An experimental study of the permeability and capillary pressure in resin-transfer moulding

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Abstract

This paper addresses issues of the inter-relationship between the permeability and capillary pressure in in-plane infiltration of assemblies of woven fabrics. Rectilinear infiltration experiments of assemblies of a plain-weave fabric at various low injection pressures were carried out to evaluate permeability and capillary pressure ($P_c$) for the woven fibre preform. Capillary pressure and the form factor, $F$, for the Young–Laplace equation were also estimated and a normalised capillary pressure was used to compare the $P_c$ values found at different porosities. Regarding the estimation of permeability, it has been shown that the higher the injection pressure, the lower the associated error of not including $P_c$ into the permeability calculation. When a corrected permeability was calculated taking into account the capillary pressure effect, the permeability proved to be independent of the injection pressure and the permeating fluid. An increase in permeability was found for pre-wetted fabrics in comparison to dry fabrics.

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1. Introduction

Resin-transfer moulding (RTM) has been studied extensively over the last 10 years and its advantages are well established as it is considered a versatile process, able to efficiently produce fibre-reinforced polymeric composites of different shapes and high structural performance for intermediate volume production runs [1]. As a result, RTM has many current and potential applications in a variety of industries, such as automotive, aerospace and sports industries.

The low injection pressure range for the RTM process reduces the cost and complexity of the required tooling. However, it is responsible for forcing the fluid to flow over long distances and infiltrate a dry preform of complex structure. Parnas and Salem [2] noted that this can be regarded as the most unique characteristic affecting the RTM process.

Under certain processing conditions such as low injection pressure and high fibre-volume fraction, the microscopic flow of resin within the fibre tow and the fibre wet-out can considerably influence the overall flow. In these cases, capillary pressure, $P_c$, presents a significant driving force for resin impregnation and, therefore, should be taken into account [3,4] to optimise the use of RTM, reducing, for example, the presence of dry spots and macro- and micro-voids [5].

2. Review on the current trends related to permeability and capillary pressure

Flow models for RTM usually consider the overall flow of an incompressible Newtonian fluid through a porous medium as equivalent to flow through multiple tubes [6] and use the well-known Darcy’s law [7] which is truly applicable only to a homogeneous and random porous medium.

The validity and applicability of Darcy’s law have been questioned by many authors. Visconti et al. [8] identified differences in the measured permeability brought about by changes in injection pressure. Gauvin et al. [9] suggested that the flow rate, pressure and the nature of the fluid are among the factors that could strongly influence...
the permeability measurements. Griffin et al. [10] and Steenkamer et al. [11] found different permeability values depending on the infiltrating fluid. Chan et al. [12] and Daveé [13] observed an increase in permeability with flow rate. Despite these and other findings, Darcy’s Law is still by far the most used flow model and, as mentioned by Parnas [1], many of these discrepancies may be due to neglecting the effects of microscopic flow phenomena on the interpretation of the macroscopic flow.

The one-dimensional Darcy’s Law equation can be expressed as:

\[ u_x = \frac{Q_x}{A} = -\frac{\kappa}{\mu} \frac{dP}{dx} \]  

for

\[ u_x = \varepsilon v_x = \varepsilon \frac{dx}{dt} \]  

where \( Q_x \) is the volumetric flow rate (m³/s), \( A \) is the cross-sectional area of the mould cavity normal to the flow direction (m²), \( u_x \) is the superficial velocity (m/s), \( v_x \) is the interstitial velocity (m/s), \( \varepsilon \) is the porosity of the preform, \( \frac{dP}{dx} \) is the fluid pressure gradient over the specimen (Pa/m) and \( x \) is the length in the streamwise direction (m). Permeability, \( \kappa \), the property that expresses how easy it is for a fluid to penetrate a porous medium, has units of m², although the unit ‘darcy’ (1 darcy = 9.87 × 10⁻¹³ m²) is sometimes used.

The use of the RTM equipment itself is the most often reported way to measure the in-plane permeability of fibrous reinforcements used in RTM. The simplicity of the set-up and the ease with which more reliable and reproducible results can be determined are said to be the major advantages of the rectilinear flow experiments over the radial ones [14]. Lai et al. [15] cited that utmost care and the use of appropriate equations, taking into account the contribution of \( P_c \), resulted in agreement, within the expected range of experimental error, between both types of flow. Misleading estimations of permeability may be a consequence of an irregular flow front, flow channelling, flow leakage over the top or under the bottom of the layers of fabrics, fibre displacement, regions of different porosities, inter-layers within the reinforcement at low compression, edge effects and neglecting the capillary effects.

The empirical Carman–Kozeny equation [Eq. (3)] is often employed to predict permeability, although it can be strictly used only for uniform or truly random packing of fibres [10]. Originally, Kozeny [16] related the pressure drop to velocity by assuming flow through an idealised isotropic porous medium consisting of tortuous capillaries. Carman [17] later modified this equation by incorporating the specific pore surface area, resulting in:

\[ \kappa = \frac{D_f^2 \varepsilon^3}{16K_0(1-\varepsilon)^2} \]  

where \( D_f \) is the average fibre diameter (m) and \( K_0 \) is the Kozeny constant in the flow direction, which incorporates a shape factor and the tortuosity of the flow path [18].

