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PERMEABILITY TESTING AND CHARACTERISATION OF GLASS FIBRE REINFORCEMENTS

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SUMMARY: At the CRC-ACS, two different test methods for measuring reinforcement permeability have been developed. The first of these, designed to be a quick and easy technique, employs a vacuum infusion process where fluid is drawn linearly through the reinforcement under a vacuum bag using vacuum. Flow position and time is recorded from which the in-plane permeability can be calculated for a single fibre volume fraction. The second method uses radial flow where a fluid is injected under constant pressure into the centre of a preform located in a fixed cavity tool. Flow position, time, flow rate and fluid pressure are recorded and used to determine the in-plane and through-thickness permeabilities of the reinforcement for a predetermined fibre volume fraction. In this paper, details of the two test methods are presented along with the analysis required to calculate the in-plane and through-thickness permeabilities. Results from tests of several reinforcements with various configurations are also presented. From the testing, it was found that the fibre orientation of the outer faces of the preform strongly influenced the permeability.

KEYWORDS: Liquid Moulding, Resin Infusion, Permeability, Flow

INTRODUCTION

Liquid moulding processes such as resin transfer moulding (RTM), resin film infusion (RFI) and vacuum bag resin infusion (VBRI) work on the principle of filling a dry reinforcement/preform with a liquid resin using an applied pressure gradient. To predict the flow behaviour of these processes, various analytical solutions and simulation codes have been developed [1,2]. Most of these are based on Darcy's Law and therefore it is important that parameters which include fluid pressure, fluid viscosity and preform permeability be known. The latter two are physical property data and therefore must be measured. The fluid viscosity is dependent on the fluid type and process temperature and can be characterised using standardised tests. The preform permeability is more difficult to characterise in that it is dependent on the fibre type, the reinforcement architecture, fibre volume fraction and fibre direction. Theoretical expressions have been derived to predict permeability [3]: however they are based on specific and simplified fibre arrangements and do not apply to general cases. Subsequently, there is a need to measure the permeability (in-plane and through-thickness) of the preform.

There are two different approaches which can be used to measure permeability [4]. The first is linear or parallel flow where a calibrated fluid is injected from a side gate and travels in a linear fashion. The second is radial flow where the fluid is injected from a central gate and flows outwards in all directions. Radial flow has an advantage over linear flow in that the in-plane permeability can be measured in both principal directions in the one test, whereas a linear flow test can only be used to measure flow in a single direction. With both approaches there are a number of variants which include injecting under constant pressure or constant flow rate, and measuring permeability during wetting or saturated flow.

At the CRC-ACS, two different test methods for measuring preform permeability have been developed. The first of these, designed to be a quick and easy technique, employs the Vacuum Bag Resin Infusion (VBRI) process where fluid is drawn through the reinforcement under a vacuum bag using vacuum. The process uses linear flow, and the flow position and time are recorded from which the in-plane permeability can be calculated for a single fibre volume fraction. The second method uses radial flow where a fluid is injected under constant pressure into the centre of the preform located in a fixed cavity tool. Flow position, time, flow rate and fluid pressure are recorded and used to determine the in-plane and through-thickness permeabilities of the preform for a predetermined fibre volume fraction.

In this paper, details of the two test methods are presented, along with the analysis required to calculate the in-plane and through-thickness permeabilities. Results from the permeability tests of several reinforcements, which include unidirectional and multi-directional non-crimp fabrics, with various configurations are also presented. Influences of fibre orientation of the outer faces of the reinforcement are also investigated and discussed.

PERMEABILITY TEST METHODS

VBRI Method

The first of two methods developed at the CRC-ACS, the VBRI method was designed to be a quick, easy and cost-effective technique for measuring the permeability of a preform. The method is based on linear flow and employs the VBRI process where a calibrated fluid is drawn through the reinforcement under a vacuum bag using vacuum.

