

Methods for permeability measurements of fibrous reinforced preforms

Métodos para determinar la permeabilidad de preformas reforzantes fibrosas

Iván Patiño, Carlos Vargas, Juan Vanegas*

Grupos Materiales Avanzados y Energía, MATyER, Instituto Tecnológico Metropolitano. Campus Robledo, Portería Principal: Calle 73 No 76A - 354 Vía al Volador. Medellín, Colombia.

(Received June 05, 2013; accepted March 31, 2014)

Abstract

Permeability is a property used to measure how a liquid flows through porous media. This property defines how a mold is filled in Liquid Composite Molding (LCM). It is important to know the different methods used to measure permeability (scopes, advantages and disadvantages) since in some cases it is possible to obtain significant errors during the measurements. Therefore it is important to compare theoretical methods by experimental measurements. In this work, a review about different techniques used in the literature to determine the permeability of reinforcement materials was done. The review was done in order to provide a reference for future study and research in the field of processing and simulation of liquid composites molding reinforced with preforms.

-----**Keywords:** Permeability, preforms, permeability measurements, liquid composite molding

Resumen

La permeabilidad es la propiedad que determina la facilidad con la que un líquido fluye a través de un medio poroso. Esta variable determina, en el caso de procesos de moldeo líquido de compuestos (*Liquid Composites Molding, LCM* por sus siglas en Inglés), el patrón de llenado de moldes. Es importante conocer los diferentes métodos de medición de permeabilidad, sus alcances, ventajas y desventajas, ya que en algunos casos se pueden inducir errores considerables en la medición, por lo que es adecuado realizar una validación entre los métodos experimentales y teóricos. El presente artículo realiza una revisión y análisis de las diferentes técnicas establecidas para determinar la

* Corresponding author: Carlos Vargas, e-mail: carlosvargas@itm.edu.co; phone number: + 574 4600727 ext. 5636 (C. Vargas)

permeabilidad de preformas reforzantes, con el fin de presentar una referencia para posteriores estudios e investigaciones en el campo de la simulación y procesamiento de resinas reforzadas con preformas.

-----**Palabras clave:** Permeabilidad, preformas reforzantes, medición de permeabilidad, moldeo líquido de compuestos

Introduction

Fibrous reinforced preforms

The term fibrous reinforced preform is referred to the part of the composite material that provides mechanical resistance and stiffness, and that is impregnated by the resin when a LCM process is employed for the manufacturing of the part. The most common preforms used in LCM are composed of inorganic materials, such as glass, carbon and aramid. The fibrous reinforced preforms have many types of geometrical configurations and its architecture is the main variable affecting the permeability.

In general terms, the reinforced preforms used in LCM processes can be classified into single scale preforms and dual scale preforms (Figure 1). Single scale preforms are the ones in which the difference among the permeability inside the bundles (micro-permeability) and the permeability in the gaps (macro-permeability) is not relevant in any situation. In double scale preforms, there are some circumstances where that difference could be significant, depending on many factors, such as the fiber volume fraction and the size of the bundles. In general, as ϕ is higher and/or the size of gaps between tows is smaller, the behavior of the preform tends to be like a single-scale preform, contrary to what happen when ϕ is lower and/or the size of gaps is greater [1,2], where the preform behaves like a dual scale preform and the differences between the micro-flow (flow inside the yarns) and the macro-flow (flow in the gaps) could be notorious.

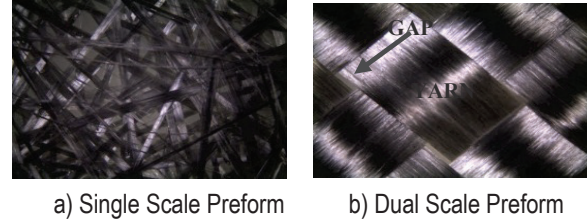


Figure 1 Single and Double scale preforms

Darcy law

Permeability can be defined as the easiness of a liquid to flow through porous media. This property was initially studied experimentally in 1855 by French engineer Henry Darcy, who measured the water volume (Q) that crossed by time unit, through a saturated column of sand of length (L) and cross sectional area (A), when a difference of hydrostatic pressure (H) was applied between two points [3].

