ONE STEP TO COMPARABLE OFF-PLANE PERMEABILITY RESULTS – AN IMPROVED MEASURING METHOD FOR TRANSVERSE PERMEABILITY AND COMPACTION BEHAVIOR OF TEXTILE FIBER MATERIALS

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Abstract

For the conventional transversal permeability test equipment, the preform cavity is limited by distribution structures for the test fluid (usually metal plates with an equidistant distribution of holes). These structures are used to provide a uniform fluid flow throughout the preform. The distance in between the structures defines the fibre volume content (FVC) of the preform under the assumption that the preform is homogenously compacted. In this study the influence of the distribution structures on the homogeneity of the fibre volume content is investigated. For this purpose, an experimental device was developed that allows the preform to be cured at identical conditions compared to the permeability test equipment. FVC measurements were made to examine the assumption. First test series confirm that the distribution structures indeed have an important impact on the results. This effect can be an explanation for the inconsistency of test results for off-plane permeability when different equipment (especially different distribution structures) is used.

1 Introduction

Permeability can be described as the ability of a fluid to penetrate a porous medium. It is one of the most influencing parameters during the impregnation process of textile fibre materials. Physically the permeability is a material parameter. However, geometric properties such as stacking sequence, compaction of textile materials, distribution of binder material and others significantly influence the permeability as well [1–4]. The characterisation of the permeability is done by a symmetric positive definite 2nd order tensor that can be diagonalized. The principal values K_{11} and K_{22} represent the in-plane permeability whereas the K_{33} characterises the off-plane permeability.

Especially for the impregnation of complex composite parts a simulation of the matrix flow, based on an accurate permeability tensor is needed. In order to quantify the permeability and to get a basic understanding of the various effects, different experimental measurement methods -saturated and unsaturated methods for in-plane and off-plane permeability values- have been developed. Nevertheless, results significantly differ between the various test methods. Thus the correlation between composite properties, the used measuring method and observed permeability has to be analysed in more detail.

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Detailed experimental methods to determine the **in-plane permeability** are summarized and rated in terms of required testing time and material usage in [5]. Several benchmark studies with the participation of numerous research institutes were carried out to investigate the dependencies of test results, equipment, user influence and evaluation method. It was shown that without any limitations to the procedures of the test, in terms of FVC, sample size, etc. the results scatter about one order of magnitude and the human factor was assumed to be the most influencing factor. A detailed procedure handbook and the repetition of the tests provided comparable results independent to the user [6–8].
For the **off-plane permeability**, measuring methods are commonly based on a saturated flow in a direction transverse to the fibre textile material (conventional method). The dry fibrous material (with a known areal density) is compacted in a cavity consisting of two distribution structures (usually metal plates with an equidistant distribution of holes). In the test, homogeneous fibre volume content across the material is assumed. Subsequently, a test fluid is injected with a predefined differential pressure until a constant fluid mass flow is observed. Off-plane permeability is finally calculated by logging pressure values and mass flow, and given density and viscosity of the fluid. Based on Darcys empirical law [9] the off-plane permeability (K_{33}) can be determined as:

$$K_{33} = \frac{\dot{Q} \cdot \mu \cdot l}{A \cdot \Delta p} \tag{1}$$

with $\dot{Q}$ being the flow rate, μ the dynamic viscosity, l the flow length (equal to the preform thickness respectively the cavity height), A the cross section perpendicular to the fluid movement and Δp the pressure gradient.
For conventional off-plane permeability equipment spacer frames with discrete dimensions are used to ensure cavity height. Stiff metal structures with equidistant holes distribute the fluid and limit the cavity on the upper and lower side of the preform. Along with the inlet and outlet structures, the setup is bolted together so that the sample is compacted to the height of the spacer frames (Figure 1). A similar method that is based on Eq. (1) is described in [10] where the mass flow is kept constant and the differential pressure is logged (continuous method). It is to be noted that the determination of the off-plane permeability in accordance to equation (1) is associated with a set of assumptions as follows: constant viscosity, pressure difference and flow rate (not for continuous method) within the measuring duration, homogeneous fluid flow perpendicular to A as well as a homogeneous FVC within the preform.

