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Technical report describing the scientific conclusions of the permeability measurements

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Content

- Objectives:

The objective of this report is to provide a scientific assessment of the experimental methodology, discussing kinetics of the resin in both transverse and longitudinal directions of the biaxial Non-crimped fabrics (BX NCFs) elaborated in the previous activity. These results will enable recommendations to modify and optimise the textile architecture of the biaxial reinforcement in order to obtain the most favourable configuration for resin impregnation.

- Materials:

The NCFs are provided by Teillage Vandecandelaère and will be compared with commercial available references supplied by Safilin, Sicomin, Terre de Lin and BComp company.

Samples	Producer	Fibres	Areal weight (g/m^2)
BX TV 312	TV	Flax	312
BX TV 424	TV	Flax	424
BX SF 600	Safilin	Flax	600
BX BC 350	BComp	Flax	350
BX TDL 250	Terre de Lin	Flax	250
BX GF 600	Sicomin	Glass	600

Saturated permeability (at UBS) characterisations were performed with canola oil supplied by METRO ($72 \leq \eta \leq 85$ mPa.s).

Unsaturated permeability (at UoCambridge) characterisations were performed using Infugreen 810 resin with standard hardener SD 8824 (Sicomin Epoxy Systems, France). Infusions took place within 30 minutes of resin mixing, with the impregnation process taking between 2 to 5 minutes. Infusion was carried out under ambient conditions (ca 15 °C), at which resin viscosity is estimated to be around 400 mPa.s.

- Methods:

Unsaturated permeability was assessed by tracking the progression of the flow front in a vacuum assisted resin infusion process, with the pressure kept between $\Delta P = 0.85\text{-}0.95 \times 10^5$ Pa (85%-95% vacuum). Three layers of the preform fabric were infused through central injection, with the rigid mould thickness kept constant at ca 2-2.5mm. A high definition digital camera was used to record the infusion process; the video files were converted into image files with time stamps.

To calculated the unsaturated permeability Kunsat of the flax preforms, the flow front distance (x) at specific orientations (45° intervals) was measured for the corresponding filling time (t) through image analysis in ImageJ. Unsaturated or transient permeability Kunsat refers to the permeability of a fabric while it is being freshly impregnated (i.e. full saturation has not occurred). Kunsat can be calculated by rearranging Darcy's law into equation 1, where x is the position of the flow front at time t, φ is the fabric porosity, ΔP pressure gradient applied, and μ is the resin viscosity. Fabric porosity φ is directly related to the fibre volume fraction (1-vf); in our case, for BX TV 312 φ = 0.76 (for 30wt% or 24v%) and BX TV 424 φ = 0.66 (for 40wt% or 34v%). Plotting the square of the flow front position x^2 against fill time t would produce a straight line graph, and the slope of the graph x^2/t can be used as an input in equation 1 to determine Kunsat. Only unsaturated permeability in specific in-plane orientations was determined.

$$K_{unsat} = \frac{x^2}{t}\frac{\phi\mu}{2\Delta P}\text{, where } \phi \equiv 1 - v_f \quad (1)$$