Theory

Applying Darcy's law in one dimension, the in-plane permeability for a linear flow can be represented as Eqn. 1. Hence, by measuring the distance-time history during the permeability test, calculating the fibre volume fraction after the test and knowing the fluid viscosity, the in-plane permeability can be determined as follows:

$$K = \frac{\mu(1 - V_f)}{2\lambda P_{inj}} \tag{1}$$

where, K = in-plane permeability
μ = test fluid viscosity
V_f = fibre volume fraction
P_{inj} = injection pressure (vacuum level)

x = one dimensional flow distance from start of preform
t = time to reach position x
λ = gradient of line of best fit through t vs. x^2

Apparatus

The test set-up (Fig. 1) comprises a glass base plate onto which the preform, measuring 200 mm wide by 500 mm long, is located. In the case of preforms with expected high permeabilities, such as knits and strand mats, the flow rates are much higher and so a preform length in the flow direction of 1000 mm is recommended. Investigations also revealed that for best results, the preform areal weight should not be less than 1500 g/m^2 for carbon fabrics and 2000 g/m^2 for glass fabrics and that a minimum of two layers of reinforcement be used in the preform configuration.

An inlet and outlet are located at opposite ends of the preform, spanning the entire width. The outlet comprises a plastic grid and barb fitting onto which a 3/8" O.D. polyethylene pipe is attached which is in turn connected to a controllable vacuum source. A vacuum gauge is used to measure the vacuum level. The inlet, which was specially designed to ensure a linear flow front, comprises a plastic 'C' section moulding (approximately 10 mm deep by 10 mm high) into which the plastic grid is placed as well as a 3/8" O.D. polyethylene pipe which is fitted horizontally into one end of the moulding. The other end of the inlet pipe is placed into the test fluid reservoir.

The resulting assembly is encased in a nylon vacuum bag and sealed using sealant tape, which is also used along the edges of the preform to prevent fluid race-tracking. The entire test set-up is elevated to ensure that the level of the test fluid reservoir is below the inlet gate. The vacuum bag also has lines drawn onto it at intervals of 10 mm. This allows the resin flow front position to be recorded against time, which is measured using a stop watch. Thermocouples are also used to monitor temperatures of the test fluid and assembly so the fluid viscosity can be accurately known.

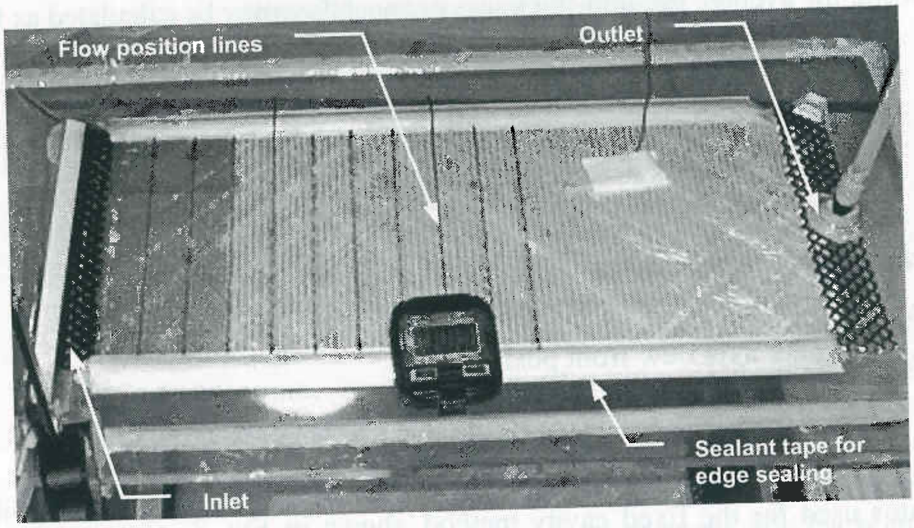


Figure 1 VBRI permeability test method in progress showing a typical flow front

To determine the fibre volume fraction of the preform, the thickness is measured after the test. This is made possible using a test fluid, in this case Dow Derakane 411-350 vinylester, which can be cured. Doing so, the infused preform can be demoulded after cure and the thickness measured at various points throughout the laminate.

Unlike conventional permeability tests which are performed using a controlled thickness (i.e. fibre volume fraction), the thickness for the proposed VBRI test is dependent on the compaction behaviour of the preform. That is, the in-plane permeability for a given preform can only be measured for a single fibre volume fraction which corresponds to the thickness of the preform under 1 atmosphere compaction pressure. Although the vacuum level can be varied to some degree (say between 50 and 100 kPa) to adjust the level of compaction, the variation in fibre volume fraction which can practically be achieved by doing this is minimal.