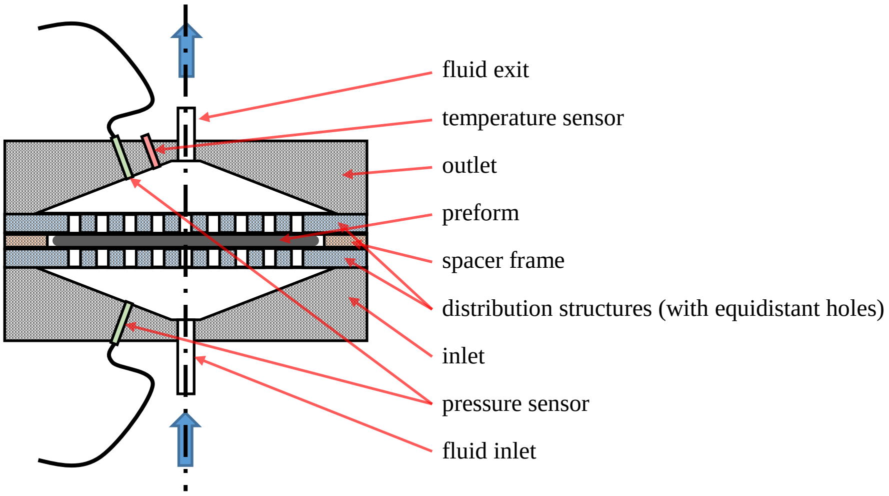

Figure 1. Conventional off-plane permeability equipment

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2 Optimization of permeability test method and experimental work

2.1 Development of a combined compaction and permeability measurement method

In order to investigate the transverse permeability and the main influencing parameters it is necessary to generate reliable measuring data. For this purpose, an enhanced permeability/flow measuring system based on the conventional transverse permeability measurement method is developed. The system is to be integrated in a universal testing machine (Hegewald & Peschke Inspekt 250) in order to measure compaction forces, additional fluid-indicated compaction forces (hydro-mechanical load) and to provide a method for the flexible variation of the cavity height. It thus becomes possible to test textiles with different areal densities at the same FVC without varying the tool hence scaling of results based on the Koceny-Carman model also becomes unnecessary. System and sensors are designed in such a way that enables the investigation of compact and low permeability materials up to 12 mm height. A further advantage is that the influence of temperature and associated fluid viscosity can be studied by integrating the entire measurement setup in a temperature chamber that might be attached to the universal testing machine. The newly developed measuring equipment and the method are schematically presented in Figure 2 and Figure 3.

