IN-PLANE PERMEABILITY MEASUREMENTS: STABILITY, REPEATABILITY AND REPRODUCIBILITY

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SUMMARY: Three methods to measure the in-plane permeability have been compared by theoretical analysis and repeated measurements. Based on this comparison the saturated parallel flow technique is suggested for continued evaluation. The stability of this method is better than 1%. However when doing repeatable measurements with new samples the averaged standard deviation increases to 14 % and 20 % for the two fabrics tested. By a round-robin comparison at three laboratories it is found that the reproducibility of the saturated parallel flow technique is in the same range as the repeatability of the method. It is clear that the scatter is due to the sample preparation, variations in the material and the method of loading the sample. Accordingly, a continued work should be concentrated on improvements of the sample preparation, and developments of methods to reduce the influence, from small errors, in sample preparation and sample loading on the results.

KEYWORDS: Permeability, measurement, in-plane, repeatability, liquid moulding, Resin transfer moulding, RTM, vacuum bag injection.

INTRODUCTION

When polymer based fibre composites are manufactured, the polymer impregnates the reinforcing fibres. The driving force for this impregnation can be an applied pressure, gravity or the capillary pressure. In order to predict the time for the polymer to impregnate a certain volume pore space and the corresponding impregnation pattern, analytical expressions and mould filling simulation codes have been developed [1-4]. These helpful tools are based on Darcy's law, which can be used for flow in porous media as long as the Reynold's number is sufficiently low, the fibres are stationary and the fluid can be modelled as Newtonian (which is frequently the case for thermosetting polymers). Darcy's law can in its general form be written as [5-7]:

$$v_i = \frac{K_{ij}}{\mu} p_{,j} \qquad (1)$$

where v is the superficial velocity, K the permeability μ the viscosity and p the pressure. The permeability is a property that is only dependent on the size of the fibres and the fibre architecture. It can be treated as a tensor and it generally has different values in different directions. It is necessary to know the permeability of the reinforcement for computations of the filling time and the filling pattern. The knowledge of the permeability values is also essential for the development of high permeable fibre reinforcements. The theoretical expressions that have been derived for the permeability are based on some specific and simplified fibre architecture and do not apply to a general case [8-17]. Hence, methods to measure the permeability have been developed. This work will focus on the in-plane permeability, which is of interest for liquid composites moulding techniques such as the Resin Transfer Moulding Process (RTM).

There are two principal ways to measure the in-plane permeability of fibre reinforcement i) by the parallel flow technique and ii) by the radial flow technique [18]. Either of these techniques has a multitude of variants such as measuring the permeability during wetting or saturated flow, driving the measuring liquid by a constant flow rate or a constant pressure. In [19] the three methods *wetting radial flow*, the *wetting parallel flow* and the *saturated parallel flow* are compared. One main result is that the wetting flow front introduces an error on the measured permeability. The wetting permeability is, for instance, higher than the saturated permeability for a random mat. This is explained by capillary action at the flow front. For a weave the wetting permeability is lower and it is assumed that this is caused by inclusion of air at the liquid flow front. The results in [19] indicates that the presence of a flow front in the porous media influence the measured permeability while the actual flow geometry (parallel or radial) does not. In [20] the parallel flow technique is used to measure both wetting and saturated permeability in one measurement. It is found that the permeability does not reach an equilibrium value until the liquid has flown out of the reinforcement for a rather long time. In [21] the differences in permeability obtain in the wetting radial flow and the wetting parallel flow are explained with differences in the flow front speed.

There are however studies that have yielded results somewhat different to those above [22, 23]. Ahn et al [22] compared results from wetting radial flow and wetted parallel flow and found no major difference in permeability. Gebart et al [23] measured permeability with wetting radial flow, wetting and saturated parallel flow. Although the wetting parallel flow method gave slightly higher permeability values as compared to the saturated parallel flow method, it is concluded that the three methods give similar results if the deflection of the mould is under control. It was also pointed out that it is more difficult to keep the mould deflection low enough with the radial flow technique since the mould must be transparent and quite large. Interestingly the measurements in [22, 23] are carried out with a constant pressure while in [Parnas, 1995 #95; Diallo, #38; [21]] the flow rate is constant.

