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► **To cite this version:**

Pierre Ouagne, Joel Breard. Continuous transverse permeability of fibrous media.. Composites Part A: Applied Science and Manufacturing, 2010, 41, pp.22-28. hal-00655500

HAL Id: hal-00655500

<https://hal.science/hal-00655500>

Submitted on 30 Dec 2011

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Continuous transverse permeability of fibrous media

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Abstract: The compaction of composite preforms and the flow of resin through the fibrous network take place simultaneously during the Resin Film Infusion process. Therefore there is a coupled loading of the porous reinforcements. A new experimental device, set up to impose combinations of hydraulic and mechanical loadings (Hydro-Mechanical loadings) to fibrous preforms is used to evaluate the transverse permeability in a continuous manner (under Hydro-Mechanical conditions) for a flax mat, a flax non crimped fabric, a carbon plain weave and a glass satin weave. For a 0.5 mm/min compression speed the continuous technique gives values similar to the “classical” technique for the four composite reinforcements of very different nature. This suggests that it is possible to use the continuous technique to evaluate the transverse permeability behaviour of fibrous reinforcements (permeability vs fibre volume) with a time reduction of about 8-10. Increasing the compression speed gives slowly decreasing continuous transverse permeability values.

Keywords: E: Resin film infiltration; A: Fabrics/textile; E: Resin flow; Continuous transverse permeability.

1. Introduction

The need for manufacturing large composite parts in the field of the aeronautic industry is increasingly important. Processes of the Liquid Composite Molding (LCM) class are widely used. These processes such as Resin Transfer Molding (RTM) involve the injection of a liquid thermosetting resin through some layers of dry and shaped preform. However, these processes are not well adapted in the case of large part manufacturing because of large tooling cost. Some void formation may also appear when the resin travels large distances as its viscosity may increase before the part is completely filled as a result of partial curing [1]. The infusion processes such as Resin Film Infusion RFI [2] consists of transmitting a stress with a vacuum bag on a stack of semi-cured liquid resin film and dry preforms (Figure 1). The whole set up is generally placed in an autoclave to ensure the correct compression stress and to control the temperature cycle. The resin flows through the preform in the direction of the applied stress. The reinforcement compressibility and the resin flow occur simultaneously and there is thus a mutual influence between the two “solid and liquid” phases. A strong coupling between the reinforcement compression and the resin flow takes place and needs to be taken into account for the modelling of the process.

Studies dealing with the modelling of resin infiltration in deformable preforms under different conditions have been published [3-5]. The fabric is supposed to be uniformly deformed in the direction of applied stress. In processes such as RFI, the principal resin flow and fabric deformation occur in the same direction. The resin pressure and the fabric compaction stress are not uniform within the thickness direction. The differential pressure and compaction stresses in the transverse direction are taken into account in the modelling of the RFI process proposed by Ouahbi *et al.* [6] and by Drapier *et al.* [7].

Trochu *et al.* [8] also used that approach to simulate the impregnation by resin of dry reinforcement during a new LCM process. Terzaghi's Law is used to couple the resin pressure and the mechanical stress imposed to the preform.

The previously mentioned models are based on experimentally determined material properties such as the permeability of the fibrous reinforcements. The permeability behaviour of the preforms and especially the transverse permeability evaluated in the transverse direction is a key entry parameter for the modelling of the process. As a consequence, a good modelling of the process depends on the accuracy of the permeability measurements. This subject was treated by numerous authors in the planar directions [9-11]. Several studies deal with the subject of through thickness permeability [1, 12, 13].

The permeability measurement for a single fibrous reinforcement requires a large amount of time as the permeability may often be evaluated in the three directions for various fibre volume fraction. Several days may be required to evaluate the permeability tensor of a single reinforcement. Buntain and Bickerton [14] developed a method to measure the in plane permeability as a function of the fibre volume fraction of glass mats in a single experiment. It consists of evaluating continuously the in-plane permeability while compressing the fibrous reinforcements with a given speed. They called it the "continuous technique".

This work deals with the measurement of the through-thickness permeability by using a continuous method. Investigations were carried out to evaluate the effect of the continuous compaction upon the transverse permeability. As such, a comparison between the continuous technique and the classical time consuming method consisting

of obtaining pressure/flow rate couples for a given fibre volume fraction is carried out on four different fibrous reinforcements.

An original experimental device was set up at Le Havre University to apply Hydro-Mechanical loads to fibrous media. Different combinations of fluid flow rates and mechanical stresses can be imposed to fibrous preforms.

2 Permeability measurements

The permeability describes the ability of a porous material to be impregnated by a fluid. The permeability tensor is used in Darcy's law [15] to relate the flow rate to the pressure gradient. Darcy's assumptions suppose that the flow of fluid across the porous medium is fully saturated. It applies to the flow of fluid in the wetted preform. This is the case for our study as saturated flows are concerned in the measurement of the continuous and the classical permeability. In these conditions, the saturated permeability is measured. However, during the RFI process, most reinforcements and especially the woven fabrics made of large bundles display a region right behind the flow front where a progressive saturation (air bubbles capture and migration) takes place. Depending on the flow velocity and on the viscous forces induced, air bubbles can be entrapped inside the tows or between the tows. The pressure field associated to a saturated flow is different from the one associated to a saturating flow. As a consequence, the permeability values measured in transient regime by using Darcy's law are different than the ones evaluated with saturated flows. Drapier *et al.* [16] compared the values of through-thickness transient and saturated permeability for carbon non-crimped fabrics. They observed that the transient permeability values are lower than the saturated ones. A ratio of eight was observed. In their experiment, they

used the classical method to evaluate both saturated and transient permeability. Optical fibres were inserted within the material to measure the advance of the fluid flow through the thickness of the fibrous network.

During the continuous permeability measurement, the fibrous reinforcements are compressed at constant speed. Therefore intrusive device such as optical fibres to measure the flow advance within the material cannot be used. As a consequence, this work concentrates on the continuous evaluation of the saturated permeability. Moreover, Darcy's law should not be used in the classical form to evaluate transient permeability as the fibrous reinforcements are not fully saturated. Numerous models were elaborated to relate the transient and the saturated permeability [17-18]. A model was also proposed to take into account the effect of the saturation during the filling of dry reinforcements in LCM processes [19]. Apart from the first stages of the filling of dry reinforcements by the resin, the majority of the wetted preform is saturated. This therefore means that the saturated permeability measurements remain essential as the pressure field within the vast majority of the wetted preform is governed by these parameters [14].

The saturated transverse permeability of fibrous composite reinforcements has been evaluated classically by several authors [1,12,13]. This method consists of evaluating pressure/flow rate couples at a given fibre volume fraction after injecting a test fluid through the fibrous preforms. This "classical" technique consumes a large amount of time as a set of pressure/flow rate couples has to be determined for every fibre volume fraction of interest. Moreover, pressure stabilisation due to changes of imposed flow rate may also take time particularly for high fibre volume fraction preforms [20].

