose is substantially the same for high quality tows and fibers 8072 with 78% and 81% respectively. The tows with shives have a little less glucose with 71%.
Between the two batches of tows, there is another difference : the tows with shives have a higher proportion of xylose, characteristic of shives. Concerning high quality tows and fibers 8072, there are some slight differences (arabinose, mannose) but not significant and that can be attributed to the different year of harvest.

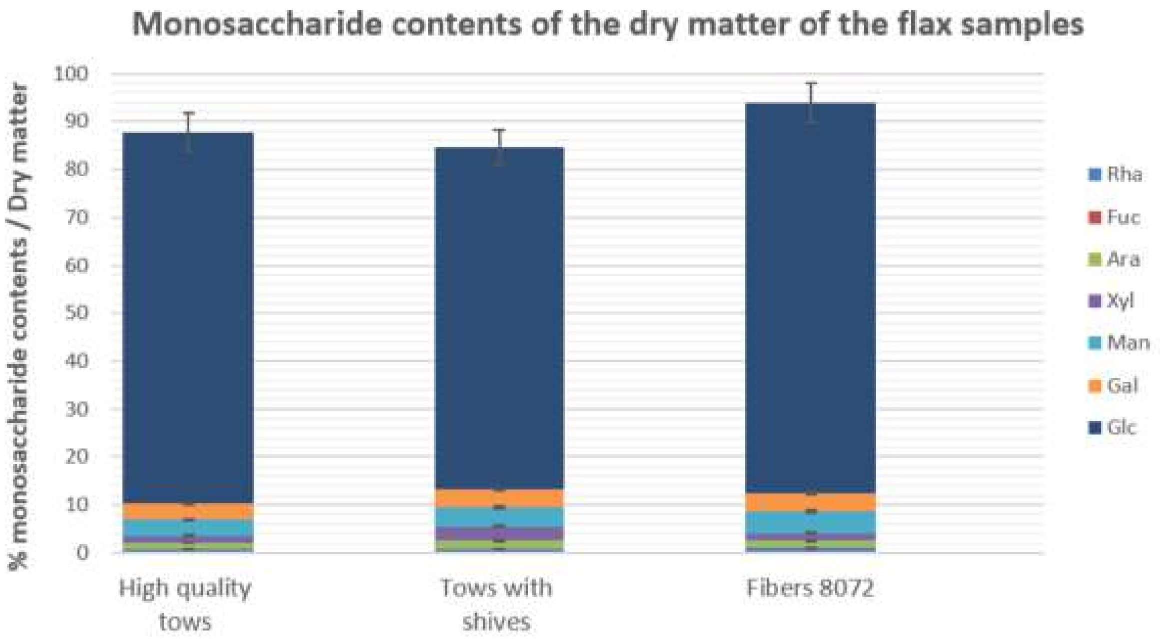


Figure 2 : Monosaccharide contents of the different flax batches

Nitrogen and carbon elements are dosed using the Dumas method. The sample is heated to 1000 ° C in the presence of oxygen for combustion. Then it is possible to quantify nitrogen and carbon.
The percentage of protein can be traced back to the percentage of nitrogen by the following formula :
$N\ (\%) * 6.25 = Proteins\ (\%)$ where 6.25 is a conversion factor.

Samples	N [%]	C [%]	Proteins [%]
High quality tows	0,35 (± 0,01)	42,45 (± 0,38)	2,21 (± 0,02)
Tows with shives	0,4 (± 0,01)	42,27 (± 0,14)	2,49 (± 0,01)
Fibers 8072	0,19 (± 0,01)	41,79 (± 1,11)	1,17 (± 0,07)

Table 1 : Dosage of C and N elements and proteins

The percentage of carbon is stable and similar for the three batches of flax. In contrast, the percentage of nitrogen is different. A factor of 2 is noted between the fibers 8072 and the two batches of tows. The result is the same for proteins : tows have twice as much protein as scutched fiber.

