COMPARISON AND EVALUATION OF TWO DIFFERENT PERMEABILITY MEASUREMENT METHODS FOR FIBRE REINFORCED MATERIALS

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ABSTRACT: The permeability of fibre reinforcement materials is one of the main material input parameter for macroscopic flow simulation in Liquid Composite Moulding. In spite of the importance of this property there is no standardized measurement method. A previous permeability benchmark has shown a high dispersion between the measurements, even though all measurements are based on Darcy´s Law which describes the flow of a fluid through a porous medium. The study presented in the paper compares two different test setups with (i) 1-D resin flow and (ii) 2-D resin flow to point out the boundary conditions that have to be respected for a reliable and reproducible permeability measurement. The viscosity of the test fluid is a possible major source of error which is minimized by using a viscosity standard fluid for the measurements. Further boundary conditions are fibre volume content, temperature, textile lay-up, volume flow, and injection pressure respectively. This study describes difficulties and limitations of each type of measurement and wishes to contribute to a normalization process.

KEYWORDS: Permeability Measurement, Flow Simulation, Fibre Reinforcements, Liquid Composite Moulding

INTRODUCTION

Among various technologies for manufacturing composite materials, *Liquid Composite Moulding (LCM)* technologies, such as *Resin Transfer Moulding (RTM)* and *Modified Vacuum Infusion (MVI)*, have the capability of manufacturing polymer composites with large size and complex shapes at relatively low cost. In the *LCM* technique, a dry fibre textile is infused / infiltrated by a liquid resin.

The flow process of the resin system through the textile can be simulated by computer modelling to predict dry-spot formation, incomplete mould filling and curing. Flow simulation software, such as PAM-RTM, Polyworx, et al., therefore become very important tools in mould designing and manufacturing, process configuration, reducing manufacturing costs, and improving quality. The flow of a fluid through a porous medium is described by *Darcy's Law*. Permeability is a measure of the ability of a porous material to transmit fluids and therefore the permeability tensor of fibrous reinforcements is one of the main material input parameter for macroscopic flow simulation in Liquid Composite Moulding.
Even though the permeability tensor has importance for the simulation of flow processes in composite materials, there is no standardized measuring method. Hence, different setups / measurement devices have been developed especially for fibrous applications.
An international permeability benchmark test described in *"Experimental Determination of the Permeability of Textiles: a Benchmark Exercise"* [1] has been organized by ONERA and Katholieke Universiteit Leuven to quantify the existing "scattering" of permeability measurements between setups and to initiate a normalization process. The first results of this benchmark show that there is a high scatter, up to three orders of magnitude, between results from different labs. Therefore, the conclusions of this first step to calibration are limited and have to be validated in further tests.
The present study evaluates and compares two different measurement methods, i.e., different laboratories that have been involved in the international permeability benchmark:

(i) 1-D measurement (unidirectional flow) at Laboratoire de Technologie des Composites et Polymères (LTC), Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland
(ii) 2-D measurement (radial flow) by Deutsches Zentrum für Luft- und Raumfahrt (DLR), Customer Office Bremen, Germany

As many parameters as possible are the same in both tests in order to allow a comparison of these two setups. That includes the textile, lay-up (orientation and number of plies), fluid and test temperature (therefore fluid viscosity), and fibre volume content. All tests are performed in a closed mould, so only in-plane resin flow is considered. Even a possible "human factor" can be eliminated since all tests are performed by the same person.
The main differences of the investigated measurement setups, besides the respective flow direction, are the used methods for resin injection and the way permeability is determined. The 1-D measurement device at EPFL uses constant pressure to inject the resin while the 2-D device in Bremen works with constant volume flow. Hence, the measured parameters to the final permeability determination differ. This study describes difficulties, limitations, and possible sources of error for each type of measurement.

MATERIALS AND LAY-UP

The measurements at EPFL and DLR are performed with the same materials to create a maximum comparability. Since the measurements are carried out with a focus on aviation industry the chosen reinforcements are carbon fibre non-crimp fabrics (NCF) usually used for aeronautic applications. A $[0°,90°]_{2S}$ lay-up is built from biaxial NCFs

(±45° + inverse; HTS 12k, 268 g/m² per layer). In that way the stitching yarn has a "neutral" position (45° angle) for the 2-D measurement regarding x- and y-direction. Previous tests have shown that the sewing yarn increases permeability of the relevant direction. Figure 1 shows the lay-up used for all tests of this study.