Scholz *et al.* [21] developed a continuous technique to measure the transverse permeability during compaction to reduce the experimental time. It consists in injecting a newtonian liquid or a gas through the preform that is compacted continuously. In their study of the debulking effect upon the continuous transverse permeability of fibrous reinforcements, they show that the continuous transverse permeability measured by injecting a gas or a test fluid give similar results. However, the possible difference between the value of the continuous and the “classical” permeability was not mentioned.

During the RFI process, a strong coupling takes place between the compaction of the preform and the flow of resin. During the process, the permeability of the fibrous medium varies simultaneously according to the evolution of preform compaction. The use of a continuous technique to measure transverse permeability would reflect better the coupling phenomenon between the flow of fluid and the compaction of the preform that takes place during the process. As a consequence the measured values of the classical transverse permeability may not be similar to the values obtained using the continuous technique as fibre rearrangement [leading to pressure and stress relaxation](#) takes place after each compaction step.

3 Experimental procedures

3.1 Experimental device and permeability measurement methods

A device was set up at Le Havre to establish simultaneous hydraulic and mechanical loads (Hydro-Mechanical coupling loads) to the fibrous preforms (Figure 2). This apparatus consists of a stainless steel cylindrical pot within which a guided piston induces the exact amount of fibres compaction in the transverse direction. The device is mounted on a universal testing machine (Instron 8802) to control the displacement speed of the piston or the stress applied to the fibrous medium. The fluid is guided in the transverse direction by perforated bronze grids. A pressure transducer is placed below the lower grid. Test samples of fibrous preforms are placed between the two grids. A six litre syringe is placed on an universal testing machine (Instron 5867) in order to apply a controlled flow rate of silicon oil to the fibrous reinforcement. The magnitude of the flow rate is controlled by the Instron 5867 crosshead speed. When a newtonian test fluid such as silicon oil is injected at a constant flow rate through the fibrous reinforcement, a pressure rise at the reinforcement entry is measured by the pressure sensor.

The same apparatus is used to measure the continuous and the “classical” through-thickness permeability. For each set of experiment, new stacks of reinforcement are inserted in the device. The number of reinforcement layers placed in the device cavity depends on the individual thickness of each preform. An initial cavity thickness of about ten millimetres is generally considered. The transverse permeability is calculated for both methods by using Darcy’s law as saturated flows take place during the measurement. Since the piston compresses the fibrous medium at a constant speed, the

transverse permeability is measured in a continuous manner as a function of the increasing fibre volume fraction.

In the classical method, step by step increasing flow rates are applied to given fibre volume fractions and the rise of pressure due to the flow of fluid across the preform is measured (Figure 3a). The classical transverse permeability is calculated by using the slope of the flow rate vs pressure curve (Figure 3b) and Darcy's law.

3.2 Setting up of the device

A preliminary study to quantify the pressure rise due to the fluid flow going through the bronze grid was carried out for different flow rates. The rise of pressure due to the grid for each flow rate is then deducted when Hydro-Mechanical loads are imposed to fibrous preforms. A silicon joint is used to avoid race tracking along the walls of the cylinder. This joint is placed at the periphery of the lower grid in a zone that does not contain any holes. As a consequence, there is no section reduction that could affect the quality of the permeability measurement. The silicon joint is applied wet directly on the side of the grid. The necessary drying period is relatively short (20 min at the surface) as the joint does not need to be completely dry. The efficiency of this joint is confirmed by the linearity of the flow rate vs pressure curve (Figure 3b). Indeed, if race tracking was taking place along the cylinder walls, the rise of pressure would not be proportional anymore to the imposed flow rate. In this case the pressure rise would be “slower” than the rise of flow rate.

3.3 Materials and experimental conditions

The materials used during this study are:

- an E glass 5 harness satin weave. This material has an areal weight of 620 g/m²
- a carbon plain weave that has an areal weight of: 625 g/m²
- a flax mat that has an areal weight of: 116 g/m²
- a flax non-crimped fabric that has an areal weight of: 500 g/m²

The fluid used in the experimental procedure is a silicon oil of 0.1 Pa.s viscosity. The size of the preforms is adjusted to the size of the cylindrical pot and a silicon joint is used to avoid preferential oil propagation on the edge of the samples. The different layers of fabric are superimposed and the yarns of each layer are disposed parallel to the corresponding yarns of the neighbouring fabric layer. For the glass satin weave, twenty layers of 100 mm diameter discs are disposed for each test. A same stack of sample is used to characterise the classical permeability on the whole range of fibre volume fraction from the lower to the higher values. A data point is obtained for each fibre volume fraction. For each fabric, the experiment is conducted three times on different stacks of samples as these ones cannot be used once compressed at high fibre volume fractions. Three data points are therefore available for each considered fibre volume fraction. For the continuous technique, an imposed compaction velocity of 0.5 mm/min is applied to the reinforcements. A constant flow rate ($6.7 \cdot 10^{-7}$ m³/s) is applied to the preform by keeping the syringe speed constant. The continuous results presented in this study are coming from a single set of measurement for each fabric. This work therefore shows the preliminary results of the continuous permeability for three of the four materials considered. The influence of compression speed upon the value of the continuous transverse permeability was only studied within the range 0.5 to 5 mm/min

for the glass satin weave. Before each continuous transverse permeability measurement, a new stack of reinforcement samples is inserted in the device. The same stack of reinforcement cannot be used to evaluate the classical transverse permeability and the continuous one.

4. Results and discussion

4.1 Permeability results

Figure 4 shows that the pressure measured at the flax preform entry raises as a function of the fibre volume fraction. Since the flow rate is constant, the transverse permeability is deduced from the pressure data (Figure 4) and Darcy's law. Figure 5 shows the evolution of the transverse permeability of flax mat as a function of the fibre volume fraction. The increase of pressure and decrease of permeability are related to the reduction of the pores volume between the fibres. Figure 5 shows a comparison between the transverse permeability values of flax mats determined by the classical method and the continuous technique. For both curves, a fast decrease in permeability is observed at low fibre volume fraction. For higher fibre volume fraction, the decrease in permeability slows down progressively. Figure 5 also indicates that the transverse permeability results determined using the "classical" method and the continuous technique give data points situated in the same range of values.

The transverse permeability behaviour of very different composite reinforcement materials (a flax non-crimped fabric, a carbon plain and a glass satin weave) were also investigated (Figure 6a, 6b and 7). The transverse permeability values of the three

materials differ by several order factors. However, for each individual reinforcement, the transverse permeability measured by the “classical” and the continuous technique give very similar results as already mentioned for flax mats. In the case of the flax non-crimped fabric, especially at low fibre volume fraction (0.2-0.3), the continuous and the classical results show a small difference. The classical transverse permeability values are slightly lower than the values observed for the continuous transverse permeability determined using a compression speed of 0.5 mm/min.