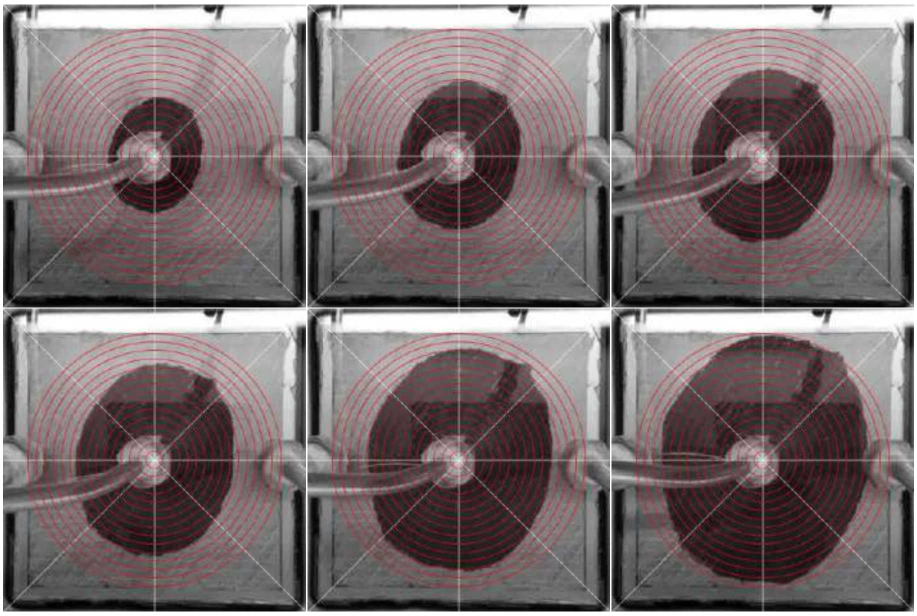


Figure 1 – Infusion of the BX TV 424 preform. Images at t = 30s, 60s, 90s, 120s, 180s and 240s. Red radial grid denotes x = 5 mm distances, with total infusion area approximately 150 mm square. Angles are at 0, 45, 90, 135, 180, 225, 270, 315 in the clockwise direction, with 0 being perpendicular to the stitch direction.

Saturated permeability was assessed by the EASYPERM bench method which is based on the modified Darcy's Law below:

$$Kxy = \frac{Q.\eta.\Delta L}{A.(Pi - Pe)} = \frac{Q.\eta.\Delta L}{A.\Delta P}$$

Where Q is the controlled constant injection flow, η the oil viscosity measured by the ISO 24314 viscosity flow cup, ΔL the sample dimensions, ΔP the pressure variation and A the feed hole section of the top injection point (fig.X).

During **in-plane** measurement the pressure variations are recorded on 6 points (One at the injection point, 2 along the X-axis, 2 along the Y-axis and the last one on the XY-axis) (fig.1). Saturated permeability is obtained by measurement of velocity flow Q when the pressures are stabilized.

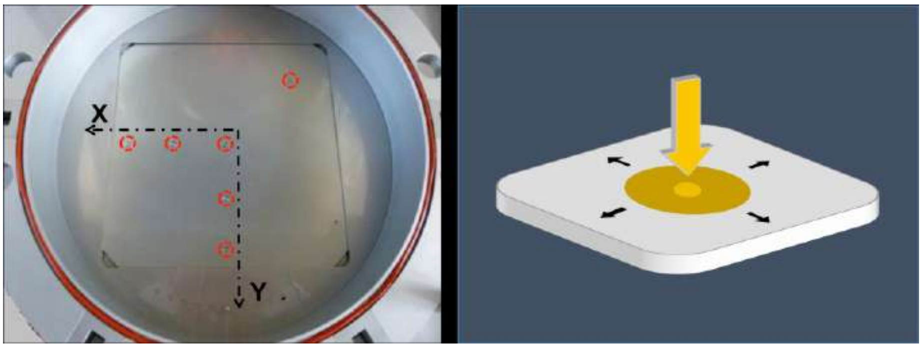


Figure 2 - Line sensors within the mould (a) and scheme of the radial injection (b)

The same method was used for **transverse** permeability characterization. Only the shape of the mold changed and pressure variations are recorded between the injection and the event point (fig.2).

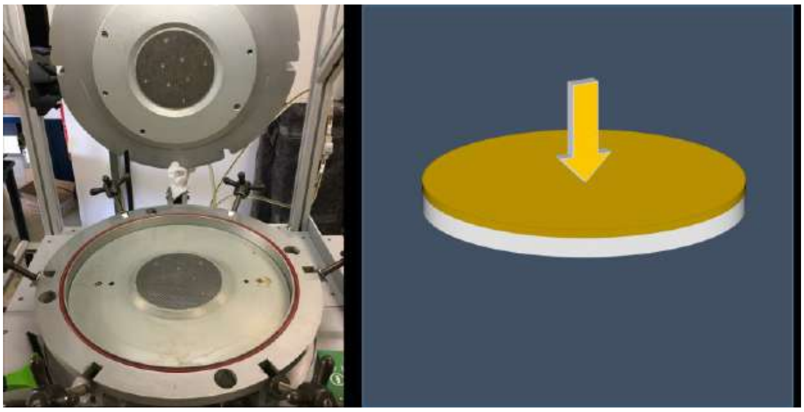


Figure 3 - Circular mould used for the transverse impregnation (a) and oil flow through the sample

Sample were cut out from one layer of the fabrics in square and circular shape for respectively longitudinal and transverse measurement. All calculations were made in triplicate. The fiber volume fraction was set to 40% by varying the thickness of the mold which remain constant during the impregnation.