The most known form of Darcy's law in one direction, equation 1, can be described as [4]:

$$(\bar{V}_j)_f = -\frac{K^{(j)}}{\mu} \frac{\partial}{\partial x_j} [(\bar{p})_f]^f \quad (1)$$

Where:

$K^{(j)}$: Permeability of porous medium at direction j . [m^2].

$(\bar{V}_j)_f$: Fluid phase volume average of velocity of injected flow at j direction. [m/s]

μ : Dynamic viscosity of the resin. [kg/ms]

$[(\bar{p})_f]^f$: Fluid intrinsic volume average of injection pressure. [N/m^2]

In porous media all properties are given in terms of averages in a Representative Element Volume

(REV). If the average is referred to the fluid phase volume of the REV, the following nomenclature is used: $(\bar{N})_f$ when it is taken the value of the property, N , in all phases and it is averaged regarding to the fluid phase volume, and $[(\bar{N})_f]^f$ when the value is taken only in the fluid phase and then averaged regarding to the same fluid phase.

Since fibrous reinforcements are ideally considered as porous medium, the permeability is the basic property to establish the parameters of the injection process in close molding such as injection pressure, location of injection points, vacuum assistance and compaction force, among others. It is also one of the most important parameters to select the type and the properties of preforms and the polymeric resin used in the injection process. Summarizing, the most important reasons to consider the permeability as the fundamental property in injection processes are the following:

- Permeability describes the behavior of the resin flowing through fibrous reinforcement at different orientations and that behavior is very important to design the preforming of parts manufactured by Resin Transfer Molding (RTM) [5-7].
- Permeability is the most important input parameter to simulate the flow front advancement using numeric methods or by means of specialized software for RTM. In other words, most of the simulation methods and software of filling of molds require the permeability of the preforms positioned in the cavity as an input parameter [8-10].

Permeability tensor

Equation 1 describes the behavior of the flow of resin in most of the injection processes using closed molds. In this equation, the permeability value (K) is different at directions X, Y, Z (K_{xx} , K_{yy} , K_{zz}) [11], which coincide with principal flow axes, but it can be expressed in terms of crossed permeability (K_{xy} , K_{yx} , K_{xz} , K_{zx} , K_{yz} , K_{zy}). Then, Darcy's law can be written to consider these terms as shown below in equation 2:

$$V_j = -\frac{1}{\mu} K_{ji} \frac{\partial P}{\partial x_i} \quad (2)$$

Where K_{ji} is the permeability tensor of the perform (Equation 3), such that:

$$K_{ji} = \begin{bmatrix} K_{xx} & K_{xy} & K_{xz} \\ K_{yx} & K_{yy} & K_{yz} \\ K_{zx} & K_{zy} & K_{zz} \end{bmatrix} \quad (3)$$

In this case, in order to simplify the volume-averaged notation, equations 4 and 5 consider that:

$$V_j = (\bar{V}_j)_f \quad (4)$$

$$P = [(\bar{p})_f]^f \quad (5)$$

For plane permeability in XY plane, equation 6 expresses the permeability tensor as:

$$K_{ji} = \begin{bmatrix} K_{xx} & K_{xy} \\ K_{yx} & K_{yy} \end{bmatrix} \quad (6)$$

In any preform of homogenous geometry, there could be identified main axes of permeability, corresponding to perpendicular axes where the permeability K_1 is the maximum, and K_2 , the minimum. Then the permeability tensor in the direction of the axes (Equation 6) is simplified (Equation 7) such that:

$$K_{ji} = \begin{bmatrix} K_1 & 0 \\ 0 & K_2 \end{bmatrix} \quad (7)$$

According to that, Darcy's law (Equation 2) at main directions of permeability, in two dimensions can be written as (Equation 8):

$$\begin{bmatrix} V_1 \\ V_2 \end{bmatrix} = -\frac{1}{\mu} \begin{bmatrix} K_1 & 0 \\ 0 & K_2 \end{bmatrix} \begin{bmatrix} \frac{\partial P}{\partial x_1} \\ \frac{\partial P}{\partial x_2} \end{bmatrix} \quad (8)$$