Two recent studies on the influence from the liquid used on the permeability have resulted in completely different conclusions. In [24] the measured permeability increased from the case when syrup/water was used as measuring liquid via vinylester to motor oil. The deviation in permeability was traced to capillary effects. On the other hand in [25] the measured permeability was shown to be independent on the contact angle and the capillary number and the value on the permeability became the same regardless of the liquid used.

The scatter in the measurements is treated in some of the studies. A derivation of the results in [24] yield that the averaged scatter is about 10% and the maximum scatter 20%. Parnas et. al. [19] derived an overall scatter of 15% but concluded that the scatter should have been as high

as 50% if the measurements with a considerably edge flow had not been excluded. High scatters were also obtained in [26], [25] and [27] with scatters up to 30%, 40% and 45% respectively.

To summarise a number of studies have been presented that consider permeability measurements on fibre reinforcements. Still some questions remain unanswered, like the influence from method, liquid etc. These questions were addressed in a recent study [28] with the aim to clarify some of the uncertainties with permeability measurements and to propose one method for a continued evaluation. Three methods to measure the in-plane permeability were compared namely the parallel wetting, the parallel saturated and the radial wetting method. The comparison yielded that the saturated parallel flow method is the most suitable method for a continued evaluation. We will here further discuss the experimental results obtained with this method. This discussion will be based on the stability, the repeatability and the reproducibility of the method. These parameters are here defined as follows:

- *Stability:* The alteration of the value during a continuous measurement.
- *Repeatability 1:* The variation in the results when the same sample is reloaded into the equipment.
- *Repeatability 2:* The variation in the results when new samples are used.
- *Reproducibility:* The variation in the results when the measurements are repeated by a new set of persons at a new location.

EXPERIMENTAL SET-UP

The parallel flow unit used in this study consists of four cavities and is commonly referred to as the multi-cavity parallel flow cell [23, 29]. The conventional way to use this equipment is to load three of the cavities with samples of the fabric, cut in three different directions. The fourth cavity can be loaded with a reference material with known permeability, capillary tubes, for instance. It can also be filled with an additional specimen. In the present study the capillary tubes were placed outside the tool and all four cavities were used for the permeability measurements. The advantage of using these capillaries is that the numerical value of the constant driving pressure and the viscosity of the liquid do not have to be known as long as they stay constant during the measurement [23]. The mould is made of one stiff steel base (750x500x25 mm^3) and two glass platens (750x180x20 mm^3) on top of this, see Fig. 1. The glass platens make it possible to follow the flow during the filling so that measurement errors due to edge effects, can be identified and excluded. The spacers used in the parallel tool are 6.35 mm (0.25 inches) for the measurement on stability and the first repeatability value. For the measurements on the second repeatability value and the reproducibility the spacers were chosen to be 3 mm and 3.3 mm.

Fig. 1: Loading of the multi-cavity parallel flow cell.

The materials utilised for the measurements are listed in Table 1. The Textile Technology fabric is a 3D weave that has been proposed to be a reference material for in-plane permeability measurements [19]. The specimens used in the measurements were cut from the fabric-roles of interest by a steel roller knife. For some of the experiments it is of highest importance that the variation in material is minimised between each specimen. In this case stacking several meters long layers of the fabric on top of each other, reduced the influence from the geometrical variation in the production direction of the fabric. The specimens were then cut out from this large stack. The number of layer was chosen so as to give a desired fibre volume fraction in the tool cavity and the size of the samples was 150x300. After being cut out, the samples were put into the mould cavities. The transparent tops were then clamped to the steel base by bolting stiff steel U-beams to the base. The screws were tightened to a torque of 40 Nm and the test liquid was injected into the mould by means of a pressure vessel, which uses compressed air as pressure source. The air pressure inside the vessel was adjusted with a standard pressure regulator and measured with a high-resolution manometer. The pressure inside the mould was measured near the inlets with a WIKA 891.13.520, 0.25 MPa, pressure transducer connected to a digital voltmeter. The driving pressure was between 0.05 MPa and 0.15 MPa. The flow rate through the fully impregnated reinforcement was obtained by weighing the amount of liquid passing through the specimen during a certain period of time (3-6 min).