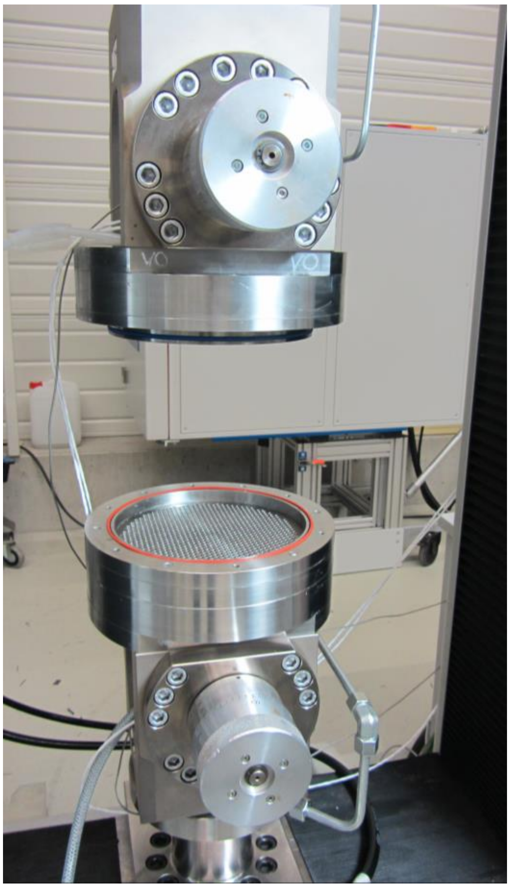

Figure 2. Transversal permeability measurement equipment

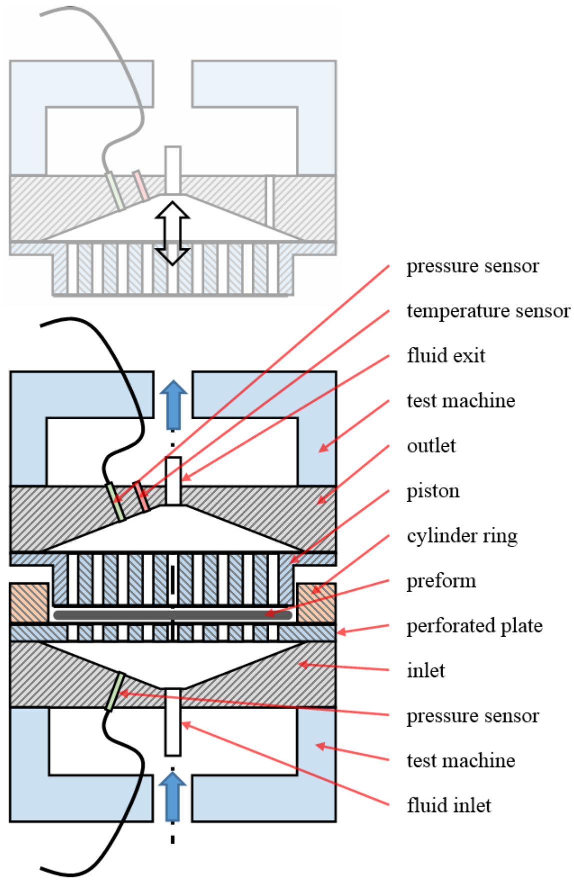

Figure 3. Schematic sketch

In the classical method for transverse permeability measurement it is widely spread to use metal distribution structures with equidistant holes to compact the preform to the final cavity height which is consequently identical to the preform thickness / flow length *l* given in Eq. (1). This distance is a calculated value, determined through the initial definition of the desired FVC to be investigated. A

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plausible reason for the scatter of transverse permeability results might be the inhomogeneous FVC due to fibre undulations using the common distribution structures with equidistant holes. When the cavity is closed and the preform is compacted to the desired FVC the fibres bend into the holes, consequently varying the FVC at this position in comparison to the surrounding positions of the preform as shown in Figure 4 and Figure 5.

2.2 Sample preparation and Materials

To explore the validity of an inhomogeneous FVC a curing device with changeable compaction inserts was developed. Test samples were infiltrated under the same conditions as in the permeability measuring device and subsequently cured. The tool used for this purpose is presented in Figure 6.
Eight layers of conventional bi-diagonal Carbon fibre NCF from Saertex with 254 g/m² and a stacking sequence of $[45/-45]_{4S}$ were impregnated preliminary with Epoxy resin (Momentive Epikote RIM 135). The impregnation was done by Vacuum Assisted Resin Infusion. After complete impregnation the vacuum bagging was opened and the wetted preform was then placed in the curing device. A universal test machine (Zwick/Roell Z050) was used to compact the preform to the desired FVC of 55% (respective 2mm cavity height). The compaction inserts (R=12.5mm) (Figure 6 and Figure 7) contained holes (R=2mm) of the same size as in the permeability measuring equipment to induce similar fibre undulation. After complete curing, samples (58x58 mm²) were tempered and cut.