Parameters affecting permeability

Permeability has area units [m^2]. It is a fundamental property to describe and simulate

the impregnation phenomenon of preforms [12-14]; this phenomenon is mainly affected by parameters such as: viscosity and capillarity of resin and geometry of preforms during the injection (type of preform, compaction pressure, deformation of preform). A more detailed description of those parameters is presented in literature [15]. The impregnation of the reinforced preforms depends on previously described variables, however, permeability, as a property, depends only on the preform's geometry during injection; nonetheless, the experimental value of this property differs when it is measured in unsaturated or saturated tests, due to the capillary properties effects (contact angle and surface energy) between the preform and the fluid used during the experiment [16].

Permeability measurements of reinforced preforms include two kind of activities: implementation of permeability tests and the use of an adequate mathematic algorithm during the test. There are several permeability tests and its adequate selection for each application depends mainly on: injection pressure during the test, capillarity pressure of preforms (compared with injection pressure), required precision and viability to establish permeability, complexity to control the measuring process, mold instrumentation and data acquisition, and material of preform and impregnated liquid [17,18].

Methods and research to measure permeability

Measurements of permeability of preforms can be classified into two main groups: experimental methods and non-experimental methods. However, permeability measurements are not performed exclusively by just one of those type of methods, and it is better to use a combination of different techniques available for each method. A classification and summary of different methods used to determine the permeability of preforms are shown in figure 2.

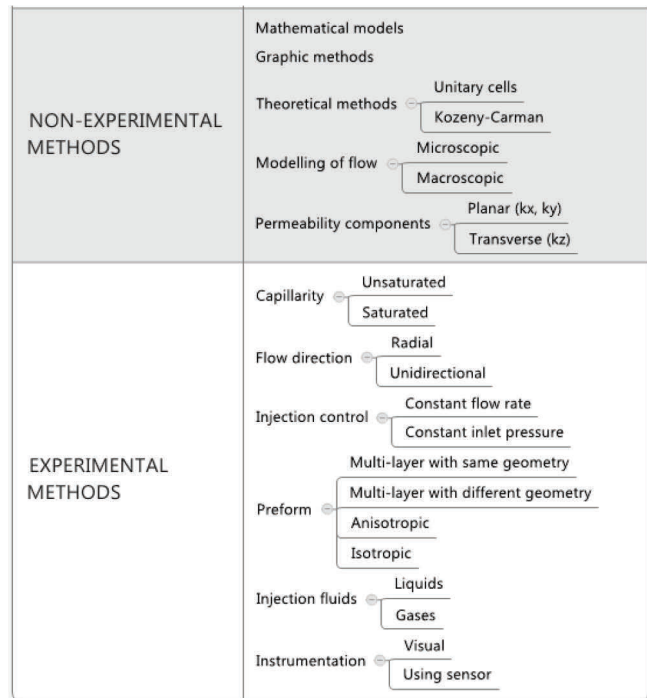


Figure 2 Methods to measure permeability of fibrous reinforced preforms

Experimental measurements require time-consuming procedures and in some cases discrepancies can be obtained as it had been presented in some studies [19]. It is a common practice to validate different techniques by using numerical methods, analytical methods or using a reference preform as a pattern of measurement [8].

Capillary effects during impregnation of preforms: Saturated and unsaturated methods

When a saturated method is used to measure the permeability, the measurements are done once the preform is completely impregnated by the liquid, while in unsaturated methods, the measurements are done while the liquid impregnates the preform (Figure 3).