Fixed Cavity Method

The fixed cavity method involves injecting a calibrated fluid into the centre of the reinforcement using an applied pressure with a predetermined thickness. The method utilises radial flow and enables the permeability to be measured for all three principal directions.

Analysis

Many reinforcements exhibit non-anisotropic permeabilities properties which will cause a radial flow pattern to be elliptical in shape. Elliptical flow in thin preforms can be characterised by two permeabilities, one for both the major axis (k_1) and minor axis (k_2) of the ellipse. These two permeabilities can be calculated using the approximate iterative analytical solution of Adams et al. [5], shown in Fig. 2, from the experimentally measured flow radius for both major and minor axis against time, the injection pressure, preform volume fraction and fluid viscosity.

An analytical technique was also developed to calculate the through-thickness permeability, k_3, for thick preforms. Using the relationship shown in Eqn. 1 of Fig. 2, and measuring the major and minor flow front positions at the time when the fluid travels through the thickness of the preform, the average through-thickness permeability may be calculated as follows:

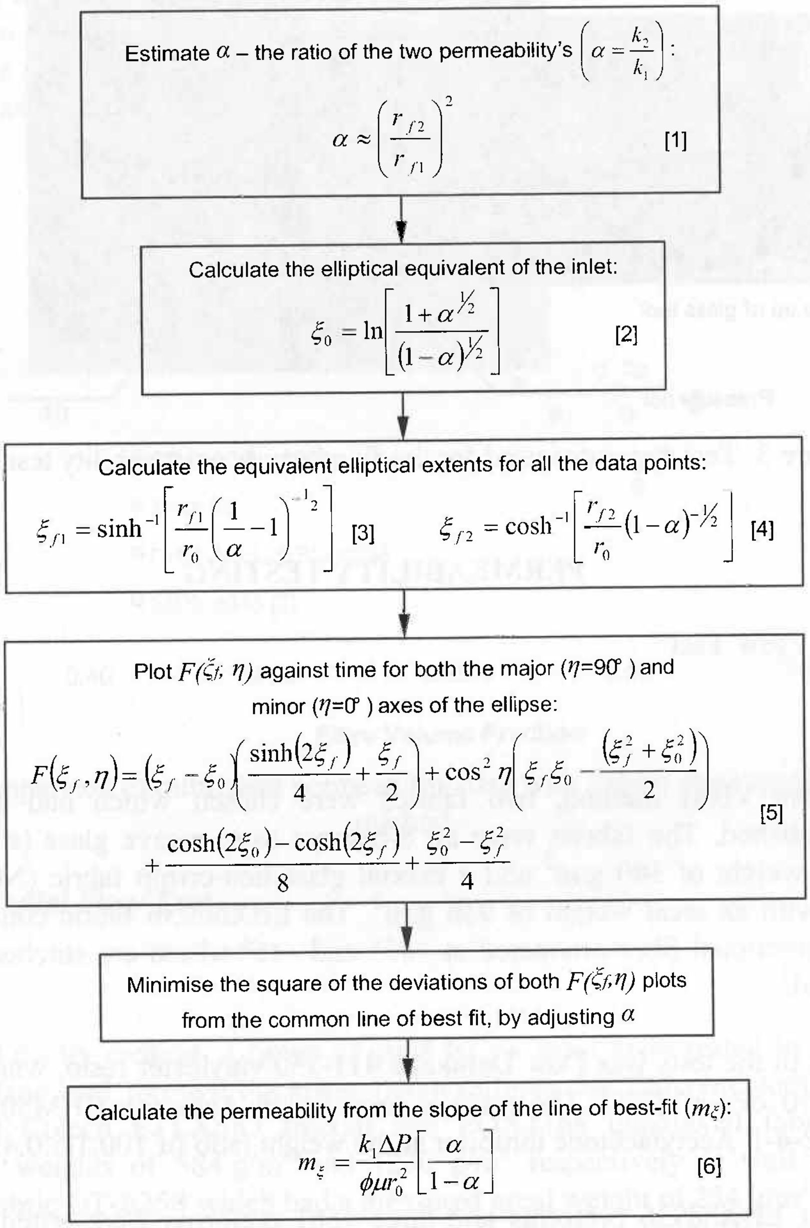
$$k_3 \approx \frac{d^2}{2}\left(\frac{k_1}{r_{d_1}^{\,2}} + \frac{k_2}{r_{d_2}^{\,2}}\right) \tag{2}$$

where,

d = preform thickness
r_{d_1} = major axis flow front position
r_{d_2} = minor axis flow front position