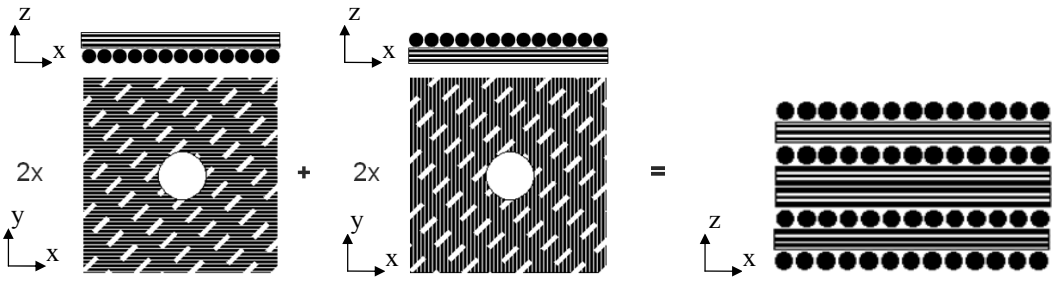

Fig. 1 Textile lay-up: 2x[90°,0°]+2x[0°,90°]=[90°,0°][90°,0°]¦[0°,90°][0°,90°]

Since the viscosity of the test fluid is considered as a possible major source of error, this influence is minimized by using a viscosity standard fluid for the measurements. The viscosity standard fluids manufactured by CANNON Instrument Company are usually used for calibration and verification of viscometers. Their liquid S2000 is chosen due to its calibrated viscosity value at high temperatures (66,25mPa·s at approximately 100 °C), which is similar to the value of epoxy resin systems at the same temperature used for aeronautic applications and shows similar permeability values (evaluated in [2]).

EXPERIMENTS

In order to solve Darcy´s law to extract the permeability values, several assumptions are made:

- The viscosity of the fluid remains constant through the experiment because the fluid used for the experiment is Newtonian and incompressible (verified in [2]), the tests are done at isothermal conditions and no cure takes place during the tests.
- The dry fabric is wetted by the advancing flow of the resin. The wetted domain is assumed to be fully saturated.
- The porous material has homogeneous interconnected porous space and it is rigid which means it does not deform or move during infiltration.
- The microscopic flow in the carbon bundle is not taken into account.
- Gravitational and surface tension effects are ignored [3]

1-D measurement at EPFL

The permeability device used at Laboratoire de Technologie des Composites et Polymères (LTC) at EPFL measures the one dimensional flow while the fluid is injected at constant applied pressure into a closed mould. The upper part of the mould is made of glass so the position of the flow front as a function of time *L(t)* can be detected visually. The methods to determine the permeability values are divided between the saturated and the unsaturated condition. For the unsaturated measurement the following equation is used (for derivation see [4]):

$$K_{uns} = -\frac{\psi^2.\mu\left(1 - V_f\right)}{2(P_g - P_{app} + \Delta P_\gamma)} \quad (1)$$

with

$$L^2 = \psi^2 t \quad (2)$$

Where ψ^2 is a kinetic parameter which represents the advance of the flow front during impregnation after a certain time, K_{uns} is the unsaturated permeability, P_g is the atmospheric pressure acting on the preform and P_{app} is a constant applied pressure. In order to have a first approach to the capillary pressure drop ΔP_γ, ψ^2 can be plotted as a function of the pressure difference $\Delta P = P_g - P_{app}$ for some experiments at different applied pressures. By Eq. (1) it can be seen that the relationship between these two parameters is linear, then by making $\psi^2 = 0$, ΔP_γ can be obtained by extrapolation. For the saturated permeability calculation, the following equation will be utilised:

$$K_{sat} = \frac{Q.\mu.\Delta L}{A.\Delta P} \quad (3)$$

Where Q is the volumetric flow rate [m^3/s], μ is the viscosity of the fluid [Pa.s], ΔL is the whole textile length [m], A is the mould cavity transverse area [m^2] and ΔP is the difference between the injection pressure and the atmospheric pressure [Pa]. Q is obtained after plotting volume vs. time (the fluid volume is measured at the outlet of the mould).