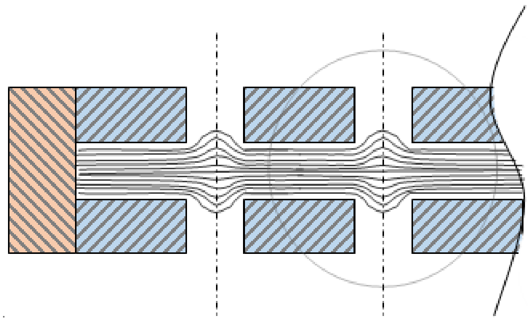

Figure 4. Bending of fibres into the holes

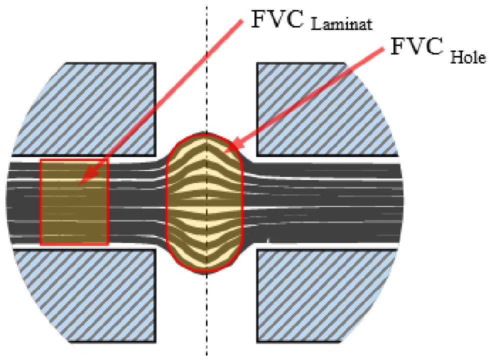

Figure 5. Details and FVCs

2.3 Characterisation

To quantify the effect of varying FVC at the hole positions and surrounding area respectively, samples were cut from the cured panel and polished to create micrographs and specimen for further geometric analysis. The specimen were cut according to the pattern depicted in Figure 7 and according to Table 1. The FVC was determined by thermogravimetric analysis (TGA) using a Netzsch TG209 F1. Due to the small size of the specimen the determination of the FVC according to the dissolution method [11] (using sulphuric acid) was not possible. The concept of FVC determination through TGA is the ignition of the polymer matrix within a composite specimen by slowly (1 K/min) heating it in an oxygen-free environment up to 1000°C. The residual mass of the fibres is virtually stable, allowing the mathematical determination of the FVC with the knowledge of fibre and composites densities. These are determined by the buoyancy method [12] using an adequate setup on a sensitive balance (Sartorius MSA 225P-100-DI).

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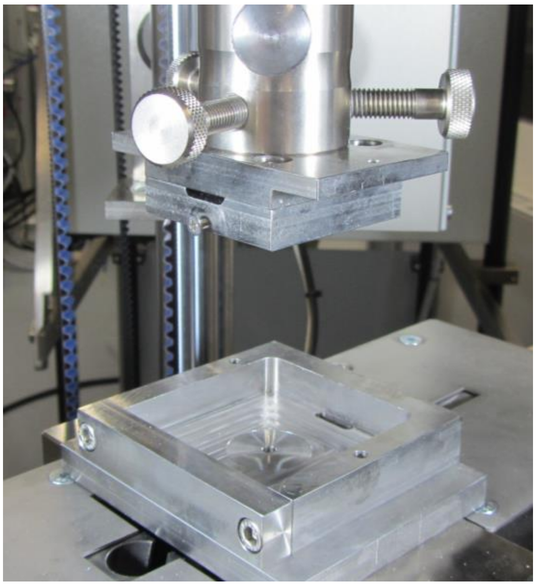

Figure 6. Sample production device for FVC measurement specimen

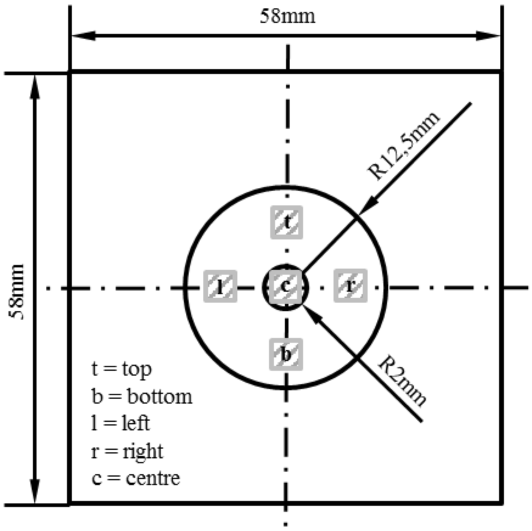

Figure 7. Sample sizes and specimen extraction points

3 Results and Discussion

The validation of the homogeneity of FVC during conventional off-plane permeability tests has not been investigated before. The preform is usually not visible when tested and in most cases curing within the permeability equipment is not possible. After the permeability measurements the preform is unloaded and the compaction state cannot further be investigated.