Apparatus

The apparatus used for the fixed cavity method, shown in Fig. 3, comprises a tool (mould) measuring 300 x 450 mm (consisting of glass lid and base clamped together with steel bars and bolts) into which the reinforcement is loaded; a pressure transducer and flow meter which measure the fluid pressure and flow rate; a pressure pot to inject the test fluid under a constant pressure; a stop watch to record time; and thermocouples for measuring temperature of the tool, preform and test fluid. The temperature is important for accurately knowing the test fluid viscosity. The fluid used for the tests is glycerine diluted with water to obtain a predetermined viscosity characterised using a Brookfield viscometer and checked prior to each test using calibrated flow cups.



Estimate α – the ratio of the two permeability's $\left(\alpha = \frac{k_2}{k_1}\right)$:

$$\alpha \approx \left(\frac{r_{f2}}{r_{f1}}\right)^2 \qquad [1]$$

Calculate the elliptical equivalent of the inlet:

$$\xi_0 = \ln\left[\frac{1+\alpha^{1/2}}{(1-\alpha)^{1/2}}\right] \qquad [2]$$

Calculate the equivalent elliptical extents for all the data points:

$$\xi_{f1} = \sinh^{-1}\left[\frac{r_{f1}}{r_0}\left(\frac{1}{\alpha}-1\right)^{-1/2}\right] \quad [3] \qquad \xi_{f2} = \cosh^{-1}\left[\frac{r_{f2}}{r_0}(1-\alpha)^{-1/2}\right] \quad [4]$$

Plot $F(\xi_f, \eta)$ against time for both the major (η=90°) and minor (η=0°) axes of the ellipse:

$$F(\xi_f, \eta) = (\xi_f - \xi_0)\left(\frac{\sinh(2\xi_f)}{4} + \frac{\xi_f}{2}\right) + \cos^2\eta\left(\xi_f\xi_0 - \frac{(\xi_f^2+\xi_0^2)}{2}\right) + \frac{\cosh(2\xi_0)-\cosh(2\xi_f)}{8} + \frac{\xi_0^2-\xi_f^2}{4} \qquad [5]$$

Minimise the square of the deviations of both $F(\xi_f,\eta)$ plots from the common line of best fit, by adjusting α

Calculate the permeability from the slope of the line of best-fit (m_ξ):

$$m_\xi = \frac{k_1 \Delta P}{\phi \mu r_0^2}\left[\frac{\alpha}{1-\alpha}\right] \qquad [6]$$

where,

k – permeability	ΔP – injection pressure
r_f – radius of the flow front	ϕ – preform porosity
r_0 – radius of the injection port	μ – resin viscosity

Figure 2 Approximate iterative analytical solution used to calculate permeability for elliptical radial flow

The glass lid and base, both of which have both a 20 x 20 mm grid drawn on them, enable the flow pattern to be recorded during the test using a digital camera. Having both sides of the tool made from glass enables through-thickness as well as in-plane permeabilities to be characterised. A 16-channel data acquisition system (IOtech Daqbook/112) is used in combination with a lap-top computer to record the flow meter and pressure transducer output.