2-D measurement by DLR in Bremen

DLR uses a 2D setup with a central injection system for the in-plane permeability measurement. Pressure sensors are placed at the bottom of a closed mould whereas the position is defined only by the radius / distance from the centre. The direction of the sensors – they are placed on the x-axis, y-axis, and in a 45° angle – is not directly included in the calculations.
DLR processes its data with the 2D solution of Darcy´s law

$$K_e = \frac{Q\mu}{2\pi z} \cdot \frac{ln(r_2/r_1)}{(P_1 - P_2)} \quad (4)$$

with K_e the effective permeability between two sensors on one straight line, Q the constant volume flow, μ the viscosity, z the laminate thickness, r_1 and r_2 the radius, and P_1 and P_2 the measured pressures. Ten pressure sensors allow the calculation of thirteen different K_e values due to the arrangement of the sensors. The average of all K_e values gives the total effective permeability.
The ratio K_x/K_y is calculated with the time at which the sensors in the respective directions have reached a certain pressure level. These calculations are performed by the software "PPerm" by Pole de Plasturgie de l'Est (manufacturer of the permeability measurement device). Ratio and total effective permeability lead to K_x and K_y values.
A verification of reproducibility for the used 2-D measurement device was performed in [5]. The observed scatter was ±12% for the same textile used in the present study.

An overview of all relevant data for the comparison of the two permeability measurement devices from EPFL and DLR is shown in Table 1. Fig. 2 shows the linear one dimensional flow and the two dimensional radial flow.

Table 1 Overview of relevant data for comparison EPFL ⇔ DLR

	EPFL	**DLR**
Experimental Set-up	*1D*	*2D*
Type of infiltration	*Constant applied pressure*	*Constant volume flow*
Fibre bed geometry	*200 mm x 180 mm*	*600 mm x 600 mm*
Variables measured	*Average temperature T, flow front as function of time L(t), volume flow Q*	*Pressure P and temperature T (as function of time)*
Permeability equation (unsaturated test)	$K_{uns} = -\frac{\psi^2.\mu(1-V_f)}{2(P_g - P_{app} + \Delta P_\gamma)}$	$K_e = \frac{Q.\mu}{2\pi h(P_o - P_f)} ln\left(\frac{R_f}{R_o}\right)$
Permeability equation (saturated state)	$K_{sat} = \frac{Q.\mu.\Delta L}{A.\Delta P}$	
Maximum Pressures [bar]	*~4*	*~13*
Visual inspection of the flow front	*Yes*	*No*
Heating System	*Lower part of the mould*	*Upper and lower part of the mould*
Pressure sensors locations	*Inlet of the mould*	*On the lower mould*
Temperature range [°C]	*97 - 104*	*98 - 102*
Viscosity Range [mPa.s]	*63.73 - 77.21*	*64.98 - 68.70*
Capillary pressure	*Up to 1 bar*	*Negligible*

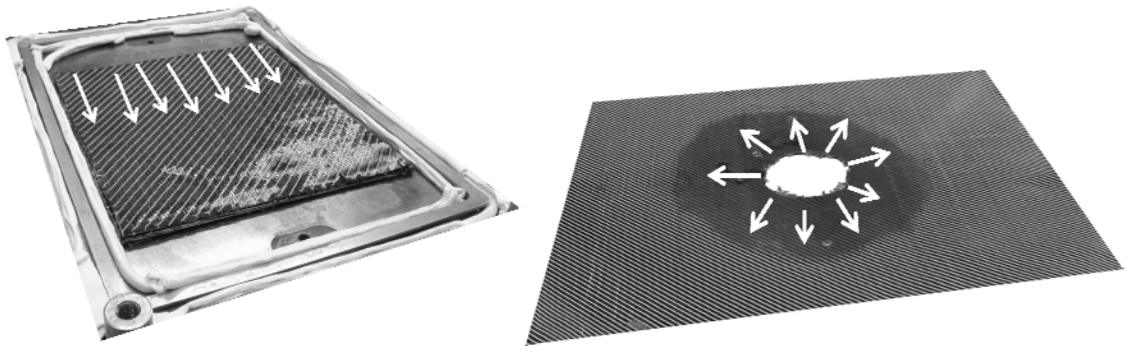

Fig 2 Linear 1-D flow at EPFL (left) and 2-D radial flow at DLR Bremen

Results

The results of unsaturated and saturated measurements in X and Y direction for different fibre volume contents (FVC) are shown in Fig. 3 (=> average values if more than one test has been performed). It is observed that the permeability values measured