❖ Hygroscopic behavior :

Dynamic Vapor Sorption tests were also conducted on the three batches in order to determine the water sorption isotherms. The measurements were made at 25°C with a sorption cycle (0% to 90%) and then a desorption cycle (90% to 0%). In both cases the bearings are made of 10% by 10% with an equilibrium time between 120 minutes and 200 minutes. For each measurement, between 5 and 10mg of matter was placed into the microbalance inside the equipment.

First, both graphs are characteristic of natural fibers. The two batches of tows exhibit similar behavior despite their different amount of anas. It is possible that in the weighed sample, the amount of shives is very small.
Then, HQ tows were compared with 8072. And a difference is observed for 90% where HQ tows are more sensitive to sorption than 8072. This difference could be explained by the presence of shives in tows.
Finally, there are three different areas in the RH range. From 0% to about10%, the different curves are superimposed ; the water is strongly bound under the effect of van der Waals forces and forms a monolayer on the sample. From 10% to 65-70%, the water is moderately bound ; the molecules continue to adsorb and pile up on the already existing monolayer forming a multilayer. And beyond 70%, the water is called free.

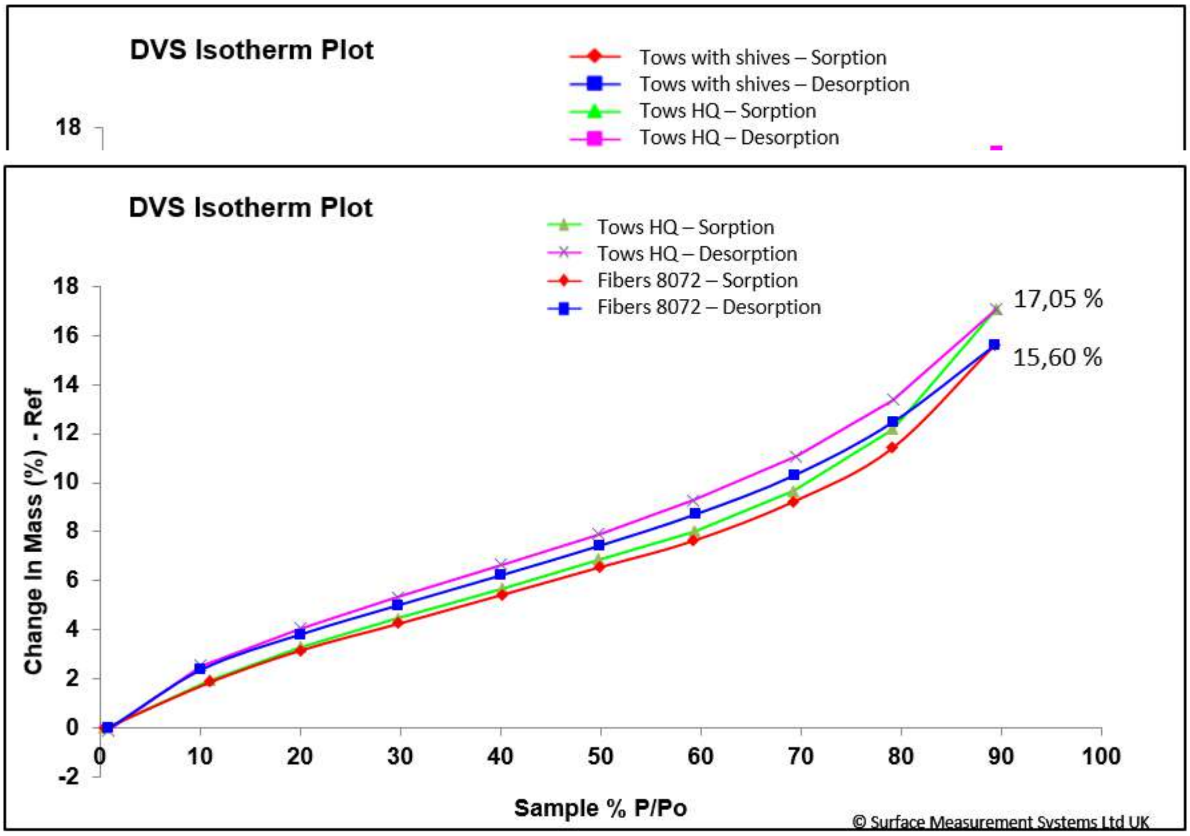


Figure 3 : DVS isotherm of the two batches of tows (upper) and DVS isotherm of fibers 8072 and HQ tows (lower)

❖ Fineness :

The fineness of the different batches was assessed by morphological analysis using Qicpic equipment. The fibres were cut in 1-2 mm and dispersed in water (50mg into 1L). The geometry of millions of particules was analysed, leading to statistical results summarized in Figure .