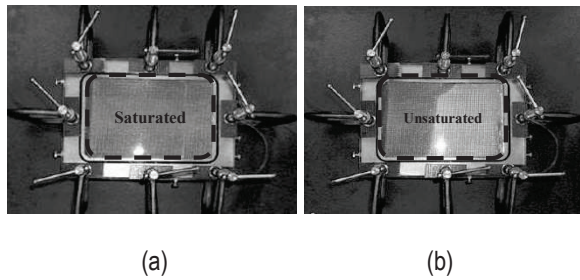


Figure 3 Measure permeability: (a) Saturated, (b) Unsaturated [5]

When a saturated test is performed, the pressure drop between two points is related only to injection pressure, and the mathematical model is based on equation 9:

$$V_x = -\frac{K_{sat}}{\mu} \frac{\Delta P}{L} \quad (9)$$

Where:

V_x : Velocity of flow in the direction of injection. [m/s]

K_{sat} : Permeability of the preform obtained in a saturated test. [m²]

ΔP : Pressure drop between two points of preform. The pressure drop is measured based on injection pressure. [N/m²]

L : Distance between the two points where pressure drop is measured. [m]

However, in unsaturated tests the pressure drop (measured between two points) has one component related to the injection pressure, ΔP , and another one to capillary pressure, P_c . Accordingly to the aforementioned, Darcy's law is written for unsaturated tests as (Equation 10):

$$V_j = -\frac{(K_j)_{ins}}{\mu} \cdot \left(\frac{\Delta P - P_c}{L} \right)_j, \text{ being } j=1, 2. \quad (10)$$

Where:

V_j : Flow velocity in direction j. [m/s]

$(K_j)_{ins}$: Permeability of the preform in direction "j" obtained in unsaturated tests. [m²]

ΔP : Pressure drop between two points of the preform in direction j, related to the injection pressure. [N/m²]

L : Distance between the two points where pressure drop is measured in direction j. [m]

P_c : Capillary pressure in direction j of the preform. [N/m²]

Unsaturated tests of permeability are only accurate when the capillary pressure (P_c) is negligible compared to the injection pressure. If P_c is not negligible, it is possible to quantify the capillary effects by measuring the total pressure drop between two points of flow ($\Delta P_{total} = \Delta P - P_c$), in order to estimate the capillary pressure based on mathematical models, or by measuring simultaneously the capillary pressure and the permeability of preform [20]. The main advantage of the unsaturated tests is that the user is able to know and obtain the data of the flow front progression of injected liquid in the preform. Moreover, it is important to establish relationships between the permeability of preforms at different orientations and their geometry. In saturated tests, it is not important to know the position of the flow front since it does not have any influence on the final measurement.

A method to measure the permeability by using a saturated test had been developed [21]. In this case, the permeability at different fiber volume fractions (V_f), from only one sample preform, is measured in a continuous experiment. Another study presented an experimental setup to measure the permeability of isotropic preforms at different fiber volume fractions (20 to 50 %) in only one test, by using a radial flow on a completely saturated preform [22]. Similar investigations have been proposed to measure permeability while the preforms are continuously compacted [23-27]. Some studies make a comparison between measurements in saturated and unsaturated regime using a same material commonly used in industry [28, 29]. Measurements of planar permeability have been reported using the ratio (K_{unsat}/K_{sat}) of unsaturated permeability (K_{unsat}) and saturated permeability (K_{sat}) with values

close to 0.7 for some strand mats and 0.4 for some oriented fabrics [30]. Some planar permeability measurements have been obtained by three different methods: two in saturated regime and the other, in unsaturated one. The authors did not report significant differences among both regimes [17]. On the other hand, different studies have concluded that the measured permeability across the thickness for preforms of fiberglass is lower in saturated regime for ratios $K_{\text{unsat}}/K_{\text{sat}}$ between 1.5 to 4 [9, 13, 31]. However, the results are closely related to fabric materials and fluids used to perform the test. Therefore, there is still no agreement among researchers between the measurements performed in saturated and unsaturated regimes. Regarding to the permeability across the thickness, there are only a few studies due to the problems to track the flow front progression in that sort of tests [13]. In addition to that, only a few studies in saturated regime have been performed especially with unidirectional flow techniques [31-34]. In a recent study [35], the authors presented a test rig with optical fibers to detect the flow front progression, and the possibility to evaluate the permeability in saturated or unsaturated regime across the thickness. The results showed that permeability value depended on the technique used to measure flow front progression. In this case, the ratio ($K_{\text{unsat}}/K_{\text{sat}}$) was controlled from 8 to 10.