at EPFL are higher than the DLR values. The shape of all curves which represents the textile behavior depending on the FVC is similar.
The permeability differences between Kx and Ky (~ factor 1.3) are comparable for both measurement devices. This is in accordance to the investigations done in [5] where Hasanovic showed that zero degree steps between two single layers hamper the resin flow *(here: two 0°/x direction layers in the middle of the lay-up)* while tool sided layers *(here: 90°/y direction layers)* show higher permeability values.
Compared to the permeability benchmark in [1] the scatter for the devices in Lausanne and Bremen is significantly lower. Whereas it has to be considered that the number of tests for this study was limited so that Fig. 3 does not / can not show the scatter for each measurement itself.

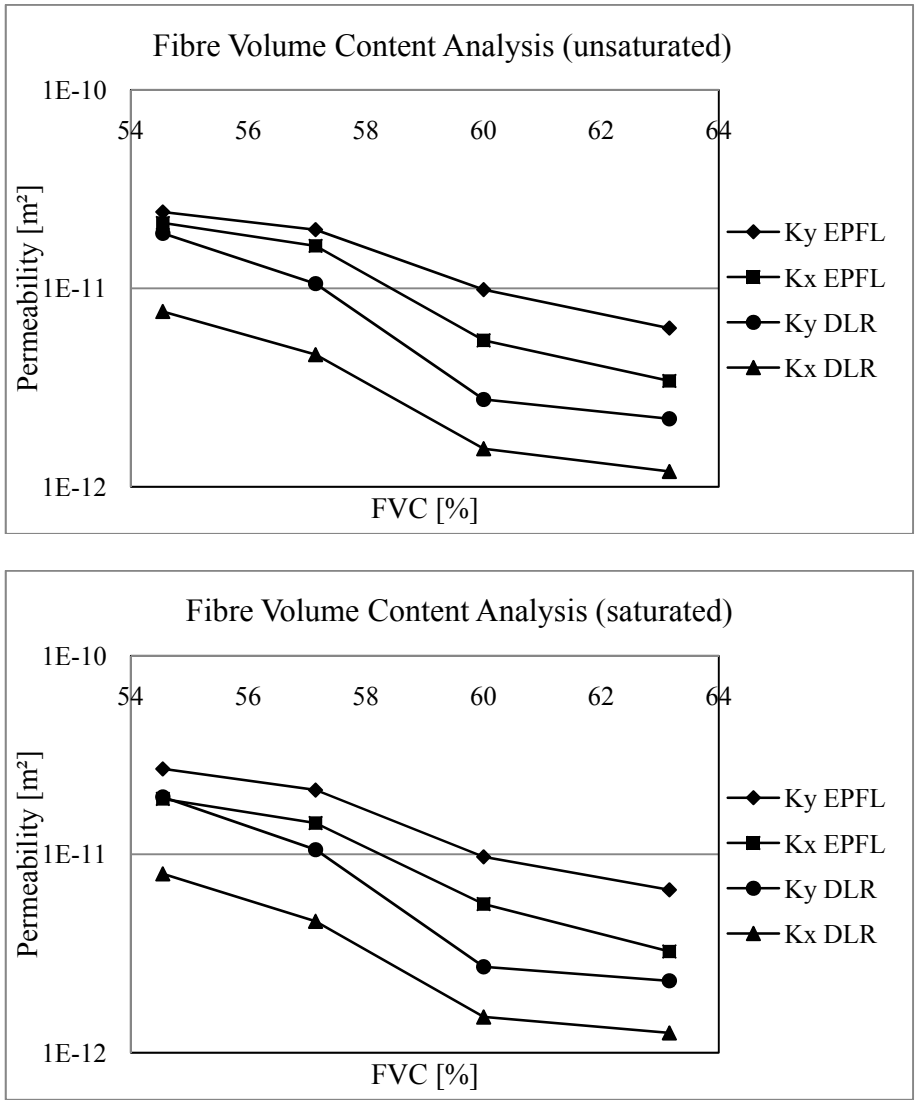

Fig 3 Measured permeability values (single or average values!)