A simplified curing device was used to completely cure preform at the same conditions compared to the permeability measurement. Visual assessment of the samples emphasised the guess of inhomogeneous FVC due to clearly visible bending of the fibres. A representative micrograph is presented in Figure 8. It can be seen that in the area of the hole significant bending occurs. The laminate thickness at the hole increases up to 126% of the thickness beyond the hole. Moreover, the increased distance between the filaments indicates a lower FVC compared to the properly compacted area beneath.

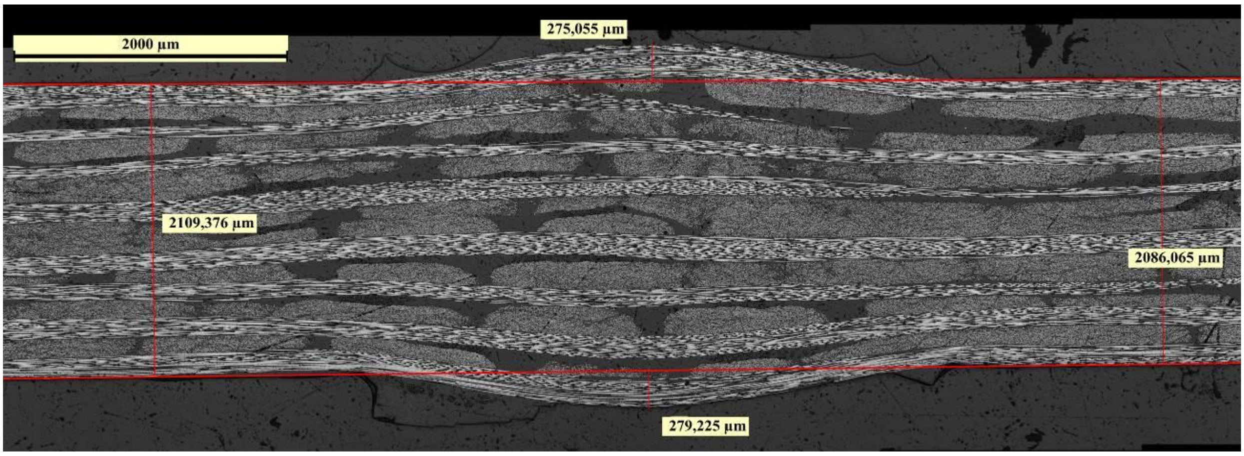

Figure 8. Micrograph of the area of a hole

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Figure 9 summarises the results of the FVC analysis. Due to the use of the unstandardized TGA method for FVC values, the average FVC for all specimen taken from positions outside the influence zone of the hole was calculated and defined as 100% reference. For each sample the FVC value of the centre specimen and the average of all FVC values from the extraction points around the hole (and the standard deviation) are shown in Figure 9.
The deviation of the average FVC values from the area beyond the hole seems unexpectedly high. The reason for this scatter might be the small size of the TGA specimen that ranges from ~10mm³ to ~23mm³ with edge lengths between 2,2mm x 2,5mm and 1,6mm x 4,3mm. The stitching of the used bi-diagonal Carbon Fibre NCF is done with a distance of ~5mm causing a locally increased resin rich area at the stitching points. The FVC therefore might vary depending on the number of stiches within the taken specimen.
The decrease of FVC for the centre specimen is 17% for the 1hole_Sample_2 and 11%, 20% for the other samples. As shown for the samples taken from the area beyond the centre this decrease of FVC is larger than the variation that can be explained by the stitching of the NCF. This proves the theory that local undulations occur in the areas of the holes where no compaction is applied, necessarily resulting in locally lower FVC.