4.2 Error analysis; comparison of the two permeability measurement methods

In the measurement of properties such as the permeability, several sources of error exist and may be added. The relative error on parameters such as the fluid viscosity, the pressure is relatively low and depends on the accuracy of the used sensor. The error on the imposed flow rate depends on the accuracy of the syringe piston speed applied by the Instron 5867 machine.. Specimens are manipulated with care and the relative error on the sample area is low and limited to few percents (typically below 2 %).. The error on the cavity thickness where the samples are placed for the tests has been identified as the main source of error particularly at high fibre volume fraction. Indeed, very small displacements can increase significantly the fibre volume fraction for some reinforcements. As a consequence the relative error on the cavity thickness measured by the displacement of the machine crosshead may exceed values of 10%. Figure 8 shows the relative error on the classical transverse permeability for the four considered materials of this study as a function of the fibre volume fraction. For the four reinforcements, an increase of the relative error is observed with an increasing fibre volume fraction. Relative errors increase in the range 2-22%. It is not surprising to observe similar types of increase for the four materials as the initial cavity thicknesses

considered for the measurements are similar. The relative errors on the classical transverse permeability are well within an acceptable range as the accuracy of such a measurement depends on the accurate measurement of five parameters (fluid viscosity, pressure, cavity thickness, flow rate and area of the samples) and on the care of the operator to prevent damages and size changes of the test samples during the manipulations.

The relative difference between the classical and the continuous transverse permeability values can be evaluated for each type of reinforcement by using Equation 1:

$$\text{Relative difference} = 100 \left| \frac{K_{\text{continuous}} - K_{\text{classical}}}{K_{\text{classical}}} \right| \quad (1)$$

The mean value of the three classical permeability data points is considered in the previous formula. The relative difference between the two permeability methods was computed for each type of reinforcement and the results are presented in Figure 9 as a function of the fibre volume fraction. The relative difference results are situated in a range of values situated between 2 and 47%. For low fibre volume fractions (up to 0.35 values), an important scatter is observed for flax mats and flax non-crimped fabric. For flax mats the maximum relative difference is slightly lower than 20 %, whereas the maximum value for the flax non-crimped fabric is of about 47%. No real tendency is observed between the relative difference values and the increase of the fibre volume fraction. The upper values of the relative difference may be due to the heterogeneity that may sometimes be observed in natural fibre reinforcements. From fibre volume fractions of 0.4, the relative difference values raise from values lower than 10 % up to about 47 % (carbon plain weave). The error on the relative difference between the classical and the continuous permeability is the sum of the relative error on the classical

value and the relative error on the continuous value. If one considers that the maximum relative error on the classical measurement is of about 22 % (Figure 8), then it can be considered that the maximum relative error on the determination of the continuous permeability is of about 25%. In this case, the error on the continuous permeability measurement is very similar to the error on the classical measurement. As a consequence, the measurement of the continuous transverse permeability under a compression speed of 0.5 mm/min is as accurate as a classical method measurement.

Figure 7 shows a comparison of the transverse permeability values measured by the classical method and the continuous technique for a glass satin weave submitted to three different compression speeds and the same constant imposed flow rate. The continuous transverse permeability seems to decrease when rising compression speeds are used during the test. This is probably due to a modification of the pressure field within the reinforcements. In the case of classical transverse permeability measurement, and if one assumes uniform fibre volume fraction, the pressure field is linear as no compression is applied. The pressure decreases linearly between the lower grid and the upper grid which is at the atmospheric pressure. If a constant compression displacement is applied on a saturated medium without any imposed flow rate, a squeezing flow occurs and a different pressure field is expected in our device with a maximum value at the bottom of the sample close to the lower grid and the atmospheric pressure at the upper grid as that one is directly connected to the outlet pipe. If an injection flow rate and a mechanical compression are applied, the two previously mentioned pressure field interact. Saouab *et al.* [22] showed that the pressure field takes a parabolic form in the case of CRTM process due to a gradient of fibre volume fraction in the thickness of the sample. In our case of study, a gradient of fibre volume fraction should also occur and a curved pressure field is expected. The curvature of the pressure field depends on the

compression speed as the injection flow rate is constant. Under slow compression speed the curvature of the pressure field is low as fast pressure relaxation may take place even during the compression as a result of fibre rearrangement. In those conditions the pressure field should not be very different than the linear pressure field expected in the case of the classical measurement. The pressure recorded should be similar or slightly higher than for the classical case. Under fast compression speed the curvature of the pressure field is higher and the pressure recorded by the sensor higher as pressure relaxation is not expected to take place in this case.

The relative difference between the classical and the continuous transverse permeability evaluated for higher compression speeds has been plotted as a function of the fibre volume fraction in Figure 9 for the glass satin weave. At a fibre volume fraction of 56%, a deviation of about 10% of the relative difference is observed for the 1 mm/min in comparison to the value calculated for the 0.5 mm/min compression speed. A deviation of about 25% is observed for the 5 mm/min compression speed. However, the value of the relative difference between the two techniques remains inferior to 50% for the glass satin weave even for the 5 mm/min compression speed. The difference between the continuous and the classical transverse permeability measurement relative to the classical permeability value is therefore influenced by the fibre volume fraction increase (error in the cavity thickness measurement) and by the increase of the compression speed in the continuous method.

Our results for transverse permeability can be related to the results presented by Buntain and Bickerton [14] and Comas-Cardona *et al.* [22] for the measurement of continuous in-plane permeability. These authors also found out that their continuous planar

permeability results were in good agreement to their classical planar permeability results especially at low compression speed.

The results commented in the previous paragraphs show that the transverse permeability measured using the classical and the continuous techniques at low compression speed give very similar results. As a consequence, it confirms that it is possible to use either the “classical” or the continuous permeability results for the modelling of processes where transverse flows occur.

4.3 Application to the RFI process

During the RFI process, a strong coupling between the reinforcement compaction and the resin flow takes place. A constant stress is imposed and the resulting compaction speed of the reinforcement is not constant. Ouagne *et al* [23] showed that the expected compression speed of the glass satin weave fabric submitted to an applied transverse stress is situated in a range 12-0.15 mm/min. In the case of fast compression speed the permeability of the fibrous reinforcement is not similar to the one evaluated classically. This suggests that the continuous transverse permeability technique may be of a particular interest to investigate the evolution of the transverse permeability of reinforcement up to high compression speeds. This would certainly improve the accuracy of the permeability value in such conditions and therefore improve the modelling of the process.

The results also suggest that the time consuming classical method may be replaced by the faster continuous technique. A time reduction by a factor of 8-10 is estimated.