Although the Carman–Kozeny equation is usually applied to predict permeability, discrepancies are often reported. As mentioned by Skartsis [19], the fact that \( K_0 \) is fairly constant for a practical range of porosities (\( \varepsilon \approx 0.4–0.7 \)) has made this equation very popular. However, the Kozeny constant, which has to be experimentally determined for real fibrous reinforcements, is a subject of controversy and different authors have reported very discrepant values for different fibrous materials, fibre arrangements, porosity values [20] and infiltrating fluids: \( K_0 = 0.1–0.8 \) [21], \( K_0 = 0.35–0.68 \) (longitudinal flow) and \( K_0 = 11 \) (perpendicular flow) [18], \( K_0 = 1.66–1.78 \) (longitudinal flow) and \( K_0 = 8.0 \) (transverse flow) [22], \( K_0 \) in the range of 0.8–6 [21], \( K_0 = 1.06 \) (longitudinal flow) and \( K_0 \approx 8 \) (transverse flow) [23], whereas Lindsay [6] and Muzzy [24] reported values in the range 3–7 and 7.6, respectively.

A liquid can be spontaneously transported (wicking) through the inter-fibre pores of the fibre preform by capillary action. This dynamic interaction process involves wetting, liquid uptake in the porous structure and, for some fibres, liquid absorption within the fibres, making the wetting measurements of fibrous assemblies more difficult to analyse [25].

Capillary pressure, \( P_c \), has been theoretically estimated using the Young–Laplace equation, which was first applied to idealised capillary tubes. A larger capillary pressure is predicted for fluid/fabric systems with a small contact angle. Furthermore, small pores should be filled first when the fabric is brought in contact with the liquid. As saturation proceeds, larger pores become filled and capillary pressure decreases, becoming equal to zero for a completely saturated medium [26]. In a reinforcing fibrous preform there is a distribution of pore sizes and shapes and distinct pore path directions and, hence, a dimensionless shape or form factor, \( F \), which depends on flow direction, has been used by Ahn [27] to merge all different anisotropic/geometric configurations of fibrous media. This relationship for one-dimensional resin flow is expressed by Eq. (4) below:

\[ P_c = \frac{F}{D_f} \left( \frac{1-\varepsilon}{\varepsilon} \right) \sigma \cos \theta \]  

where \( \sigma \) is the surface tension of the wetting fluid (Pa m) and \( \theta \) is the contact angle between the liquid and the solid. In the case of a unidirectional fibrous preform [21], \( F \) is considered 4 for axial flow and 2 for transverse
flow. For complex fibre alignment such as a woven fabric, \( F \) may be only determined indirectly from experimental evidence.

Different methods can be used to measure capillary pressure of a fibrous preform [26,28] but perhaps the most widely used are the experiments based on the Wilhelmy plate method [19,23,25]. Ahn [27] designed an apparatus to measure the factor \( F, P_c \) and transverse permeability of an assembly of plain-weave carbon-fibre fabrics. The main equation used was derived from the unidirectional form of the Darcy’s law for the constant pressure condition, which can be rearranged as:

\[
\frac{x^2}{\mu_e} = \frac{2\kappa}{\mu_e}(P_m + P_c) \frac{t}{l}
\]

where \( x \) is the resin penetration thickness into the fibrous preform and \( P_m \) is the applied mechanical pressure. The simultaneous determination of both \( \kappa \) and \( P_c \) in experimental studies is a very interesting possibility, and this method of determining \( P_c \) and \( \kappa \) was applied to the analysis of the RTM experiments in this study and will be further depicted later.

Thus, this study focuses on the determination of permeability and capillary pressure in the rectilinear infiltration of fibrous porous media in RTM. The scope of the experimental work is: (a) to apply a methodology to determine permeability and capillary pressure using fabric assemblies in an RTM set-up under different experimental conditions where the permeability value is decoupled from the capillary pressure and, therefore, to investigate the effect of capillary pressure in the measurement of permeability; and (b) to examine the effect of injection pressure, infiltrating fluid and saturation on the permeability.

3. Experimental

3.1. Materials

A plain-weave glass-fibre fabric was used for rectilinear RTM experiments as received from Fothergill Engineered Fabrics and its main properties, as supplied by the manufacturer, are shown in Table 1. This fabric has identical warp and weft yarns where each yarn contains three bundles twisted together, each of them made of continuous fibre filaments 9 \( \mu \)m in diameter. Although there is a slight difference between the number of picks and ends per square meter, this fabric is regarded as isotropic. The diameter of the coated fibre filament (\( \approx \)10.5 \( \mu \)m) was estimated by optical microscopy and dynamic contact angle (DCA) analysis. The fabric had a methacrylato chromic chloride (Volan) finish to enhance fibre compatibility to polyester, epoxide and vinylester systems.

![Micrograph of the cross-sectional area of an epoxy hardened warp fibre-yarn showing E-glass fibre filaments; magnification (20\( \times \)).](image-url)
that the viscosity was constant during the experiment. All experiments were conducted at room temperature.

The preform consisted of a different number of rectangular layers of fabrics carefully stacked following the same orientation of the fibres in relation to the mould. The rectilinear flow experiments were carried out in the warp direction and a new set of reinforcement was used in each experimental run, except for the experiments carried out with pre-wetted fabric.

The porosity for a single type of fibre reinforcement ($\varepsilon$) was estimated by Eq. (6):

$$\varepsilon = 1 - V_f = 1 - \frac{N_f a \rho_a}{H \rho_{glass}}$$

where $V_f$ is the fibre-volume fraction, $N_f a$ is the number of layers of fabric, $\rho_a$ is the areal density of the fabric, $H$ is the thickness of the mould and $\rho_{glass}$ is the density of E-glass. This equation does not take into account the presence of any voids within the moulded part.