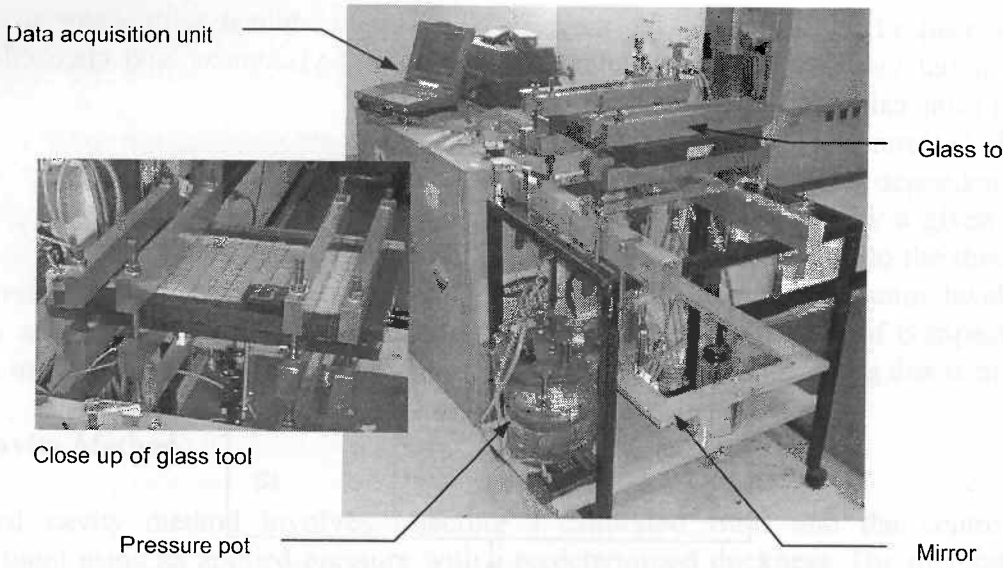


Figure 3 Test apparatus used for the fixed cavity permeability test method

PERMEABILITY TESTING

VBRI Linear Flow Test

Materials

To evaluate the VBRI method, two fabrics were chosen which had permeability data previously published. The fabrics were an 8-harness satin weave glass (style 1581) with a nominal areal weight of 300 g/m^2 and a biaxial glass non-crimp fabric (NCF) from Cotech (EBXhd936) with an areal weight of 936 g/m^2. The EBXhd936 fabric comprises two equal layers of unidirectional fibre orientated at +45° and –45° which are stitched together with a polyester thread.

The fluid used in the tests was Dow Derakane 411-350 vinylester resin, which has a nominal viscosity of 350 cPs at 22°C. The resin is mixed with Andonox CHM50 catalyst, CoNap promoter and 2-4-P Acetylacetone inhibitor at the weight ratio of 100:1.5:0.4:0.1.

A total of four EBXhd936 preforms and three 1581 preforms were tested using the VBRI method. The preforms comprised of three layers and seven layers respectively and were tested at a vacuum level of 80 kPa.

Results

The results from the linear flow tests revealed that the flow front was quite linear throughout the test although the flow front tended to be a little more inconsistent towards the end of the run. A typical flow front pattern can be seen in Fig. 1. Plotting the time (t) against the square of the flow distance (x^2) for each of the runs revealed a linear relationship between the two variables. This is in accordance with Darcy's law and justifies the application of the theoretical model, for calculating permeability, to the experimental data. Using the test results along with Eqn. 1, the in-plane permeabilities for each run were calculated and are given in Fig. 4.

As well as evaluating the consistency of the permeability test, the results were also compared to published data to determine the accuracy of the measured permeability data. The measured in-plane permeabilities for the 1581 and EBXhd936 fabrics are compared with published data in Fig. 4. As can be seen, the results for these materials compare well with the published data, particularly when one takes into account that the tested fabrics were slightly different to the published fabrics (i.e. 1581 and EBXhd936 compared with 7781 and EBXhd948). Even the scatter in the measured values is of the same magnitude as the published data.