For each sample, three replicates were completed at QICPIC ; this is the average curve that can be seen in Figure 4. The fibers 8072 have a larger average diameter than the two batches of tows. At the same time, the two batches of tows are relatively similar from a diameter point of view. The presence of anas in different quantities does not seem to influence the measurement of the diameter. And the distribution density chart shows a larger number of bundles for the fiber 8072. Further investigations will be conducted to explain the origin of the diameter difference.

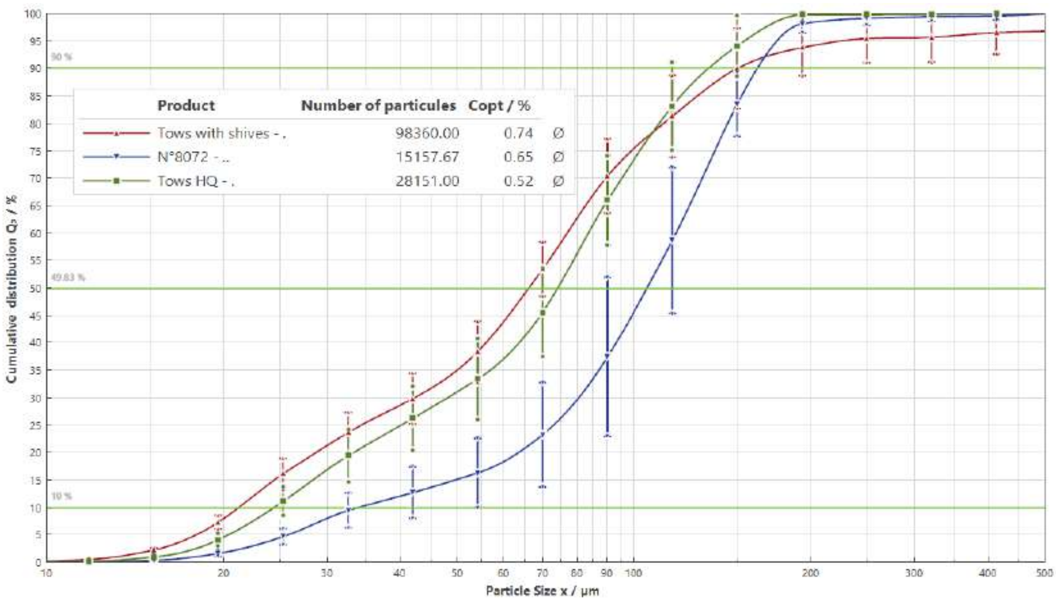
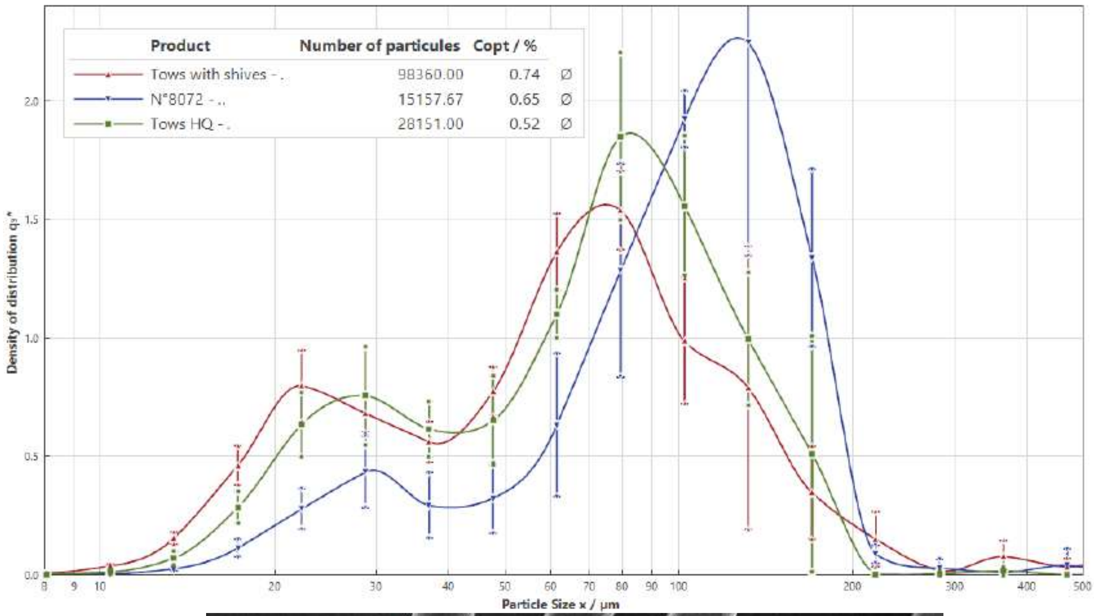
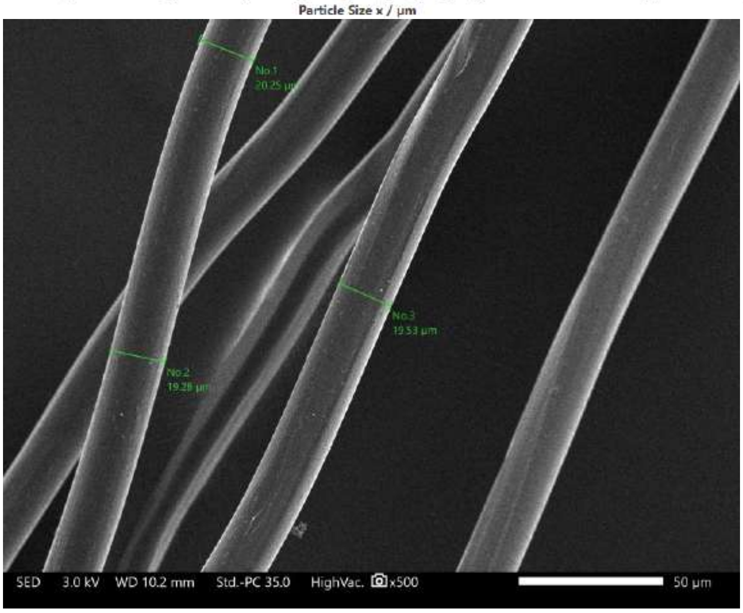