4.4 Current work

This work shows the preliminary results obtained on four reinforcement materials. Parametric studies are in progress to study the influence of high compression speeds on different fabrics. Various works on techniques to follow the flow front for transient permeability measurement as well as the understanding of the fibre rearrangement during relaxation are also currently in progress.

5. Conclusion

A device was developed at University of Le Havre to impose combinations of hydraulic and mechanical loads. It is used to investigate the transverse permeability determined by a continuous technique. This work was performed on four different fibrous reinforcements (a flax mat, a flax non-crimped fabric, a carbon plain weave and a glass satin weave). For a 0.5 mm/min compression speed the continuous technique gives similar values to the “classical” technique for the four composite reinforcements even though they are of very different nature. This is the case if fibre and bundle rearrangement has the time to occur within the reinforcements during the compression of the reinforcements. A rearrangement of the fibres and bundles in a more “stable” positions favours pressure relaxation, a reduction of the measured pressure and prevent a drop in permeability. This suggests that it is possible to use the continuous technique to evaluate the transverse permeability behaviour of composite reinforcements (permeability vs fibre volume) with a time reduction of about 8-10. Increasing the compression speed seems to give slowly decreasing continuous transverse permeability

values. Continuous permeability results evaluated under high compression speeds may be of interest as fast compression takes place during a process such as the RFI.

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Captions to figure

Figure 1: Schematic of the Resin Film Infusion process (RFI)

Figure 2: The continuous permeability device

Figure 3: Part a: Pressure changes measurements: classical method; Part b: Linearity of the flow-rate-pressure curve: classical method.

Figure 4: Pressure evolution during compression of flax mat preform submitted to a constant flow rate

Figure 5: Continuous and “classical” transverse permeability of flax mat

Figure 6: Continuous and “classical” transverse permeability: (a): flax non-crimped fabric; (b) carbon plain weave fabric.

Figure 7: Continuous and “classical” transverse permeability a glass satin weave: Influence of the compression speed on the continuous technique results.

Figure 8: Relative error evaluated on the classical transverse permeability values

Figure 9: relative error between the continuous and the classical transverse permeability method relative to the classical method.

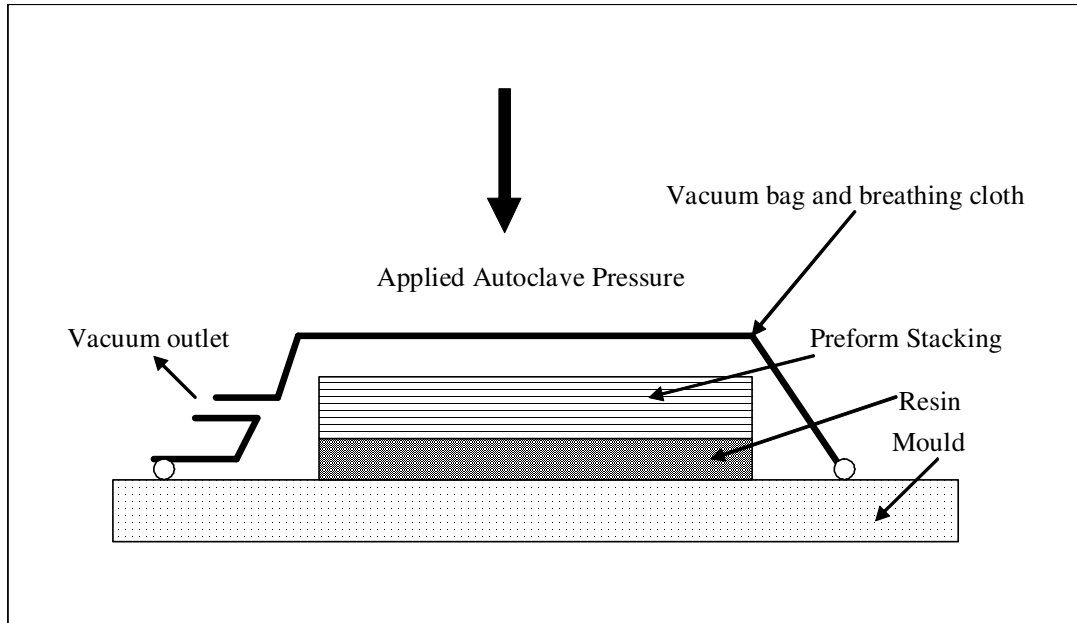
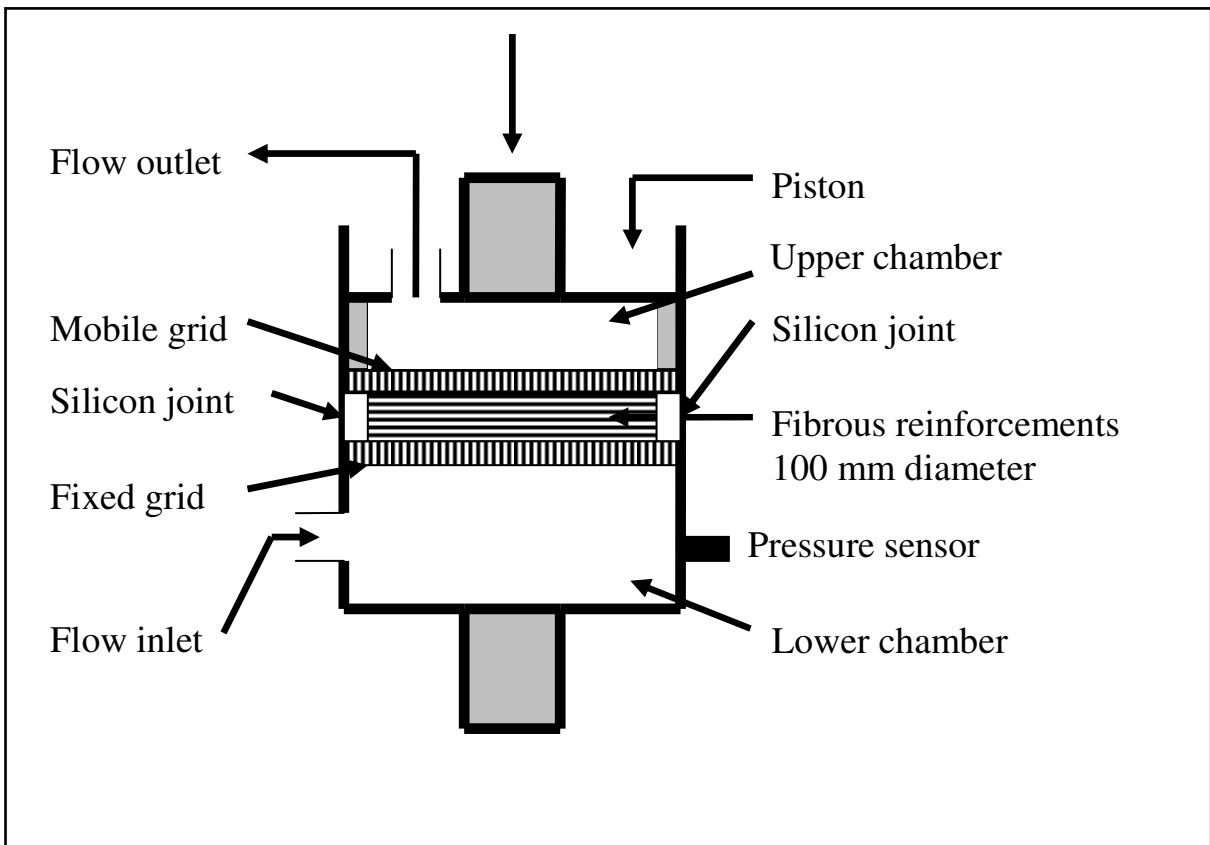