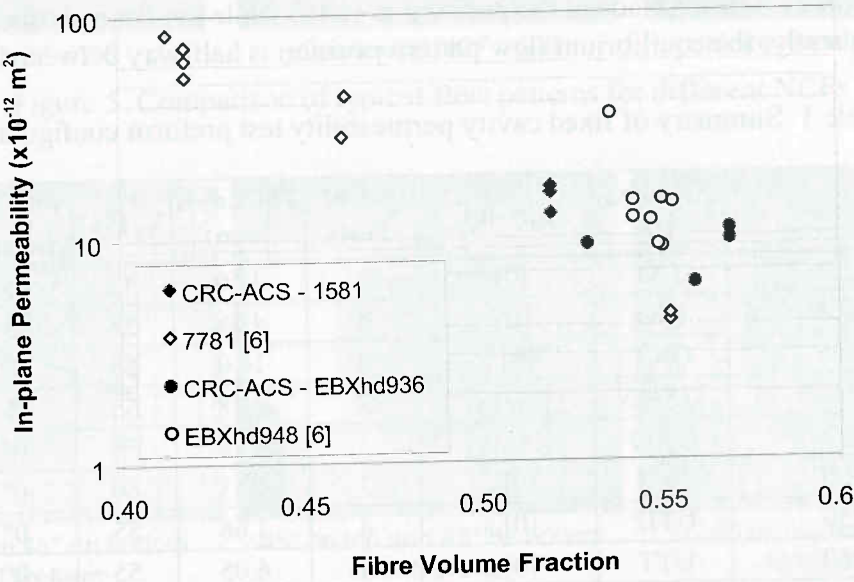


Figure 4 Comparison of published permeability data with values measured by the VBRI method

Fixed Cavity Radial Flow Test

Materials

Using the fixed cavity method, a range of glass NCFs were investigated to determine their respective in-plane and through-thickness permeabilities for different configurations. The NCFs included Cotech ETLX583 triaxial and EQX1168 quadraxial fabrics which had measured areal weights of 584 g/m^2 and 1230 g/m^2 respectively as well as SP Systems unidirectional fabric UT-E250 which had a measured areal weight of 234 g/m^2. The ETLX583 fabric comprises three layers of unidirectional fibre orientated at 0°, –45° and +45°, with the 0° layer making up 50% of the fabric weight. The EQX1168 fabric comprises four equal layers orientated at 0°, 90°, –45° and +45°. With both fabrics, the fibre in the top layer is oriented in the 0° direction while the fibre in the bottom layer is in the +45° direction. To investigate the influence the outer ply fibre orientation has on the permeability, tests were performed on a range of different preform configurations which are summarised in Table 1. The fluid used in the tests was water-diluted glycerine (25% water by weight) which had a viscosity of 35 cPs at 21°C. All tests were performed using an injection pressure between 450 and 500 kPa.

Results

The results from the radial flow tests, which were found to have high repeatability, have been summarised in Table 2 while typical flow patterns are shown in Fig. 5 and 6. As can be seen from the results, the flow patterns and subsequent in-plane permeabilities are dominated by the fibre orientations in contact with the tool surfaces and the proportion of fibres in a particular direction. In the case of the triaxial and unidirectional NCFs (TXI & UTI), the flow pattern is elliptical with the major axis parallel to the 0° fibre direction (see Fig. 5). Larger the proportion of 0° fibres, greater the aspect ratio of the ellipse. In the case of the quadraxial fabric the flow pattern is also elliptical (but to a lesser degree), however the major axis of the ellipse is rotated by approximately 22° from the 0° fibre direction. The rotation of the flow pattern is attributed to the fibre orientation of the outer surface plies. In the case of the QXI preform, the fibre on one surface of the preform is at 45° while the fibre on the other surface is at 0°. Consequently, the equilibrium flow pattern position is half-way between 0° and 45°.