Figure 4 : Cumulative distribution (upper) and density of distribution (lower) of the diameters of the fibres

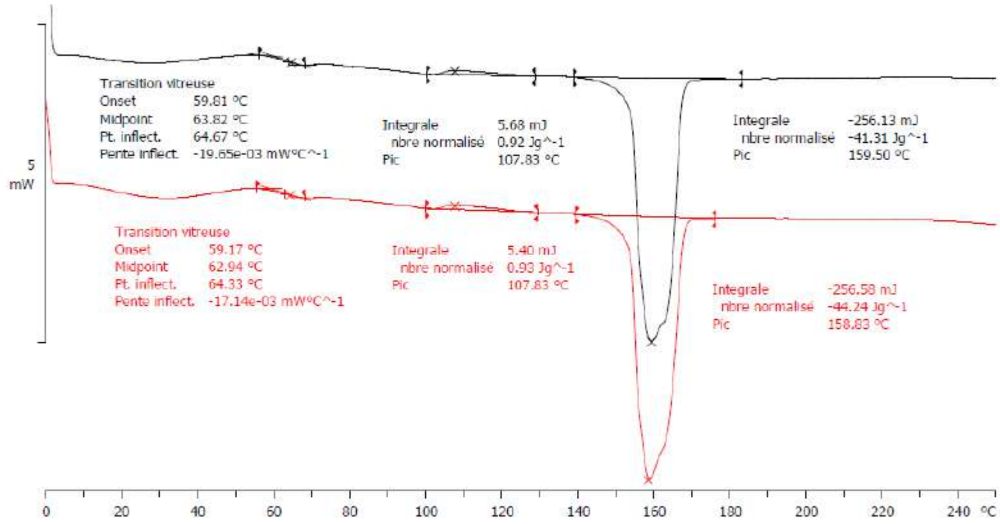
❖ Characterization of PLA fibres :