Figure 1: Schematic of the Resin Film Infusion process (RFI)



(a)

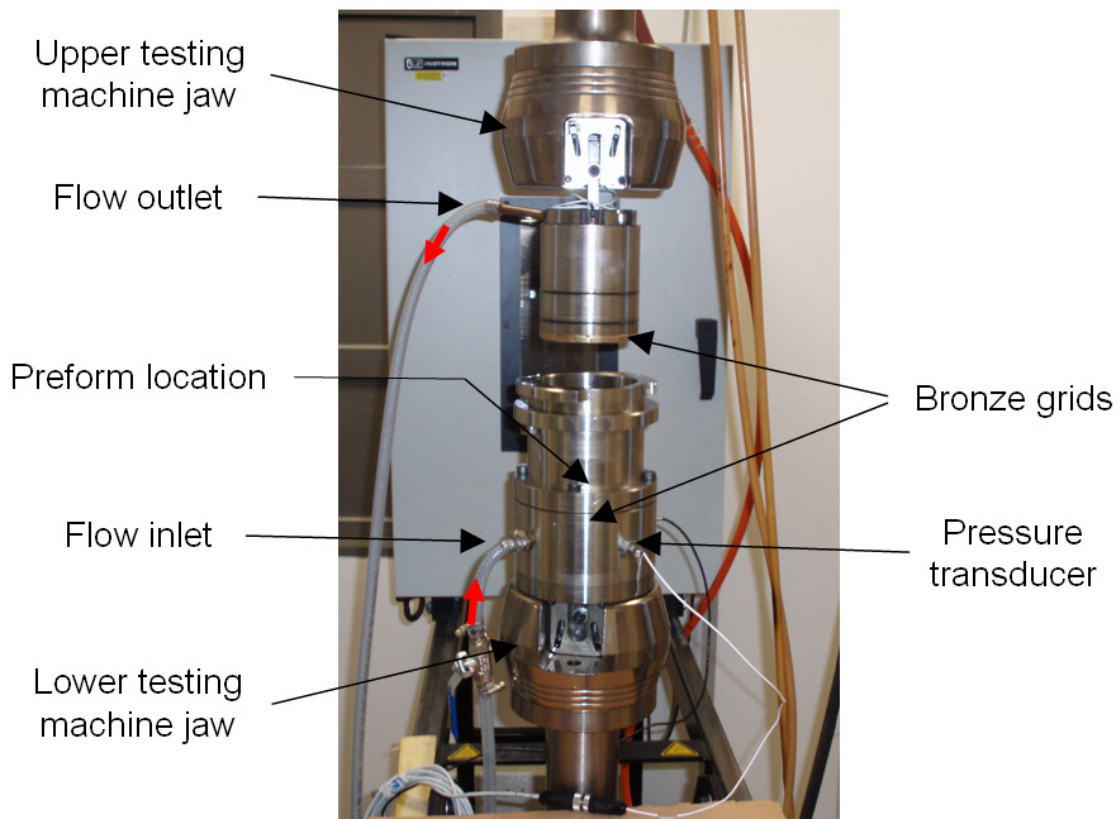
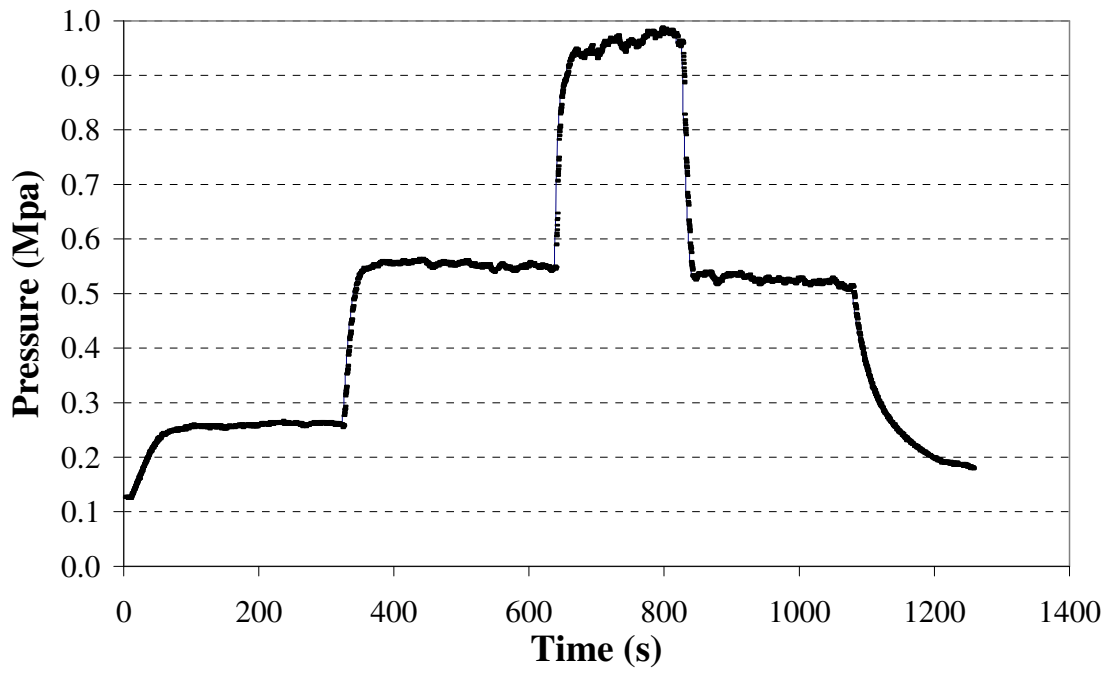
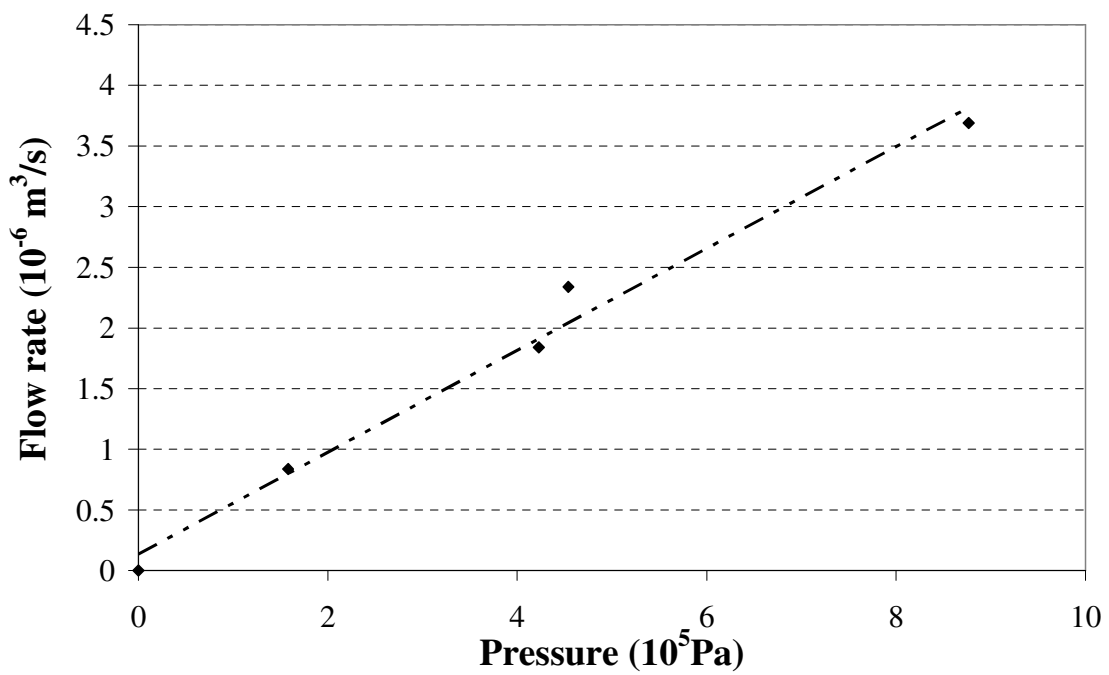