Table 1. The reinforcement used in this study.

Designation in this paper	**Fabric**	**Term**	**Type**	**Material**	**Surface Weight (g/m^2)**
NIST	Textile Technologies	-	3D Weave	Glassfibre	4180
NCS	Devold	DBT 800	Non-crimp stitched	Glassfibre	800
Weave	Ahlström	2400 tex	Weave	Glassfibre	800

The experiments carried out may be divided into the following three groups: i) Stability and repeatability of results with the same specimen. ii) Influence from sample preparation i.e. repeatability 2 iii) A round-robin study. The main aim with the *first experimental series* was to test, if the permeability of a certain specimen varied during the measurements but also, as well, the conformity between the results generated in the different cavities as the influence from pressure was checked. Two specimens were taken from the NIST fabric and each specimen was placed in a cavity, while the two other cavities in the tool were blocked. With this set-up the permeability was first measured by the wetting method at 0.05 MPa and then by the saturated method as a function of pressure in the following succession: 0.05, 0.1 and 0.15 MPa. The samples were then moved, so that the permeability was measured on each sample in each cavity. Finally the specimens were placed in their original cavities and the permeability was, once again, measured as a function of pressure. This time realised by starting at the highest pressure. *The second experimental series* considered the scatter in the sample preparation. The specimens were placed in the cavities in random order. The permeability was measured for five and three sets of samples of the weave and the NCS fabric, respectively. As a *third experimental series* a sort of round-robin study was performed. The parallel flow cell was sent to the participating industries, which measured the permeability on the same fabrics as the ones used in the second experimental series. In this case the permeability was only obtained from the calibrating unit and three sets of samples was used for each fabric and fibre volume fraction. The measuring techniques were partly transferred at a workshop and partly through written guidelines.

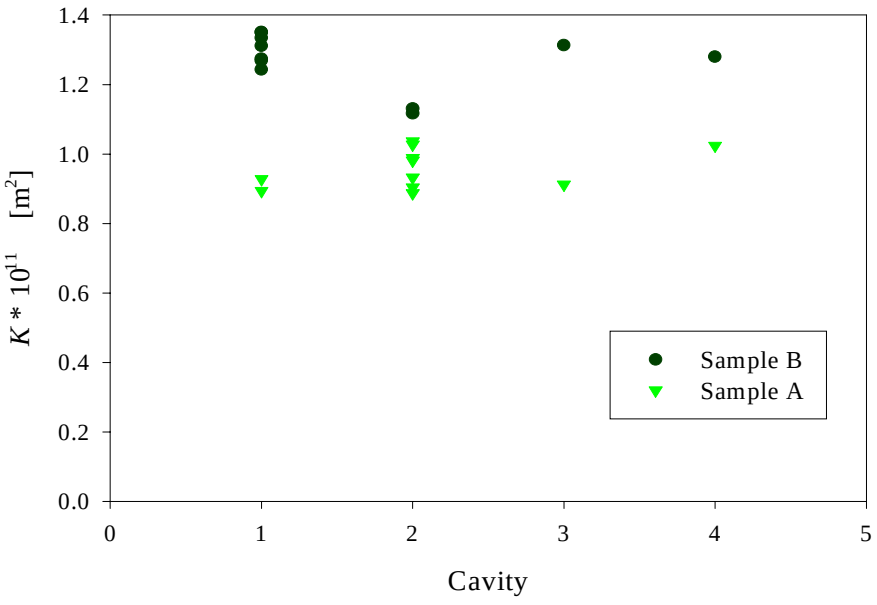

Fig. 2: Permeability as a function of cavity in the multi-cavity parallel flow cell for the two specimens used in the first experimental series.