Due to different pressure levels in both devices, ~4 bars at EPFL and ~13 bars at the device used by DLR, the possible influence of the pressure inside the mould was investigated in a further test series in Bremen. Experiments were performed using the same NCF at 60% FVC in a saturated state and varying flow rates, leading to varying pressures at the centre of the mould. The results of these experiments strongly suggest a linear relationship of permeability and pressure (Fig. 4). A deformation of the tool is not considered due to size, material, and closing pressure of the mould.

Since the permeability values rise with higher pressure, this phenomenon can not be seen as a reason for the differences between EPFL and DLR. On the contrary EPFL values had to be even higher for equal pressure levels since their injection pressures are lower.

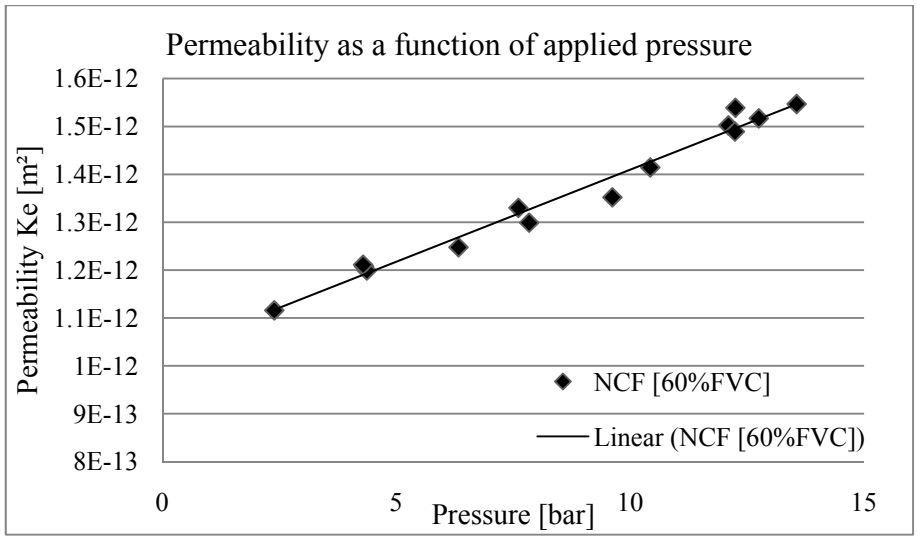

Fig 4 Permeability values of a NCF with 60% FVC for varying pressures

CONCLUSIONS

The present study shows that the scatter of permeability measurements can be minimized if all relevant boundary conditions are taken into account. Especially the usage of a correct viscosity value, and therefore the temperature control, is mandatory since viscosity has a linear influence on the measurement.
However there is still a high scatter compared to the measurement of other values (e.g. temperature, voltage, time, etc.). On the one hand this is caused by the inhomogeneous behaviour of the textile itself, so a scatter of ±12% as observed in [5] seems logical and unavoidable. Even for the same textile batch with the same lay-up a different permeability for different samples has to be expected.
On the other hand the measurement devices have to be optimized in hardware (e.g. measurement technology), software, and handling by the user. The practical experience with both used devices showed that especially the manual analysis, i.e. the human factor is a possible source of error. For many measurements errors have been observed on the second view. The work instructions have to be defined in more detail and the analysis has to be automated to avoid those mistakes.

A question that is raised is the validity of the assumption of rigid porous medium when solving Darcy´s law. The observed relationship between permeability and pressure can not be explained if the assumptions of both a rigid, non-deforming fabric and an incompressible fluid are maintained. As the difference in permeability is quite distinct, even for low pressure differences, fluid compression alone is insufficient to explain it. Thus, a possible explanation is that the assumption of a rigid porous material is not applicable for flow in fibrous media. Instead, the fluid flowing through the media alters the porous material, i.e. by widening bottlenecks in the pore network. With a rising FVC, this effect would become more and more negligible, as the share of inter-yarn

flow in the permeability drops. To support this theory further tests have to be performed.
In general it is recommended to use pressures that actually appear in LCM processes for the measurement of permeability values used as input data for resin flow simulations.

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