Figure 2: The continuous permeability device
(a) Schematic diagram. (b) Picture view



(a)



(b)

Figure 3: (a): Pressure changes measurements: classical method
 (b): Linearity of the flow-rate-pressure curve: classical method.

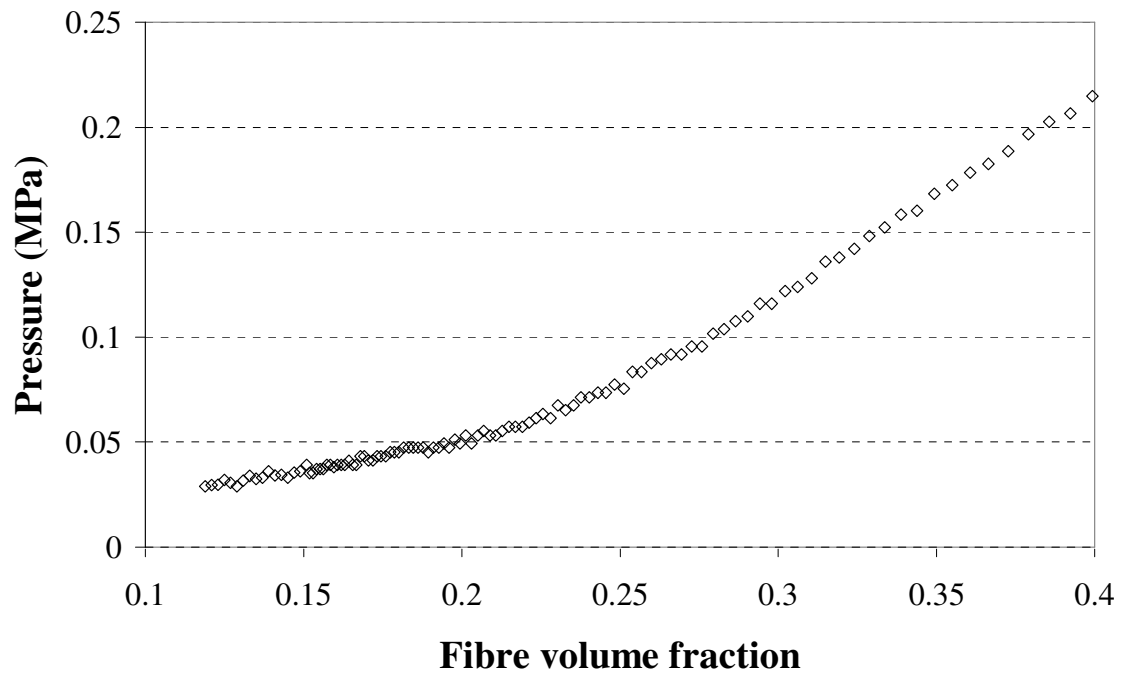


Figure 4: Pressure evolution during compression of flax mat preform submitted to a constant flow rate: continuous method.