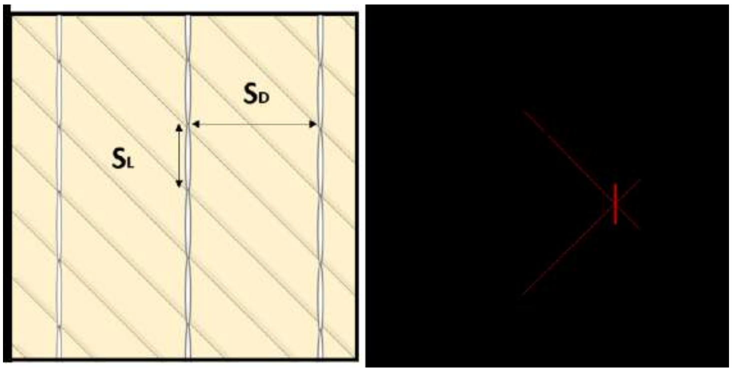


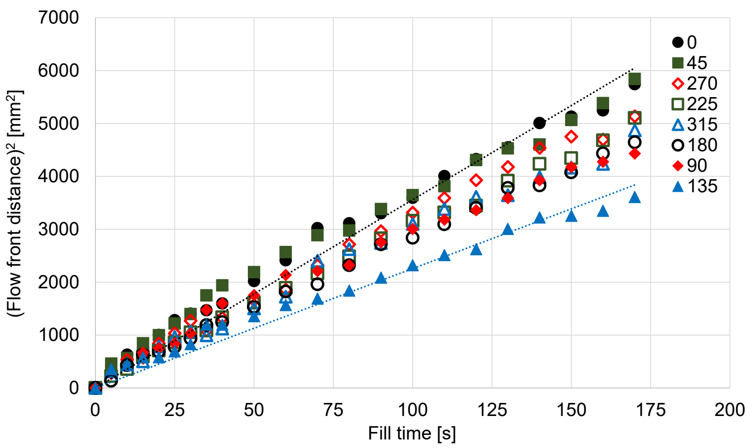
Figure 4 - Scheme of the NCF with stitching parameters: stitch length (SL) and stitch distance (SD) (a) and the roving orientation with the machine direction (b)

During in-plane measurements, the stich yarn of the NCFs (machine direction in fig.3) was aligned with the Y-axis sensor line (fig3).

- <u>Results:</u>

Unsaturated permeability

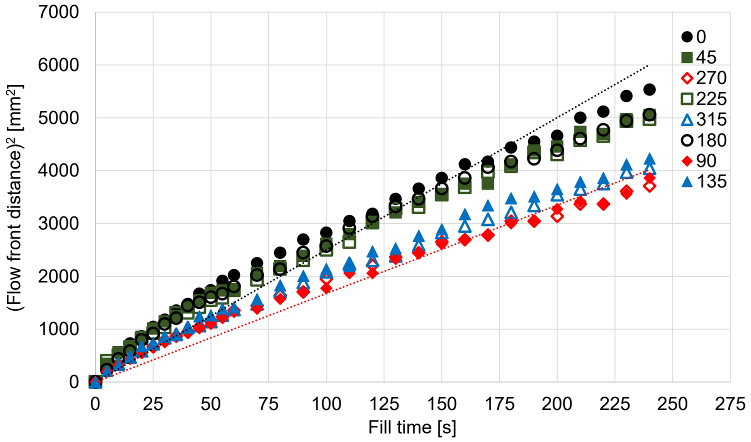
BXTV312



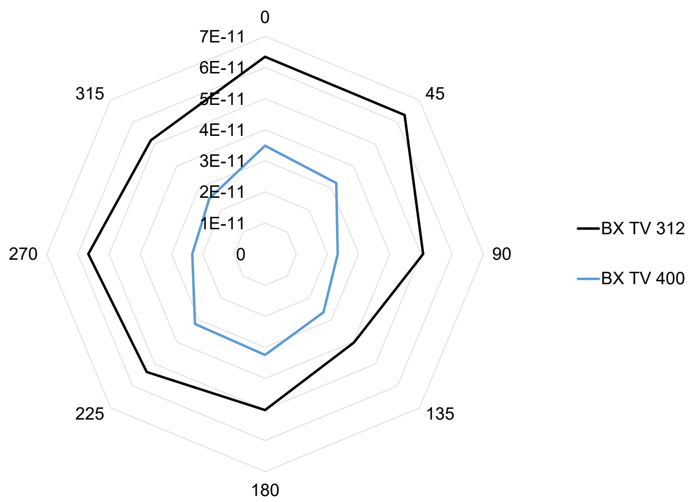
Orientation	Kunsat [m²]
0,180	5.68E-11
45,225	5.85E-11
90,270	5.37E-11
135,315	4.60E-11
average	5.37E-11
stdev	7.57E-12
cov	14%