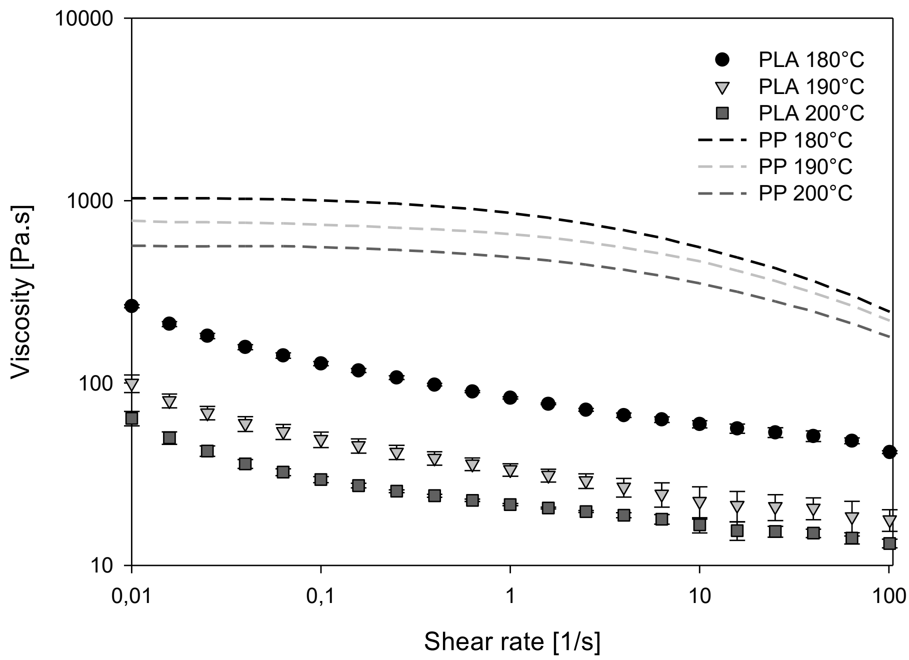
SEM image of PLA fibres

With an average diameter of 19.7 ± 0.3 µm, diameter of PLA fibers is reproducible and in the same range of flax fibres' one.



DSC thermogram of PLA fibers

DSC investigations evidences a melting temperature of 159°C. This temperature is fully compatible with degradation temperature of flax fibres; PLA can be used for the manufacturing of industrial parts made of PLA and flax and processed by thermocompression cycles.



Evolution of the viscosity of PLA fibers according to the selected temperature. Comparison is done with PP

Interestingly, for same temperature and shear rate, PLA exhibits a lower viscosity than PP, showing the interest of this matrix in term of impregnation. For example viscosity of PP and PLA at 190°C are 551 Pa.s and 27 Pa.S, respectively. These values are given for a shear rate of $4s^{-1}$, representative of a compression moulding process. Due to the low viscosity value of PLA, adaptation of the process conditions will be probably mandatory to obtain a satisfying quality of parts or plates.

	Young' modulus [GPa]	**Max. strength [MPa]**	**Strain at failure [%]**
PLA (DS fibres)	3.04 ± 0.06	43.5 ± 1.2	2.6 ± 0.47
PLA (3001D)	3.80 ± 0.14	61.5 ± 0.8	3.85 ± 1.15
PLA (*Oksman 2003*)	3.4 ± 0.1	50.3 ± 2.4	2.0 ± 0.2

Mechanical performances of PLA fibres (after a cycle of compression moulding)

A plate has been manufactured by compression moulding from the provided fibres, dog bone specimens have then been shaped and tensile characterized. Results are signicantly lower than usual mechanical properties of PLA.

NEXT STEPS

- Dosage of lignins by the acetyl bromide method
- Curves of Dynamic Vapour Sorption for the shives
- More investigations with the QICPIC