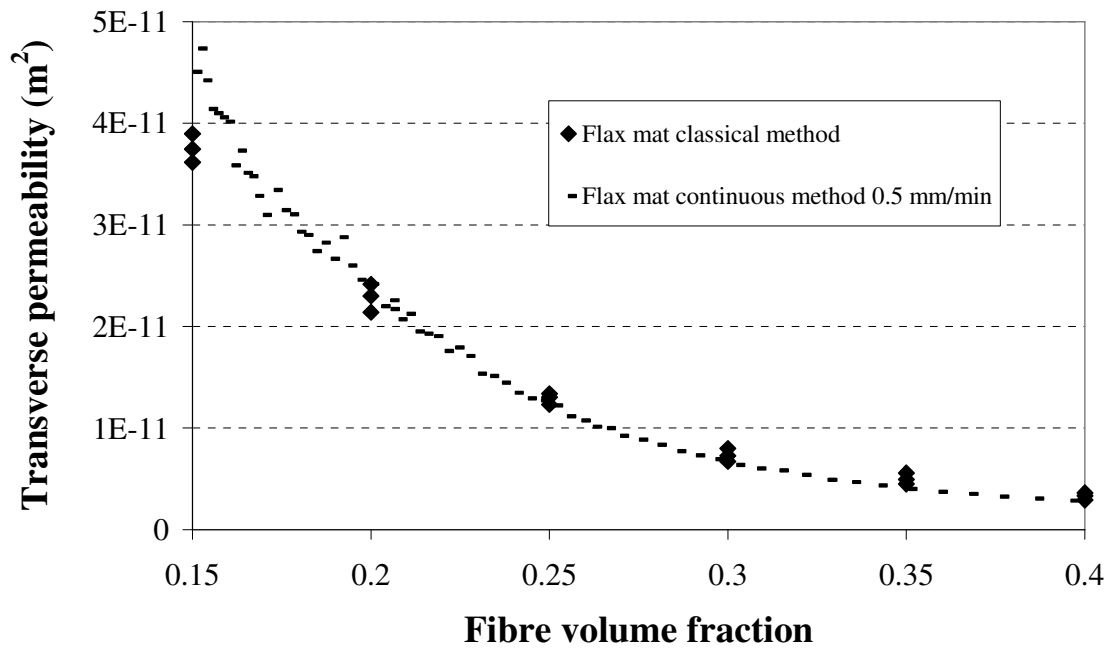
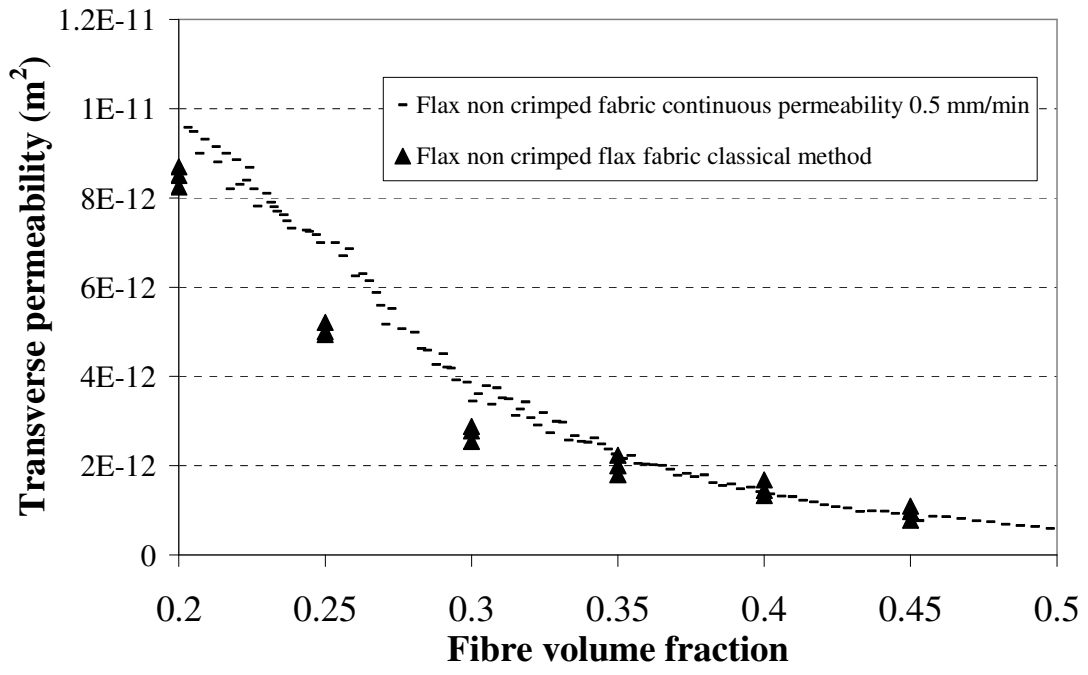
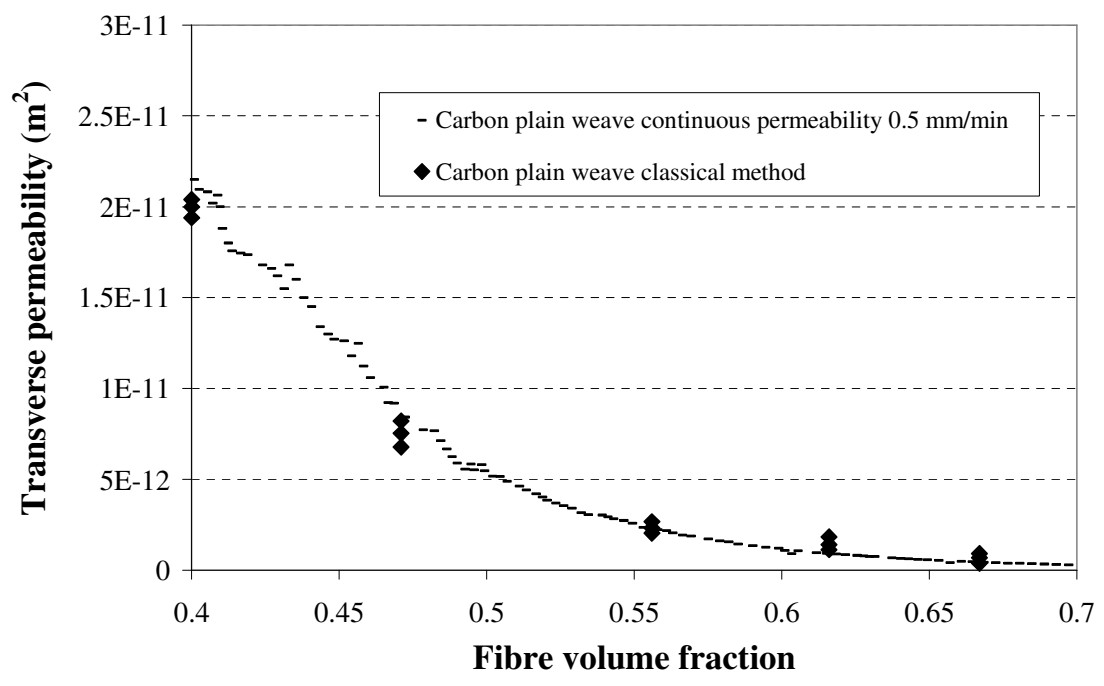


Figure 5: Continuous and “classical” transverse permeability of flax mat.



(a)



(b)

Figure 6: Continuous and “classical” transverse permeability: (a): flax non-crimped fabric; (b) carbon plain weave fabric.