3.2. Properties of the fluids

A few important properties of the infiltrating fluids were measured prior to experimental runs. These properties are described below and the values found are presented in Table 2.

3.2.1. Density

Density has been experimentally measured by weighing known volumes of liquid and an averaged result is presented in Table 2.

3.2.2. Viscosity

As suggested by analysing Darcy’s law [Eq. (1)], viscosity is one of the most important properties of fluids in the context of liquid moulding processes such as RTM. Viscosity measurements in this work were carried out using a syncro-electric concentric cylinder type Brookfield’s viscometer. Viscosity readings (usually at a frequency of 10 rpm) were taken before each RTM infiltration experiment. Both silicone oil and epoxy proved to be Newtonian fluids in the temperature range of interest to this work, since their viscosity remained constant (within ±5%) in experiments carried out at different shear rates (frequency from 1.5 to 12 rpm).

3.2.3. Surface tension

The relatively short length of time between the macroscopic impregnation of the preform and the rapid viscosity rise which accompanies the curing reaction is one of the limitations of liquid moulding processes compared to those based on pre-impregnated reinforcements. As a consequence, the time available for fibre wetting and development of a strong fibre-matrix interface is limited, making the understanding of the wetting process important to prevent a reduction of the mechanical performance of the composite [29].

A liquid is expected to wet and spread over a solid surface if the surface energy of the solid is lower than the surface energy of the liquid. Although there are other methods to measure surface tension [30], the DuNoüy ring tensiometer method, first described by Lecompte Du Noüy in 1919, is usually mentioned in the literature. Thus, a digital tensiometer was used to measure the maximum force which surface tension can exert on a wire ring (platinum-iridium in this work) as it is withdrawn from the surface of a liquid.

3.2.4. Contact angle

The wetting of the individual fibres is influenced by the surface energy of resins and fibres and is particularly important in high fibre volume fraction structures where capillary flow is an important impregnation mechanism [29]. A useful indication of the degree of wettability of the fibres is provided by the contact angle. A low contact angle implies a good wetting between the fibre and the liquid. According to Hsieh [31], surface wettability of any fabric containing a single fibre type is identical to the wettability of its constituent single fibres and therefore the values found for the single fibre filament were applied to the fabric. However, as mentioned by Steenkamer [5], despite the attractiveness of using the contact angle as a direct indication of the degree of wetting, $\theta$ is actually the result of three independent parameters, the surface tension of the solid/vapour, the solid/liquid and the liquid/vapour interfaces.

Contact angle values were determined by the single fibre pull-out test or the Wilhelmy plate method [23,31] using a dynamic contact angle analyser, where a balance measures the wetting force exerted on the fibre, $F_w$, when contact between the test fluid and the fibre occurs. In practical terms, the fibre is suspended on a balance and the fluid in a beaker is slowly (50 $\mu$m/s) moved upwards via a movable platform, which will eventually make the fibre contact the fluid surface; this experimental procedure produces a measurement of the advancing contact angle. The contact angle is given by $\cos \theta = F_w/(\phi \sigma)$, where $\phi$ is the perimeter of the fibre ($\sim 33 \mu$m) and has been determined by using a standard fluid, hexadecane, with known surface tension, $\sigma$, and low contact angle ($\theta < 20^\circ$). Contact angle can also be measured by visual observation of a droplet of liquid.

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Properties of the wetting fluids</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid</td>
<td>Density (kg/m$^3$)</td>
</tr>
<tr>
<td>Silicone oil</td>
<td>850–855</td>
</tr>
<tr>
<td>Epoxy resin</td>
<td>1130–1133</td>
</tr>
</tbody>
</table>
resting over a single fibre filament under the microscope [32].

3.3. RTM infiltration experiments

In this work, horizontal unidirectional laminar flow RTM experiments at constant pressure were carried out. The fluid was injected at the side gate of the mould, generally following rectilinear flow. In order to evaluate \( \kappa \) and \( P_c \), low injection pressures were used (less than \( 10^5 \) Pa) so that the contribution of the capillary pressure to the global flow would be considerable. A camera was used to monitor the progress of flow front in the mould.

The main features of the RTM equipment are discussed below.

3.3.1. Mould

A parallel-sided, flat rectangular composite mould with an inner cavity of 0.548 m × 0.352 m and a top 20 mm thick glass face, to enable observation of the flow during the injection stage, was used. A thick clamping frame was constructed to ensure minimum deformation. The mould permits rectilinear and radial flow experiments by using its side or central gate, respectively. Rubber rings and various metallic spacers were used to control the thickness of the mould and the flatness of the produced part. The mould was clamped with a set of four nuts/bolts tightened to a pre-determined clamping torque. Since the injection pressure never exceeded 1 bar \((1 \times 10^5 \) Pa) and the clamping pressure was low, mould deflection is expected to have been minimum.

3.3.2. Pressure transducers

Four pressure transducers of piezoresistive and gauge type (pressure range: 0–0.2 MPa) were installed in the base of the lower mould part at favourable positions to monitor the pressure of the rectilinear flow. A pressure transducer was also incorporated to the fluid injection line, close to the inlet gate. A computerised data acquisition system was used to assemble the pressure readings from the pressure transducers (one reading per second), display the results and store them on a PC. The PICOLOG data logging software converts the signal into pressure readings instantaneously, facilitating the control of the process. In this work, the pressure against time data, used in the estimation of permeability, was obtained either from the pressure transducer situated closer to the side injection gate, or from the transducer incorporated to the injection line, close to the inlet gate. The pressure transducers were calibrated with the aid of a pressure controller.