EXPERIMENTAL RESULTS AND DISCUSSION

In what follows the effective permeability values will be discussed, since these are the values directly obtained from the measurements. From these values the principal permeability values can be computed by the technique outlined in [30]. The effects from the approximations made should, however, be considered [29]. The results of the measurements from the *first experimental series* are shown in Fig. 2. As appears, there is no correlation between the number of the mould cavity and the permeability. It is, however, clear that the permeability is higher for specimen B than for specimen A. This indicates that the geometry of the mould cavities is better controlled than the sample preparation and/or the sample material itself. A small increase of the permeability with the pressure can be spotted in Fig. 3, where the first and last three saturated parallel flow measurements in this series are presented. The dependence on the pressure is much less than the difference in permeability between the two samples. Furthermore, the measured permeability is higher in the repeating experiments than in the original ones. A paired comparison yields an average difference of $0.08 \cdot 10^{-11}$ m^2 (about 7 % of the average absolute value) and a 99% confidence interval of $\{0.01 \cdot 10^{-11}, 0.15 \cdot 10^{-11}\}$ m^2. It must, however, be noticed that the samples were moved among the cavities between these measurements, which might have affected the geometry of the samples. In order to get an estimate of the stability of the test method without movement of the samples, the influence from the pressure on the first three first saturated measurements were compensated by using the results from the last three measurements. These calculations yield that the deviation between the highest and lowest value of the three first measurements is about 1% for both specimens which, in fact, is a value on the stability of the method.

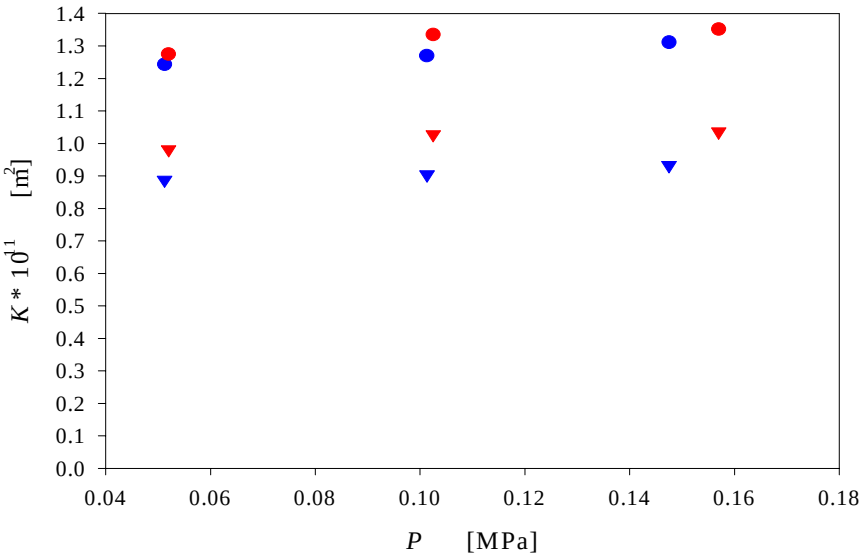

Fig. 3: Permeability as a function pressure as measured at the beginning of the first series, the blue symbols (the slightly lower values), and the permeability measured at the end of it, the red symbols (the slightly higher values). The triangles and the circles denote sample A and B respectively.

In the *second experimental series* a measure of the repeatability in specimen preparation and the material itself was derived, see Table 2 and 3. The results are based on the permeability values as calculated from the permeability reference. These results are, with only a few exceptions, very close to those obtained from the measured pressure and the viscosity. On the average the pressure/viscosity permeability is only 1.7 % higher than the reference permeability with a maximum discrepancy of 7.1 %. Also the evaluation of the permeability in time was checked without noticing any general trend. Such a trend would certainly have been discovered since the experiments were carried out in a random order. This encouraging outcome allows us to further discuss the results in Table 2 and 3. The standard deviation is higher for the weave than for the NCS fabric. The probable cause of this is that the rather coarse weave is easy to deform, when the samples are prepared. The scatter can at a first glance seem to be rather high but it is in agreement with the results presented in the literature [19, 24-27]. A possible way to reduce the scatter is to improve the sample preparation, for instance, by stamping the specimens instead of cutting them. Two other alternatives are to improve the sealing in the mould and to measure just the bulk flow through the sample. One must, however, realise that there is a variation of the permeability within each role of fabric. In these measurements the affect from variation along the production direction of the fabric was minimised by the special technique described in the previous section. However, any variation perpendicular to the production direction will show up as a scatter.