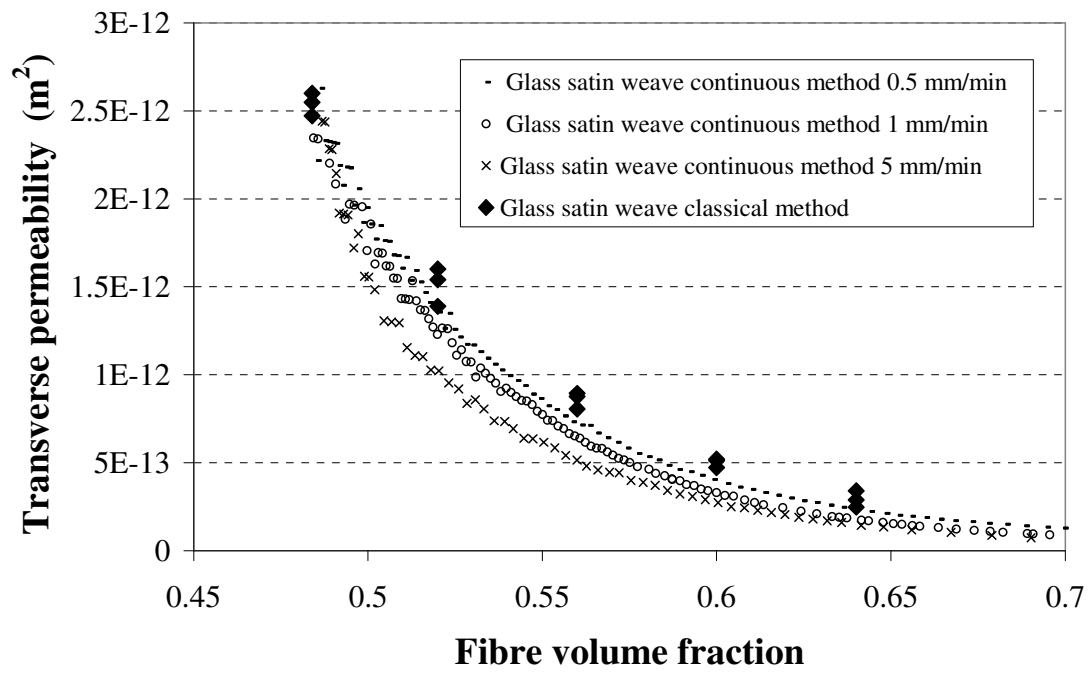


Figure 7: Continuous and “classical” transverse permeability a glass satin weave: Influence of the compression speed on the continuous technique results.

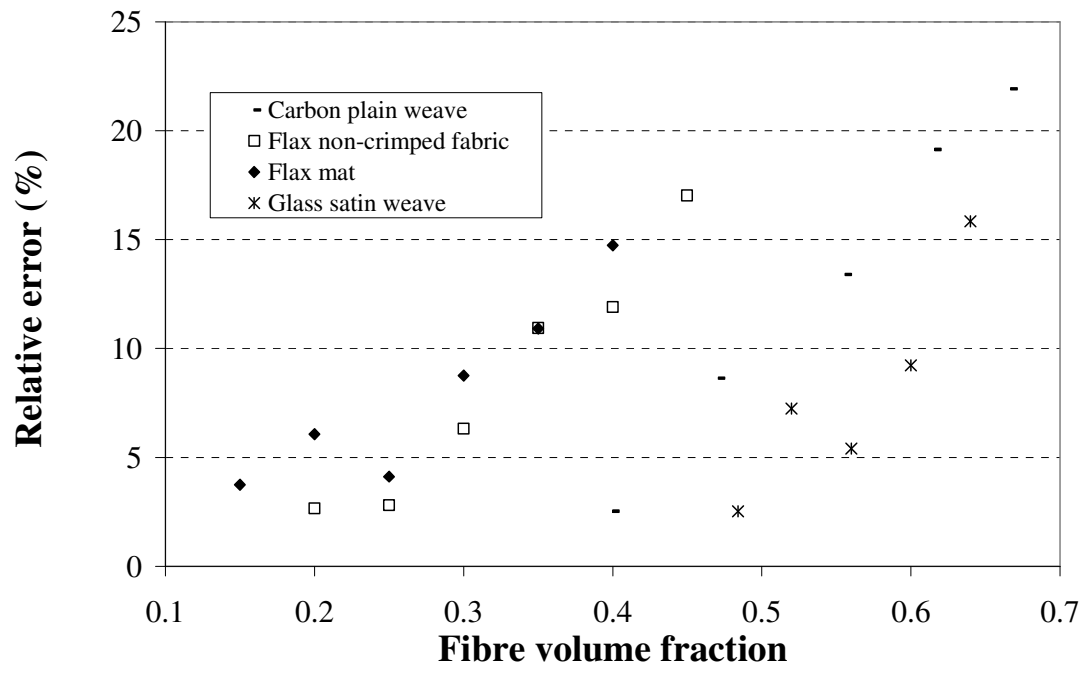


Figure 8: Relative error evaluated on the classical transverse permeability values

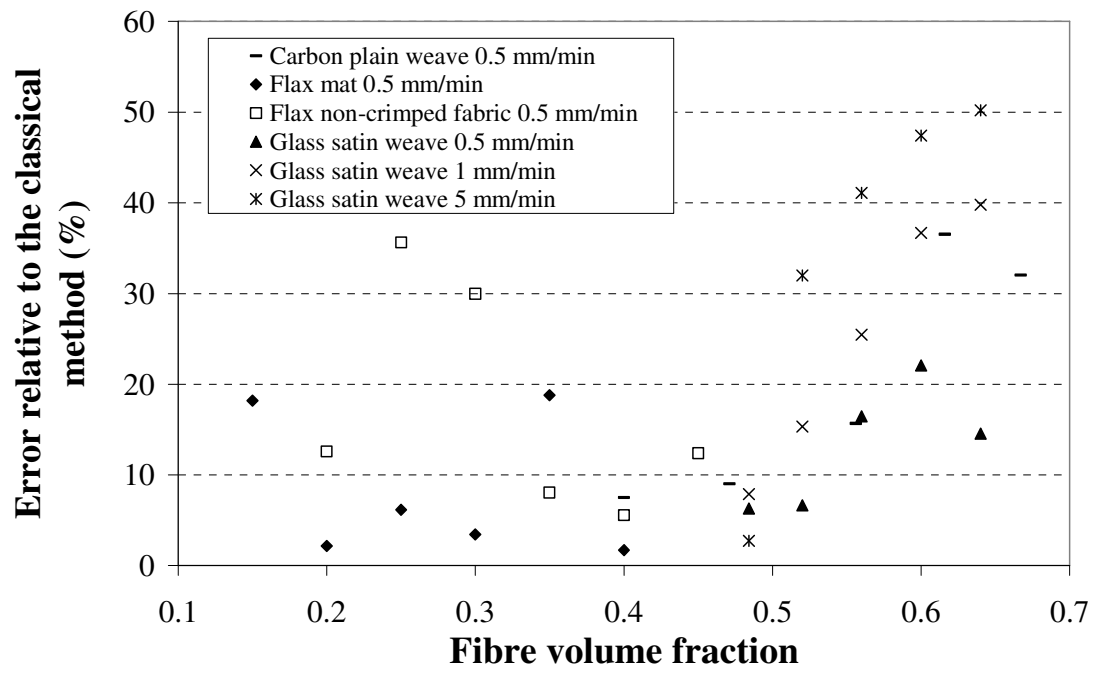


Figure 9: relative error between the continuous and the classical transverse permeability method relative to the classical method.