3.3.3. Pressure pot

This pot received air pressure from the pressure line and, as a result, the pressurised impregnating fluid stored was injected through a silicone tube to the chosen injection gate in the mould.

3.3.4. Manometer

Situated on the top of the pressure pot, it was initially responsible for controlling the injection pressure. The pre-set pressure varied in the range 15–90 kPa in the various runs. It was noticed throughout the experimental work, however, that there was an inherent inability of such equipment to be used to control the pressure. Besides, the accuracy of this manometer is of \( \pm 10 \) kPa, and therefore not suitable to be used to measure the pressure in the low-pressure experimental runs.

3.3.5. Pressure controller

A Druck sub-rack pneumatic digital pressure controller (accuracy of \( \pm 5 \) kPa) was used during the experiments. The air-line was connected to the controller, which had its outlet connected to the pressure pot. The valve of the pressure pot, in this new set-up, was kept fully open and therefore the pressure controller became responsible for controlling the injection of air to the pressure pot.

3.4. Characterisation of laminates

Composites were produced via RTM experiments to check the thickness of the laminate. Their innermost area was cut into nine sections about 450 mm long and 30 mm wide using a diamond saw for each laminate; thickness measurements were carried out with a micrometer at regular length steps so that a contour map of the thickness of the laminate could be produced in order to assess the flatness of the composite.

4. Analysis of the experimental data of the RTM infiltration experiments

The pressure term in the Darcy’s law may be expressed as shown in Eq. (7) [27]:

\[
\Delta P = P_m + P_g + P_v + P_c
\]

where \( \Delta P \) is the total pressure difference; \( P_m \) is the mechanical pressure or injection pressure; \( P_g \) is the gravitational pressure; \( P_v \) is the vacuum pressure and \( P_c \) is the capillary pressure.

Apart from the \( P_g \) term, which is considerably lower than the others in the majority of RTM cases, \( P_v \) and \( P_c \) may be comparable to the applied mechanical pressure. If vacuum-assisted resin injection is not being used, like in this work, \( P_v \) is also equal to zero, making Eq. (7) equal to \( \Delta P = P_m + P_c \). Eqs. (1) and (7) can then be combined to produce:

\[
\frac{dx}{dt} = \frac{\kappa (P_m + P_c)}{\mu \varepsilon} x
\]
Rearranging:

\[
\frac{\kappa}{\mu \varepsilon} \frac{dx}{dt} = \frac{\kappa}{\mu \varepsilon} P_m + \frac{\kappa}{\mu \varepsilon} P_c
\]  

(9)

or

\[
P_m = \frac{\mu \varepsilon}{\kappa} \frac{dx}{dt} - P_c
\]  

(10)

Hence, a plot of \( P_m \) against \((xdx/dt)\) would fit a straight line, with the intercept equal to \(-P_c\) and the slope \(\mu \varepsilon/\kappa\). The position of the flow front \((x)\) and the \( P_m \) against time data during the experiment were obtained from photographs and the readings of the transducers, respectively.

In practical terms, however, this method of estimating \( P_c \) and \( \mu \varepsilon \) proved to be troublesome due to the fact that the \( P_m \) tends to an asymptotic value (see Fig. 2), because of the constant injection pressure, invalidating the use of Eq. (10) after a variable period of time. Besides, \( P_m \) versus \( xdx/dt \) curves were very sensitive to localised errors in the experimental data, since \( dx/dt \) has to be determined for each experimental point.

Considering the viscosity of the fluid constant throughout the experiment, integration of Eq. (9) under constant injection pressure will produce an expression for the permeability of a dry preform due to mechanical and capillary pressure [Eq. (11)]. If the experimental conditions are such that \( P_c \) can be neglected when compared to \( P_m \), the second term on the right side of Eq. (11) is equal to zero, producing Eq. (12).

\[
\frac{x^2}{2t} = \frac{\kappa}{\mu \varepsilon} P_m + \frac{\kappa}{\mu \varepsilon} P_c
\]  

(11)

\[
x^2 = \frac{2\mu \varepsilon}{\kappa} P_m \ t
\]  

(12)

where \( x \) is the distance between the flow front and a reference point, which may be either the position of the pressure transducer (for when the \( P_m \) readings are taken by the transducer located at a certain distance from the gate) or the beginning of the reinforcement (in case the used transducer is located at the injection line, close to the inlet gate).

The use of Eq. (12) to estimate permeability of fibrous reinforcements via RTM experiments is referred to in the literature [22] and it has the advantageous side effect of providing a spatial average of the permeability and of minimising the effect of reading error.

In case the capillary pressure cannot be neglected when evaluating permeability, \( P_m \) data \((x\)-axis\) from different experiments at different injection pressures are plotted against \( x^2 \) \((y\)-axis\) for a certain period of time: according to Eq. (11), the data for the different experiments should lie on a best fitting straight line, from where the capillary pressure can be determined as the horizontal intercept axis value, since at that point \( P_c = -P_m \) (Fig. 7).

Thus, the use of Eq. (11) allows the determination of (a) \( \kappa \), from the slope of the \( x^2 \) versus time curve (Fig. 3) for constant pressure and porosity, and (b) capillary pressure, considering the variation of \( x^2 \) data for a certain period of time as a function of \( P_m \) for experiments carried out at different injection pressures.