A method named “Through-Thickness Unsaturated Permeability” (TTUP) was implemented, measuring the capillarity pressure from infiltration velocity in preforms of carbon fibers and glass fibers [36]. The measured permeability using this method is in agreement with Kozeny- Carman and modified Gutowski models, the Carman–Kozeny equation and Gebart’s model. However, for greater than 60%, the permeability value did not show significant differences for the evaluated preforms.

Injection control: constant flow rate or constant inlet pressure methods

During injection control, when the permeability is measured using a constant flow, the pressure of

injection machine increases when the flow front is moving forward. The pressure is increased to keep constant the injected liquid volume during the test.

A more detailed description of both methods (constant flow and contact pressure) can be found in different researches [18, 37]. Another relevant feature in those methods is the change of total pressure between two points in the flow front of injected liquid. [38, 10]. In those cases, the injection pressure, the capillarity pressure, the pressure due to body forces and the vacuum pressure must be taken into account during the measurements.

Flow direction: Unidirectional and radial methods

The permeability at several relative orientations between the flow and the preform can be measured using unidirectional and radial methods. In unidirectional test, the liquid is injected from the border of the preform, while in radial tests, the liquid is injected from the center of the preform. Unidirectional tests allow measuring the permeability of preforms in only one direction by each experiment. In one radial test [39-42], it is possible to determine the permeability values of the preform at different orientations. However, the data can be obtained only if the geometry of preform is homogenous.

A more detailed description of unidirectional methods can be found in [17, 18]. The unidirectional method is affected by irregularities of flow front progression, particularly in zones close to injection point, where the injection pressure is higher [5]. It is also possible the formation of race-tracking zones (RTZ). RTZ are zones where the injected liquid flows preferentially by open channels because of damaged edges during the cutting operation, deformation or incorrect fitting of preform’s edges in the walls of the mold.

On the other hand, radial methods are prone to form flow’s irregularities at the mold inlet and in the flow front progression. Accordingly, a

bigger quantity of radial tests could be required in order to accurately determine the permeability of reinforced preforms [43].

Some studies allowed to integrate radial methods and constant inlet pressure, and they were pioneers to measure the planar permeability of preforms using the mentioned methods [44]. Other studies have developed an experimental setup and a mathematical model based on previous works about porous media [45], to determine the permeability of anisotropic preforms. Their main contribution was the introduction of Equivalent Isotropic System (EIS) to represent flow fronts of anisotropic preforms in a quasi-isotropic coordinate system in order to calculate and taking into account the positions of the flow front.

Regarding unidirectional methods to measure permeability, some results have showed that unidirectional methods have better repeatability than those obtained by radial methods [46]. Thus, by gaining repeatability, the main disadvantage of unidirectional methods over radials tests (a larger number of tests required to measure the permeability of anisotropic preforms) is overcome.

After different works in radial methods, another authors have performed radial tests at constant flow rate and established a theoretical-experimental model to calculate the anisotropy grade of regular preforms and based on that, the main permeabilities of anisotropic preforms, K_1 and K_2 , were calculated by mean of simple iterative method [47,48]. Simultaneously, others measurements of permeability were performed in woven preforms trying to compare radial and unidirectional unsaturated methods [49]. The capillary effects have been measured in permeability tests of woven preforms, these researches have permitted obtain a mathematical model to calculate the capillary pressure as a function of geometrical characteristics of preform and capillarity properties the of injected liquid [20]. Later, Another model to calculate capillary pressure in reinforced preforms was presented [50].