Table 2: Effective permeability values of the weave.

Saturated flow	V_f **(%)**	$K_{eff} \cdot 10^{11}$ **(m²)**		
		0°	**45°**	**90°**
Mean value	50.2	6.35	6.94	11.05
Standard deviation		*20.7 %*	*13.7 %*	*15.9 %*
Conf. interval (95%)		*25.7 %*	*17.0 %*	*25.3 %*
Mean value	54.8	2.67	2.71	4.07
Standard deviation		*24.8 %*	*18.3 %*	*30.1 %*
Conf. interval (95%)		*30.7 %*	*22.7 %*	*37.3 %*

Table 3: Effective permeability values of the NCS-fabric.

Saturated flow	V_f **(%)**	$K_{eff} \cdot 10^{11}$ **(m²)**		
		0°	**45°**	**90°**
Mean value	52.5	6.72	7.39	9.78
Standard deviation		*7.71 %*	*11.7 %*	*11.1 %*
Conf. interval (95%)		*19.1 %*	*29.0 %*	*27.6 %*
Mean value	57.2	3.48	3.95	5.14
Standard deviation		*16.9 %*	*19.3 %*	*15.1 %*
Conf. interval (95%)		*42.0 %*	*47.9 %*	*37.5 %*

In the round-robin study (the *third experimental series*) two additional sets of data were produced at two additional labs. These permeability values are in most cases lower than those presented in Table 2-3. The mean discrepancies vary between -11 % and -34 %; cf. Table 4. However, two of the four confidence intervals presented include the possibility that the institute measured lower values than the industries. The implication of this is that if the spread in the results is accounted for it cannot with 95% security be stated that the industries measured lower values than the institute. Furthermore, industry 1, on the average, gets lower permeability on the weave than industry 2, while it is vice versa for the NCS-fabric. It is also

clear that the spread of the results from the Round-Robin study is in the same range as the spread obtain in the material and the sample preparation; cf. Table 2-4. Consequently, it seems to be possible to obtain similar permeability results at another laboratory as long as the transferring of the know-how of the measuring technique is done in a proper way. For instance at a work-shop.

Table 4. Mean discrepancy from the saturated permeability values derived at the institute. The discrepancies are obtained by a paired comparison **[31]**.

Laboratory	**Material**	**Mean discrepancy (%)**	**Confidence interval 95 % (%)**	
Industry 1	Weave	-33.8	-19.8	-47.8
Industry 1	NCS-fabric	-12.6	4	-29.2
Industry 2	Weave	-11.0	3.6	-25.6
Industry 2	NCS-fabric	-19.7	-9.7	-29.7

CONCLUSIONS

The statistical data on the multi-cavity parallel flow cell are summarised in table 5. As seen approximately half of the scatter in the repeatability value can be traced to the loading of the sample while the rest can be traced to the sample preparation and the variation in the material it-self. Hence, improvements of loading techniques and sample preparation would certainly result in a better repeatability in the results.

Table 5. Statistical values on the saturated parallel flow method.

Measure	**Deviation**	**Comments**
Stability 1	1 %	This value is obtained without reloading the sample. The value is obtained from a total of three measurements.
Repeatability 1	7 %	This value represents the whole test method including loading of the sample and it was obtained after a total of five measurements.
Repeatability 2A	Std 20 %	This value is based on five sets of specimen from the weave used in this report.
Repeatability 2B	Std 14 %	This value is based on three sets of specimen from the NCS-fabric used in this report.
Repeatability 2C	Std 10 %	This value is based on five sets of specimen from the NCS-fabric used in [29].
Reproducibility A	- 11.0 % - 33.8 %	The values represent the deviations in results as obtained at the industries for the weave.
Reproducibility B	- 12.6 % - 19.7 %	The values represent the deviations in results as obtained at the industries for the NCS-fabric.

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