![Fig. 2. Plot of the variation of the readings of the pressure transducer and the position of the flow front with time.](image-url)
5. Results and discussion

Fig. 2 presents typical data from a pressure transducer. As mentioned before, some time is required for the pressure to reach a plateau—maximum pressure value, which depends on the injection pressure and the position of the transducer in the mould. At the side injection gate, the pressure is very close to the injection pressure, $P_{inj}$. From the injection point, the pressure decreases linearly through the length of the flow, reaching $P_c$ at the flow front. The pressure in the transducers varies with time and the rate of variation in pressure decreases with time.

The transducers were calibrated with the help of the pressure controller and the manometer of the pressure pot. The readings of a pressure transducer located near the manometer of the pressure pot were found to vary linearly with the injection pressure reading in the manometer as presented in Fig. 4.

Photographs of the position of the flow as a function of time were taken at regular periods, in synchronicity with the time showed for the transducers. In order to calculate the permeability, $\kappa$, of the fibrous reinforcement, a formula derived from Darcy’s law [Eq. (12)] is used [22]. Thus, if the square of the position of the flow is plotted against time, the slope $(s)$ of the best fitting straight line is used according to Eq. (13) to evaluate $\kappa$.

\[
\kappa = \frac{\mu \Delta F}{2 \Delta P} \tag{13}
\]

Eq. (13) was derived for a constant pressure condition and, as Fig. 2 shows, some time is required for the pressure to reach a constant value, in this case 28400 Pa. In order to use Eq. (13) for a particular experiment, only the points in a valid period of flow can be used. If one observes Fig. 2, which also shows the variation of the position of the flow front for the same experiment, it can be noticed that only after the pressure stabilises, the term $x^2$ varies linearly with time. Therefore, in each experiment the range of valid points has to be identified before the linear fitting procedure can be used. Furthermore, the more pictures in the valid region of pressure data, the more the number of points to be used in the fitting procedure.

The concept of a constant pressure is subject of questioning. In this work, in order to identify the range of valid points, a fixed percentage of the maximum obtained pressure, usually around 96%, was utilised as $P_{inj}$.

5.1. Influence of the injection pressure on the evaluation of permeability

Three rectilinear RTM experiments with silicone oil were carried out for seven layers of plain-weave fabric well compressed into an average mould thickness of 3.4 mm, resulting in a porosity $\varepsilon = 0.56$. Fig. 5 shows the progress of the flow front with time for experiments 01, 02 and 03 carried out at pre-set injection pressures of 30, 20 and 15 kPa, respectively, in the pressure pot. As expected, the higher the injection pressure the faster the flow; besides, as the flow progresses, there is a decay in
the rate of increase of \(x\). This happens since the advance of the flow front varies according to \(\Delta P/\Delta x\) and, for a constant \(\Delta P\), this term decreases following the increase in \(x\) [see Eq. (1)].

Fig. 3 shows the variation of the distance square of the flow front for experiments 01, 02 and 03. Although the curves do not have the same starting point due to a distinct required time to reach different levels of pressure, this will not affect the calculations of permeability since a period of time \((\Delta t = 255 \text{ s})\) has been utilised instead of a fixed time after the start of the experiment, and therefore the slope for each experiment is the only important parameter from this figure. The slope of the best fitting straight lines for the linear portion of the \(x^2\) versus time curves for the different experiments increases at higher injection pressures.

Table 3 presents the findings for experiments 01, 02 and 03. If only \(P_{\text{inj}}\) is used in \(\Delta P\) to estimate permeability in Eq. (13), \(\kappa\) shows a decrease with the injection pressure, and experiment 03 seemed particularly discrepant, considering that all three experiments were carried out with the same number of layers of fabric at a constant thickness of the mould and therefore should show a constant permeability value. This happens because the
Table 3
Findings from experiments 01, 02 and 03, carried out at different injection pressures with silicone oil as the infiltrating liquid

<table>
<thead>
<tr>
<th>Experiment</th>
<th>$\varepsilon$</th>
<th>Filled length at 255 s (mm)</th>
<th>Pre-set pressure in pressure pot (Pa)</th>
<th>Reached pressure in transducer (Pa)</th>
<th>Permeability$^a$ (m$^2$)</th>
<th>Corrected permeability$^b$ (m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>01</td>
<td>0.56</td>
<td>61</td>
<td>30000</td>
<td>16240</td>
<td>$4.9\times10^{-10}$</td>
<td>$3.7\times10^{-10}$</td>
</tr>
<tr>
<td>02</td>
<td>0.56</td>
<td>30</td>
<td>20000</td>
<td>5685</td>
<td>$7.0\times10^{-10}$</td>
<td>$3.6\times10^{-10}$</td>
</tr>
<tr>
<td>03</td>
<td>0.56</td>
<td>16</td>
<td>15000</td>
<td>177</td>
<td>$1.2\times10^{-8}$</td>
<td>$3.8\times10^{-10}$</td>
</tr>
</tbody>
</table>

$^a$ $\Delta P = P_{inj}$ in Eq. (13).
$^b$ $\Delta P = P_{inj} + P_c$ in Eq. (13). $P_c$ values were determined as described in Section 5.2.

capillary pressure was not taken into account in the $\Delta P$ term when estimating $\kappa$. The lower is $P_{inj}$, the more misled will the calculation of $\kappa$ be, due to the increasing relative importance of $P_c$ in the $\Delta P$ term ($\Delta P = P_c + P_{inj}$). Alternatively, a corrected permeability value was calculated for $\Delta P = P_c + P_{inj}$ in Eq. (13) and the values of corrected permeability derived from the three experiments showed an excellent agreement. Therefore, the permeability proved not to depend on the injection pressure.