During the mid-nineties, Carter and others proposed a graphical method to validate data obtained from radial tests, based mainly in four graphics: normalized radius vs Angle, ratio a/b vs. Number of isochronous, R_y vs. R_x and $f(R_x\sqrt{S_1})$ vs T [51], where R_x and R_y are the flow front radii in the major and minor directions, respectively. The method is valid if the suppositions governing the mathematical models of radial test are satisfied during the actual test. Contemporarily, a comparative study between radial and unidirectional tests had been published [18]. This study compared permeability values of certain preforms obtained by two types of tests: unsaturated unidirectional at constant flow rate and radial at constant flow rate. The main conclusions of the study were the following: the permeability measurements are seriously affected by increasing the flow rate beyond a certain limit, confirming that Darcy's law is not valid above certain values of Reynolds number, and radial tests conducted at constant flow showed to be more sensitive to injection flow rate than unidirectional tests.

A mathematical model has been proposed and improved to calculate permeability from unidirectional tests, named the Concurrent Procedure for Measurement of Permeability model (CPMP model) [52].

The main contributions of this model are: 1) it allows calculating permeability in three ways (elemental, punctual and interpolated) and 2) suggests two parameters to improve the test reliability (minimum length of preform and maximum injection pressure).

A device consisting of four cavities of unidirectional flow to measure simultaneously the different directions of perform had been manufactured [53], eliminating the main disadvantage of typical unidirectional methods. In radial methods, it was a common practice to measure the positions of flow front only in the main directions of permeability. However, a mathematical model was proposed to determine the permeability of anisotropic preforms from

measurements of flow front in three different directions, 0°, 45°, and 90°, and no one of them had to coincide with some of the main directions of permeability [40]. The main contribution of this method is that it allows doing several radial tests for the same preform, in different orientations of the flow in order to minimize the error.

In a recent study using radial methods [54], the authors measured the permeability using analytical models, and they used the Adams model [44] and numerical and experimental validations with the purpose to show the relevant advantages of radial method compared with unidirectional method. The results were used as standard models for the permeability calculation and they showed the advantages of radial methods, being some of them: less complicated experimental setup, accurate measurements of permeability tensor in its three components and low sensitivity to errors caused for channeling effects.

Calibration of injection fluids

From the beginning of the implementation of planar permeability tests, several issues were observed when thermoset resins were used. Resins are very expensive to perform real-scale tests, and most of them are toxic to human beings because of volatile emissions. In addition to that, resins attack acrylic making them unsuitable for radial and unidirectional unsaturated tests.

Accordingly, researchers tried to use other injection liquids in order to calibrate properly and reduce the cost of the tests. Some works took into account the effect of the injected liquid using water, corn syrup, motor oil and Dow 200 and it was found that the type of liquid affected the permeability measurements [55]. Six years later, another researchers found that the permeability of reinforced preforms did not change significantly when the injection fluid was modified. The results showed that three important requirements shall be met to make the homologation of liquids in permeability tests: same resin viscosity, similar capillarity properties and similar rheological behavior. The researchers used water, corn syrup

and epoxy resin [56]. Later, a research group performed several tests using two liquids with dissimilar capillary properties: silicone oil with good wettability with the fiberglass and honey-corn, having low wettability on fiberglass. This study concluded that at low V_f , the permeability measurements were not affected by the capillary properties of the injection liquid, probably due to the low capillarity pressure [57]. Corn syrup and silicone oil have been used currently to perform permeability tests, but glycerin has been widely used recently because of its purity, low cost and because its viscosity can be easily modified when it is mixed with water [10, 58].

There are other techniques using gases or air to measure the permeability [59-64]. The main advantage of using gases compared with liquids is that the measurements are performed quickly and the preform is not destroyed since it is not wetted. Another studies used air with a unsaturated radial methods in preforms with random orientation, compared the measurements with the ones done with liquids and found similar results [7]. On the other hand, other researches showed that the permeability value depends on the fluid for unsaturated method [16].