BXTV424

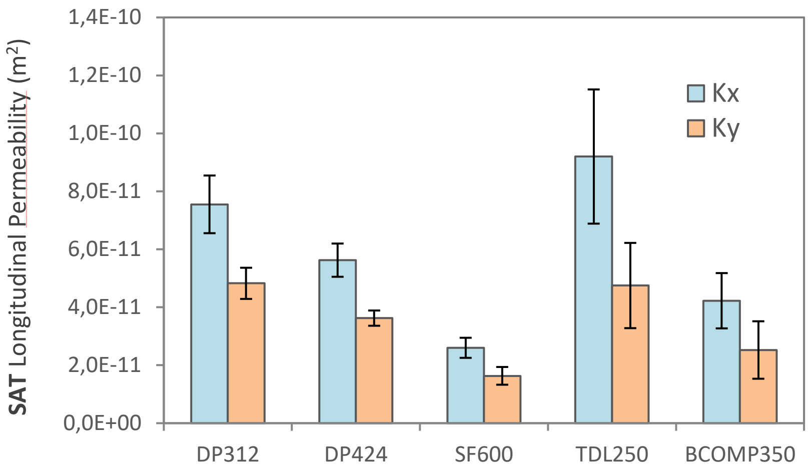
Orientation	Kunsat [m²]
0,180	3.36E-11
45,225	3.20E-11
90,270	2.33E-11
135,315	2.59E-11
average	2.87E-11
stdev	4.61E-12
cov	16%



Unsaturated in-plane permeability of the BX TV 312 and BX TV 424 flax reinforcements were obtained at various orientations. On average, for BX TV 312 fabric (at φ = 0.76) Kunsat = 5.37E-11 ± 7.57E-12 m^2, whereas for BX TV 424 (at φ = 0.66) Kunsat = 2.87E-11 ± 4.61E-12 m^2. The coefficient of variance describing the scatter in permeability values of the different orientations was 14-16%, similar to the effect of a 10-15% change in pressure or fibre content. Indeed, BX TV 312 has higher permeability, though this is in part due to the lower fibre content.

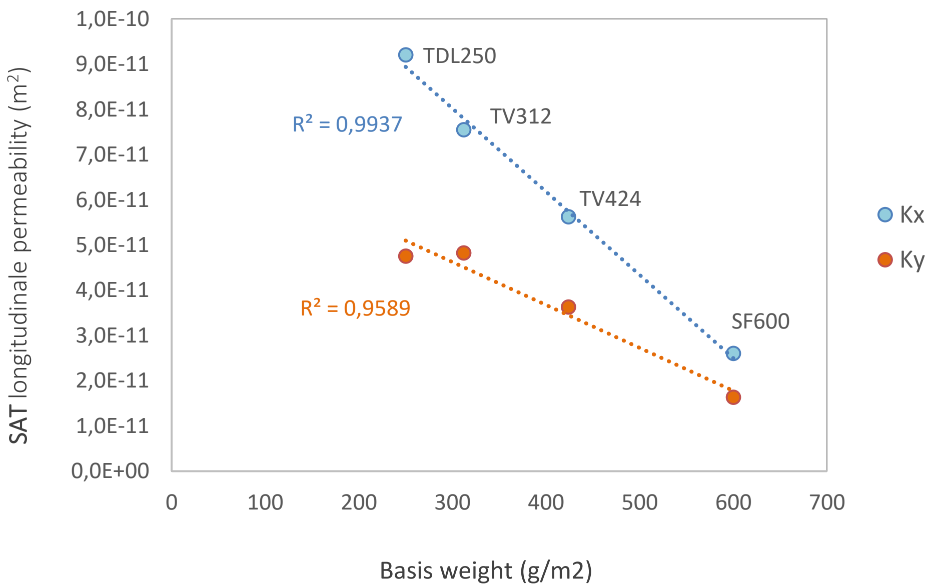


What was particularly notable from our measurements is the asymmetrical (elliptical) flow map, depicting permeability anisotropy. Specifically, permeability was higher in the 0/180 orientations (perpendicular to the stitch direction) than in the 90/270 orientation. This is also visualised in a spider map presenting the permeability of the reinforcements at the different orientations. This observation is also made in the saturated permeability measurements presented below.