Another set of experiments was carried out at a lower porosity value ($\varepsilon = 0.47$) by using 10 layers of fabric. This new set of experiments produced $x$ versus time and $x^2$ versus time graphs similar to the ones previously presented in Figs. 5 and 3. Table 4 presents the findings for experiments 04 and 05 carried out at different injection pressures but for the same porosity. The results support the findings of the previous set of experiments, i.e. $\kappa$ shows a decrease with the injection pressure, the lower the $P_{inj}$, the more misled will the calculation of $\kappa$ be, and the corrected permeability derived from each of the two experiments showed an excellent agreement. Besides, as it was expected, the second set of experiments, with a lower porosity, showed a lower permeability.

In order to evaluate the mould thickness, a composite was produced for each thickness value and its innermost area was cut into nine sections about 450 mm long and 30 mm wide. As presented in Fig. 6 for the case of 10 layers laminate, the thickness of the composite showed some variation especially at the edges of the mould, which were invariably thinner than its innermost part due to the localised clamping at the edges. An average value of 4.0 mm ($\pm 0.1$ mm) was found in this case.

5.2. Estimation of capillary pressure via RTM permeability experiments

The long experimental time necessary for capillary experiments to give proper estimation of $P_c$ and permeability [33] drove the investigation into a method of measuring these properties through RTM experiments. This requires a few experiments at the same conditions but different $P_{inj}$ to estimate the actual $P_c$ value of the fibrous preform. This procedure, described below, makes the determination of $P_c$ not only much faster than capillary experiments but also easier, since no extra apparatus is needed to carry out the estimations apart from the RTM equipment already used for mouldings.

According to Eq. (11), the permeability for constant pressure and porosity as well as the capillary pressure can be estimated considering the variation of $x^2$ data for a certain period of time (255 s, in this case) for experiments carried out at different $P_{inj}$ [5]. In Fig. 7, the term $x_2^2 - x_1^2$ was plotted as a function of $P_m$, where $x_2$ is the position of flow front, $x_1$ is the position of the pressure transducer in the mould near the side gate and $P_m$ is the pressure at 255 s measured by the pressure transducer at $x_1$ for each experiment. As can be seen in Fig. 7, the data for the different experiments lie onto a best fitting straight line, where the capillary pressure was determined as the horizontal axis intercept value ($x_2^2 - x_1^2 = 0$), where $x_2^2 - x_1^2$ is the variation of the position of the flow front for a period of time of 255 s and therefore, at that point $P_c = -P_m$, in this case, $P_c = 5505$ Pa (for $\varepsilon = 0.56$). The form factor for the longitudinal impregnation of the plain woven fabric was calculated according to the Young–Laplace equation [Eq. (4)] for $\varepsilon = 0.56$, $\sigma = 21 \times 10^{-3}$ N/m and $\theta = 23^\circ$. Thus, $F$ was

Table 4
Findings from experiments 04 and 05, carried out at different injection pressures with silicone oil as the infiltrating liquid

<table>
<thead>
<tr>
<th>Experiment</th>
<th>$\varepsilon$</th>
<th>Filled length at 255 s (mm)</th>
<th>Pre-set pressure in pressure pot (Pa)</th>
<th>Reached pressure in transducer (Pa)</th>
<th>Permeability (m$^2$)</th>
<th>Corrected permeability (m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>04</td>
<td>0.47</td>
<td>9.5</td>
<td>30000</td>
<td>16310</td>
<td>$6.3\times10^{-11}$</td>
<td>$4.7\times10^{-11}$</td>
</tr>
<tr>
<td>05</td>
<td>0.47</td>
<td>7</td>
<td>25000</td>
<td>10010</td>
<td>$7.7\times10^{-11}$</td>
<td>$4.8\times10^{-11}$</td>
</tr>
</tbody>
</table>
calculated as \( F = 3.81 \), an intermediate value between 2 and 4 but close to 4.

Similarly, for the second set of experiments \( P_c \) was calculated as \( P_c = 7957 \) Pa (for \( \varepsilon = 0.47 \)). Hence, the lower porosity fibrous preform (\( \varepsilon = 0.47 \)) displayed a higher \( P_c \) as expected from the Young–Laplace equation. Although only two experiments were carried out for this porosity, the calculated form factor \( F = 3.79 \) is also in the expected range, between 2 and 4, very close to the one calculated for the first set of experiments. The \( P_c \) value determined in these experiments for the plain-weave fabric takes into account micro capillary pressure within the fibre yarns and macro capillary pressure in the pores between the fibre yarns. The micro capillary pressure is of course expected to be dominant.

The range of variation of \( F \) is between 2 for a totally transverse unidirectional flow, and 4 for a totally longitudinal unidirectional flow. The estimated value of \( F = 3.8 \) in this study demonstrates that in this fabric assembly longitudinal micro-flow within the fibre yarns is the dominant term in determining \( F \). Some divergence reported in the literature about the values of the form
factor $F$ should be mentioned at this point. Not only this value is reported to present variations [5], but also the contribution of each flow (flow within fibre yarns, inter-yarn and inter-layer flow) to the total flow is unknown, changing the form factor. Furthermore, the applicability of this equation to model experimental data for the capillary flow through a fabric in RTM has also some uncertainty [23].