Table 1 Summary of fixed cavity permeability test preform configurations

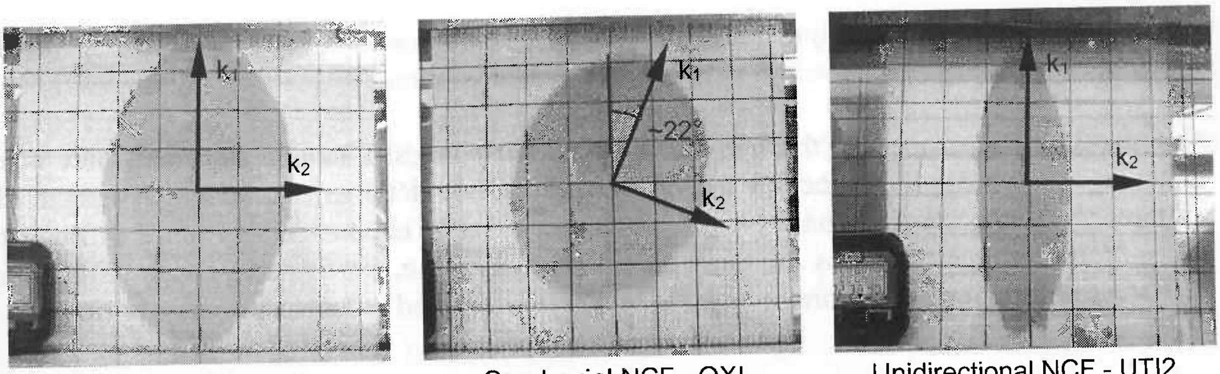
Fabric	**Preform ID**	**Lay-up**[*]	**No. of Tests**	**Thickness (mm)**	**V_f (%)**	**Fibre Orientation**[$]	
						Bottom	**Top**
ETLX583	TXI	$[0]_7$	7	3.10	52	45°	0°
EQX1168	QXI	$[0]_5$	5	4.38	55	45°	0°
EQX1168	QXT	$[0]_{16}$	4	14.0	55	45°	0°
EQX1168	QXI45	$[0_3,0_{F2}]$	2	4.38	55	45°	-45°
EQX1168	QXI0	$[0_{F3},0_2]$	3	4.38	55	0°	0°
UT-E250	UTI1	$[0]_{10}$	2	1.53	60	0°	0°
UT-E250	UTI2	$[0]_{10}$	4	1.66	55	0°	0°
UT-E250	UTT	$[0]_{36}$	4	6.05	55	0°	0°
EQX1168 + UT-E250	QXUT	$[0_1^Q,0_8^U,0_{F1}^Q]$	3	3.10	55	45°	-45°
EQX1168 + UT-E250	QXUT45	$[0_1^Q,0_8^U,0_1^Q]$	1	3.10	55	45°	0°
EQX1168 + UT-E250	QXUT0	$[0_{F1}^Q,0_8^U,0_1^Q]$	3	3.10	55	0°	0°
ETLX583 + UT-E250	TXUT0	$[0_{F1}^T,0_4^U,0_1^T]$	4	1.52	55	0°	0°

[*] F subscript refers to flipping of the ply. Q, T & U superscripts refer to Quadraxial, Triaxial & Unidirectional NCF respectively.

[$] Orientation of fibre in contact with top and bottom tool surfaces.

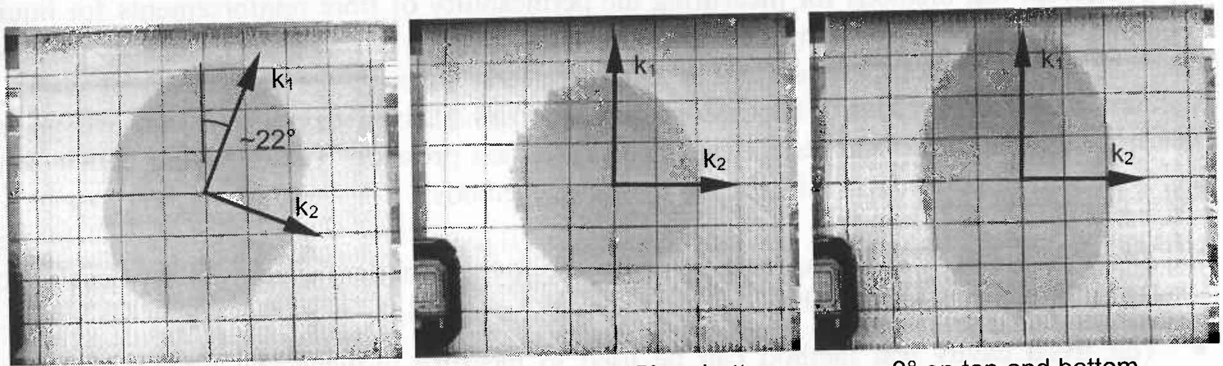
Table 2 Summary of fixed cavity permeability tests with non-crimp fabric preforms