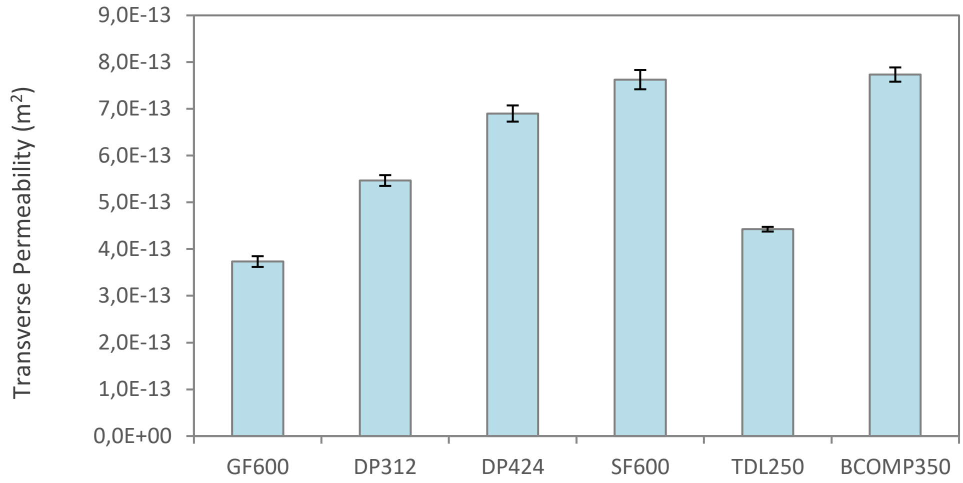


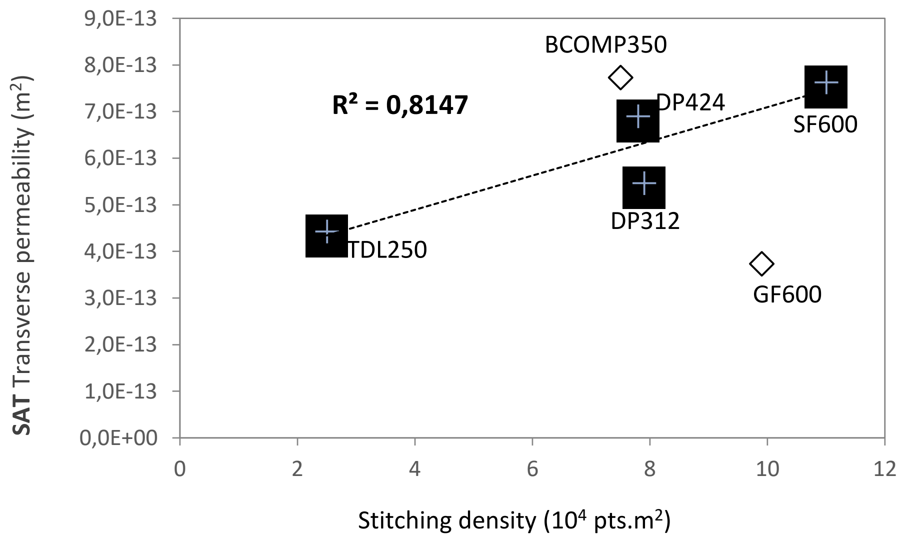
The permeability of the GF600: K_X=2,7.10^{-10} and k_Y=4,1.10^{-11} which is not plot on the graph above is higher by almost one order of magnitude. According to the literature, the glass NCF permeability along the stitch yarn is higher than the perpendicular one. As discussed above, the opposite behaviour is observed for all flax NCFs assuming that capillary and swelling effects are involved

We have tried to correlate these variations with various parameters such as stitching length, distance, density, fibre individualization or misalignment but the one which make more sense is the areal weight of the fabric, especially if we are regarding to the Depestele fabrics with similar stitching parameters.



Regarding to the transverse measurement the Standard deviation is much lower than the in-plane and we highlighted that the transverse permeability is less affected by the compaction phenomena but mainly driven by the stitching density of the NCF which act as resin pathways.





Conclusion

The in-plane permeability was measured using pressure variations and flow front tracking with time. We have found that both methods are reliable and, above all, comparable. The table below display the permeability of the Depestele fabrics:

		Permeability (10^{-11} m^2)	StDEV (10^{-12} m^2)
BX 312	Unsaturated	5.37	7.57
	Saturated	6.2	7.65

BX 424	Unsaturated	2.87	4.61
	Saturated	4.6	4.15

The transverse permeability, lower by two order of magnitude, was only assessed by the saturated method:

		Permeability (10^{-13} m^2)	StDEV (10^{-14} m^2)
BX 312	Saturated	5.5	1.2
BX 424	Saturated	6.9	1.7

Regarding to commercially available references of biaxial flax fabrics, Depestele products despite good permeability properties. However, in order to improve these properties many parameters, acting simultaneously, could be modified such as the stitching length, the areal weight, the stitching density or the fibre individualisation.