The $P_c$ values found via RTM experiments can not be directly compared to the values obtained by capillary experiments of silicone oil through a single layer of fabric [33] because of differences in porosity. Therefore, in order to compare both experiments, a procedure was applied using the concept of normalised capillary pressure ($P^*_c$), as shown in Eq. (14), which depends only on the fluid and the reinforcement but not on the porosity of the reinforcement.

$$P^*_c = \frac{P_c}{(1 - \varepsilon)} = \frac{F}{D_f \sigma \cos \theta}$$

The $P^*_c$ values for the capillary (one layer and $\varepsilon = 0.38$) and RTM experiments with seven ($\varepsilon = 0.56$) and 10 layers ($\varepsilon = 0.47$) of fabric, were 7122, 7035 and 6972 Pa, respectively. The small difference in these values may be attributed to interlayers and inter-yarn spacing which lower both $P_c$ and $F$ since the flow is not necessarily parallel to the fibres. Alternatively, small variations of surface finish in different batches of fabrics, influencing $\sigma$ and $\cos \theta$, might also be responsible for this small variation.

It is important to mention here that the RTM experiments were carried out within a limited range of pressure and porosity and, although the findings seem to be consistent to the currently accepted knowledge in this area, deviations can be expected, for instance, for highly packed or highly porous fibrous beds.

### 5.3. Influence of the saturation of the fabric on the permeability

Experiments 04 and 06 were carried out under similar conditions apart from the fact that in experiment 06 the fibrous preform was pre-wetted in order to analyse the variation in permeability. Fig. 8 shows the variation of the squared distance of the flow-front for both experiments and, considering that other factors were constant, an increase in the permeability of the pre-wetted fabric is immediately noticed by the slope of the best fitting straight line. Table 5 presents the calculated values for permeability where a threefold increase in permeability can be found for the pre-wetted fabric. The change in permeability for pre-wetted fabrics is in agreement with the reviewed literature [4,34].

The increase in permeability does not appear to be in anyway related to the injection pressure. Fig. 9 shows the readings from the pressure transducer for experiments 04 and 06 and it is clear that the profile curves for both experiments are practically coincident except for the final part of the curve which displays a relative shift of less than 5%, related to fine adjustment of the valve of the pressure pot. Besides, it has already been concluded that $P_{inj}$ is not expected to alter permeability values.

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**Fig. 8.** Plot of the variation of the distance square of the flow front as a function of time for experiments 04 (●) and 06 (○) where the preset injection pressure is 30 kPa in both cases.
Experiments 01 and 07 carried out at a higher porosity also showed an increase in permeability for the pre-wetted fabric. However, this increase did not exceed 52% of the original value. This can be explained by the fact that the micro-flow has a relatively greater importance for fibrous preforms with a lower porosity. In fact, for highly packed fibrous beds (low porosity) or for low injection pressure, the micro-flow will be crucial in determining the behaviour of the flow.

5.4. Influence of the test fluid on the estimation of permeability

Estimations of permeability via RTM experiments with seven layers of plain-weave fabric (ε = 0.56) were also carried out with an epoxy resin without the curing agent (μ = 1.7 Pa s, σ = 44 × 10⁻³ N/m and θ = 57.1°) for different injection pressures (experiments 08, 09 and 10). The results, summarised in Table 6, display the

![Fig. 9. Plot of the variation of the readings of the pressure transducer for experiments 04 and 06.](image)

<table>
<thead>
<tr>
<th>Table 5</th>
<th>Findings from experiments 01, 04, 06 and 07, carried out at different injection pressures with silicone oil as the infiltrating liquid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Experiment</td>
<td>ε</td>
</tr>
<tr>
<td>01 (dry)</td>
<td>0.47</td>
</tr>
<tr>
<td>06 (wet)</td>
<td>0.47</td>
</tr>
<tr>
<td>01 (dry)</td>
<td>0.56</td>
</tr>
<tr>
<td>07 (wet)b</td>
<td>0.56</td>
</tr>
</tbody>
</table>

a Corrected permeability is not applicable since a pre-wetted fabric is presumed saturated and therefore does not display capillary pressure.
b This experiment has been carried out with a higher viscosity silicone oil (μ ≈ 0.22 Pa s).
c A higher P_inj is not supposed to affect the determination of permeability.

<table>
<thead>
<tr>
<th>Table 6</th>
<th>Findings from experiments 08, 09 and 10, carried out at different injection pressures with epoxy as the infiltrating liquid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Experiment</td>
<td>ε</td>
</tr>
<tr>
<td>08</td>
<td>0.56</td>
</tr>
<tr>
<td>09</td>
<td>0.56</td>
</tr>
<tr>
<td>10</td>
<td>0.56</td>
</tr>
</tbody>
</table>
same features found for the silicone oil experiments. The epoxy resin experiments were much slower than those with silicone oil due to the large difference in viscosity. The most important finding from this table is, however, that the corrected permeability values obtained with the epoxy resin \( (k \approx 3.6 \times 10^{-10} \text{ m}^2) \), by taking into account \( P_c \) in \( \Delta P \), were very close to the ones found for the silicone oil \( (k \approx 3.7 \times 10^{-10} \text{ m}^2) \), Table 3. The small difference between these values is within the expected range of experimental error and therefore cannot be attributed to the properties of the fluids. Hence, the permeability of the fibrous preform does not seem to depend on the permeating fluid.

The capillary pressure for the epoxy resin experiments was again determined as the horizontal axis intercept value in Fig. 10, as \( P_c = 6841 \text{ Pa} \) and the form factor in the Young–Laplace equation for the impregnation of the RTM assembly of plain woven fabrics was calculated as \( F = 3.84 \). This value is very close to the previously calculated value from the RTM experiments with silicone oil \( (F = 3.79–3.82) \), proving to be independent of the infiltrating fluid. This could be expected since \( F \) is a property of the specific fabric with its characteristic distribution of warp and weft yarns and particular structure.