Preform ID	**V_f (%)**	**Flow Orientation**	**k_1 ($\times 10^{-12}$ m^2)**	**k_2 ($\times 10^{-12}$ m^2)**	**k_3 ($\times 10^{-12}$ m^2)**
TXI	52	0°	15.4 ± 1.8	6.78 ± 1.00	-
QXI	55	22°	10.9 ± 1.3	6.09 ± 0.47	-
QXT	55	22°	-	-	0.81
QXI45	55	0°	7.25 ± 0.76	5.40 ± 0.67	-
QXI0	55	0°	13.5 ± 1.1	6.24 ± 0.36	-
UTI1	60	0°	3.02 ± 0.40	0.31 ± 0.01	-
UTI2	55	0°	6.46 ± 0.65	0.66 ± 0.08	-
UTT	55	0°	-	-	0.032
QXUT	55	0°	12.5	6.92	-
QXUT45	55	22° bottom, -22° top	5.79 ± 0.66	4.16 ± 0.81	-
QXUT0	55	0°	11.5 ± 2.5	3.90 ± 1.08	-
TXUT0	55	0°	18.1 ± 2.0	5.08 ± 0.41	-



Triaxial NCF - TXI Quadraxial NCF - QXI Unidirectional NCF - UTI2

Figure 5 Comparison of typical flow patterns for different NCFs



0° on top and 45° on bottom - QXI preform -45° on top and 45° on bottom - QXI45 preform 0° on top and bottom - QXI0 preform

Figure 6 Comparison of flow patterns for EQX1168 with different fibre orientations on the top and bottom surfaces of the preform

The extent of this surface ply influence can be seen with the QXI45 and QXI0 preforms where the fibre orientation of the outer surface plies was changed from 0°/45° to 45°/–45° and 0°/0° respectively. As can be seen in Fig. 6, having 45°/–45° fibre on the outer surface makes the flow pattern almost circular and eliminates the rotation of the flow pattern. Similarly, having 0° fibres on the outer surfaces also eliminates the rotation of the flow pattern but makes the flow pattern more elliptical. The changes in flow pattern translates to variations in the preform in-plane permeability. In the case the EQX1168 NCF, k_1 varies by 100%.

Results from the hybrid preforms (QXUT, QXUT45 and QXUT0) also revealed the same flow pattern changes with the outer surface fibre direction, as was exhibited with the quadraxial NCF preforms. However what was also observed with the QXUT45 preform was that the elliptical flow patterns on the top and bottom surfaces were rotated at –22° and 22° respectively. It appears that the inclusion of the unidirectional NCF in the centre of the preform allowed the outer quadraxial layers to act independently.

The dominating influence which the outer surface plies have on the permeability is attributed to flow channels which exist between the hard tool surface and gaps between fibre tows. For NCF, these channels are clearly defined because of the single fibre direction on the outer plies. In the case of woven fabrics such as plain and satin weaves, these channels are not as evident and therefore woven fabrics do not exhibit the dominant surface ply orientation effects. From these results it can be seen that when measuring permeability values for NCF preforms, it is important that they are tested in the same configuration as they will be implemented in the liquid moulding process. Otherwise, discrepancies of over 100% may exist with measured data which will result in major errors in flow simulation and subsequently incorrect mould and process design.

Comparing the in-plane and through-thickness permeabilities, it can be seen that there is an order of magnitude difference for the quadraxial NCF and up to two orders of magnitude difference for the unidirectional NCF. This implies that it is considerably more difficult to force fluid through-thickness of the preform than in-plane, particularly for unidirectional materials where fibres are more inclined to bunch together and reduce gaps between fibres.

CONCLUSIONS

Two different test methods for measuring the permeability of fibre reinforcements for liquid moulding processes have been developed and are presented in this paper. The first of these uses the VBRI process where fluid is drawn linearly through the reinforcement under a vacuum bag using vacuum. The second method uses a fixed cavity tool into which the reinforcement is placed and resin is injected at constant pressure. The following conclusions can be drawn from results obtained from these two methods:

- The VBRI method is a quick and easy permeability test method which yields permeability data that compares favourably with published values.
- The fixed cavity test method can be used to measure in-plane and through-thickness permeabilities.
- Flow characteristics and permeability values of non-crimp fabrics are strongly influenced by the orientation of fibres on the outer surfaces. For a preform consisting of the the same NCF, the apparent in-plane permeability in the radial test can be changed by as much as 100% simply by flipping the outer surface ply.

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