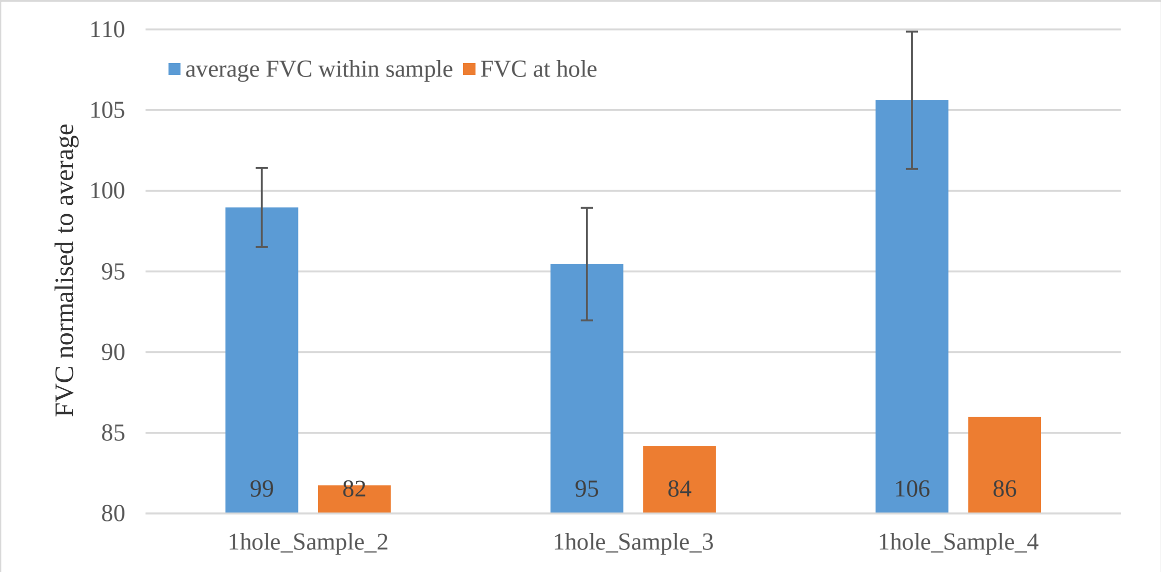

Figure 9 Results of FVC measurements with TGA (normalised)

Based on the results of this investigation it can be stated that the FVC significantly differs across the preform compacted by structures with equidistant holes. In consequence the assumption of a homogeneous FVC is disproved with this study.
For the measurement of transverse permeability with these structures it is therefore obvious that permeability depends on the size, the distance and the arrangement of the holes. The mean FVC will result from the intensity of the undulations at the holes, the compacted area of the preform and a transition zone in between in which the amount of bending/undulation of the fibres depends on the used material in terms of stitching, binder usage, textile design etc.

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Table 1. Results of FVC measurement with TGA

Specimen Type	No of specimen	Mean FVC of specimen outside centre normalised	Standard deviation	FVC of centre specimen normalised	Extraction points
	-	%	%	%	
1hole_Sample_2	3	99	2,4	82	c,b,t
1hole_Sample_3	5	95	3,5	84	c,l,r,b,t
1hole_Sample_4	5	106	4,2	86	c,l,r,b,t

4 Conclusions

This study evolved during the validation of the new developed and optimised permeability measurement method. The common assumption of homogeneous FVC during the measurement of off-plane permeability was investigated using a curing device to produced samples. Measurements of the FVC were made and the results show a significant deviation of the FVC from specimen taken of the area of the holes and the area beyond, reaching up to 20% of local FVC reduction. Resulting from this effect it can be stated that off-plane permeability values depend on the layout of the metal distribution plates and the hole sizes and pattern. Hence the comparability of the measurement values will only be possible when identical distribution structures are used.

5 Further Work

In context to the results of this investigation Fraunhofer FIL is further optimising the transverse permeability test method. The usage of alternative distribution structures is examined and the fluid behaviour within the preform and the surrounding structure is simulated with Ansys CFX. The influence of the material, design and process parameters are investigated to provide optimisation strategies for low permeability preforms and reliable data for impregnation simulation software.

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