5.5. Analysis of edge effects in RTM

Analysis of the first RTM experiments carried out in this study showed flow front perturbation, or edge effects. In other words, the flow velocity in the channel between the porous medium and the mould edge was much more developed than the bulk flow, inside the porous reinforcement. Given to their importance in rectilinear flow experiments, edge effects have been studied experimentally and also taken into account in computer simulations of the mould filling process. Two main factors are known to cause this adverse flow, high porosity at the edge region and taping of the edges.

Firstly, tape was used to avoid fabric fraying during cutting and/or subsequent use of the layer but it seemed that the tape was leading the flow. So, one experiment was conducted with tape in only one side and, indeed, the side with the tape produced a larger edge effect. This supposition was further confirmed by capillary experiments [35], which also exhibited an irregular flow front in the early experiments with fabrics with tapered edges. So the use of tape was omitted for the subsequent experiments. Parnas [4] also mentioned the influence of the tape on fluid flow behaviour and on the measurement of permeability. In their case, to minimise edge effects, a tape of only 1.6 mm width was used.

Secondly, the flow will occur more readily if the porosity is higher. In fact, it was noticed that the manual cutting produced layers of different widths, that will occasionally be responsible for gaps (or regions with higher porosity) between the edges of the layers (or some of them) and the inner walls of the mould. Gauvin [14] mentioned that even gaps as narrow as 1 or 2 mm can be responsible for edge effects.

A few approaches were tried to avoid edge effects, like using narrow extra layers or thin rolls of fabrics in the edges, but they proved to produce rather variable results, such as complete blocking of the flow or irregular flow in the layers, even because the thickness of the mould near these regions would be affected. Therefore, the best way found to overcome these difficulties

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Fig. 10. Variation of the epoxy resin infiltration in a period of 255s as a function of the applied injection pressure for experiments 08 (●), 09 (■) and 10 (▲), where the preset injection pressures are 40, 70 and 90 kPa, respectively.
was to employ layers of fabric purposely cut slightly wider than the mould cavity and, prior to their placement in the mould, to remove the tape. Also, silicone tubes were used to redefine the edges inside the mould. Although this is not an ideal approach, if there is any, it proved to be effective, and it was used in all RTM experiments described here. Nowadays, moulds are equipped with a dynamic primary resin seal which uses an external vacuum and pressure controller to suit varying cavity thickness and injection pressure, allowing a better control of edge effects; nevertheless, even then the use of a fabric width higher than the width of the mould is still recommended.

6. Conclusions

Rectilinear infiltration experiments with assemblies of a plain-weave fabric in an RTM set-up at various low injection pressures were carried out to evaluate permeability and capillary pressure of the woven fibre preform. This methodology of measuring the capillary pressure of assemblies of woven fabrics avoids the problem of excessively long equilibrium times required in capillary experiments for the determination of capillary pressure and provides the global capillary pressure present in the RTM process, incorporating all micro- and macro-effects.

Regarding the estimation of permeability it has been shown that the higher the injection pressure, the lower the associated error of not including $P_c$ into the permeability calculation [by using Eq. (12) instead of Eq. (11)]. When a corrected permeability value was calculated taking into account the capillary pressure effect, values from different experiments showed excellent agreement and the permeability proved to be independent of the injection pressure. These findings were further confirmed by two sets of experiments at different porosities.

The experimental analysis of the flow measurements was also able to estimate $P_c$ for the assembly of plain-woven fabrics. A form factor, $F$, for this fabric being infiltrated by silicone oil or epoxy was calculated in the range of $F = 3.79 - 3.84$, demonstrating a dominant micro-flow along the fibre direction. A normalised capillary pressure, $\overline{P}_c$, was used to compare the $P_c$ values found at different porosities. The $\overline{P}_c$ values from RTM experiments with seven and 10 layers of fabric and also the $\overline{P}_c$ value estimated from the capillary micro-flow through a single fabric layer showed a negligible variation, which was attributed mainly to interlayer and inter-yarn flow.

An increase in permeability was found for pre-wetted fabrics in comparison to dry fabrics. A smaller increase was found for RTM experiments at a higher porosity, which was explained by the fact that the capillary micro-flow has a relatively greater importance for fibrous preforms with a lower porosity. Estimations of corrected permeability values with the epoxy resin and the silicone oil used alternatively as infiltrating liquids were similar and, thus, the permeability of the fibrous preform was found to be independent of the permeating fluid.

It is important to mention here that the RTM experiments were carried out within a limited range of pressure and porosity and although the findings seem to be consistent with the currently accepted knowledge in this area deviations can be expected, for instance, for highly packed or highly porous fibrous media. Nevertheless, it is a conclusion of this work that in cases where the main goal to be achieved by RTM experiments is the determination of the permeability of the fibrous preform, experimental runs at a relatively higher $P_{inj}$ should be preferable since this minimises the influence of the usual lack of $P_c$ data on the estimations. In this case, no correction of the determined permeability is needed incorporating $P_c$ and Darcy’s law excluding $P_c$ may be used in the determination of permeability.

In the case of low injection pressures, $P_c$ needs to be included in Darcy’s law to determine the correct permeability value which should be the same as the permeability determined at higher injection pressures, provided that no fabric deformation has taken place in the latter case. RTM simulations for the progress of the global flow front should be based on the correct value of permeability, and should include the capillary pressure if the RTM injection pressure is low.

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References