Equipment and experimental setup used during permeability measurements

Regarding to the equipment, experimental setup and data acquisition systems, there are important contributions in the last decade. Most of mathematical models used to calculate permeability by constant inlet pressure assume that the pressure drop in the distance is linear. Accordingly, only one pressure transducer is required in the injection point in the mold and a vacuum transducer if a vacuum line is used during the test. The assumption represents a good approach of the variation of the liquid's pressure whether the velocity is low and the capillary effects are negligible. For permeability tests at low injection flow rate and when the V_f is low, the assumption is valid and the tests can

be performed using only one pressure transducer. However, some authors use more transducers to validate the assumption of linear change of pressure or just to calculate the pressure drop based on the experimental measurements. When the V_f is increased, the capillary effects are more significant and the linear assumption is not valid anymore, generating inaccuracies during the tests. For V_f higher than 45% , it is convenient to use more pressure transducers to measure the pressure in several points in order to account for the capillarity pressure. Being aware of this phenomena, researchers leaded by Ken Han proposed a radial visual method to measure the permeability of anisotropic preforms at high V_f using four transducers carefully located in the mold [65].

Another important contribution in molding data acquisition systems is the PIERS method (Permeability Identification Using Electrical Resistance Sensors) [42]. The most important characteristics of PIERS is that it is not a visual method and a continuous visualization of the flow front is not required since the location of that flow front in different directions is sensed by electrical sensor located inside the cavity of the mold. The advantage of this method is that it eliminates the error by visual inspection of the flow front and the top plate of the mold can be made of a stiffer material. If the sensors are calibrated and their response time is adequate, the repeatability is better than in visual methods. However, since the sensors are expensive, they demand calibration and the method itself does not allow the observation of the flow patterns, it has not been massively implemented. In addition to that, the use of PIERS method with conductive fibers such as Carbon fibers is restricted.

Mathematical models used to calculate permeability

Regarding the mathematical models used to calculate permeability from the tests, they could be mentioned two significant contributions in the last decade. The first important contribution

is the iterative ellipse-specific fitting [43]. The model allows calculating the permeability ellipse of a determined preform by using the values measured at two or three random orientations by unidirectional tests. The mathematical model of the iterative ellipse-specific fitting method is related to the permeability CPMP model, which was already aforementioned in this work.

The second contribution is the Mixed Numerical Experimental Technique (*MNET*) [66]. The method is related to PIERS method and it measures the time required for the liquid to achieve the sensor position in the plate. After that, a finite element simulation using an adaptive mesh is performed. The permeability value is obtained by iteration until there is an acceptable agreement between the arrival time to every sensor of the PIERS method and the time obtained by the simulation.

Concerning to the mathematical models of permeability of multi-layer preforms, a conceptual model for interlayer flow was proposed [67], based on the hydraulic radius theory, that quantifies the effect of interlayer micro-structure on the effective permeability of multilayer fabric preforms.

A numerical-experimental method to predict the 3D permeability tensor was proposed too, based on the location and the time taken to achieve the position of the flow front in the mold [68]. Later on, it was proposed a numerical optimization by using a least square method between experimental data and predicted permeability using a commercial RTM simulation software.

When the permeability is measured and an important deformation of V_f [23-27] takes place, it is important to evaluate the effect of irregular V_f and the fiber velocity due to the deformation, during the measurements. In this case, the experimental verification is laborious. However, by using numerical simulation of preforms deformation and measurements of resin flow at mesoscopic and microscopic scales, it is possible to obtain a good prediction of the fiber velocity due to its deformation. [69-71]. Although,

simultaneous simulation of preforms deformation and resin flow is a hard task since there is no mutual influence between the flow-induced preform deformation and the changes in the microstructure of the preform. In order to solve this issue, another numerical alternatives were presented [72, 73] based on mass conservation and force equilibrium at macroscopic scale.

On the other hand, there are several important numerical models in the literature to estimate the permeability, where the Stokes and Brinkman equations are solved using a finite element model to calculate the permeability [74,75]. In 2D simulations for unidirectional fibers some authors have studied the transversal permeability in random fibers using boundary elements methods, and the concept of biperiodic domains to predict the permeability in elements with fictitious domain was implemented employing a finite element method [76-79].

In 3D simulations, a meso-scaled flow in channels between fibers had been modeled to investigate the local distribution of permeability in non-crimp fabrics using the effect of transversal fibers and some geometrical aspects [80,81]. The authors reported that the local permeability is mainly influenced by the geometrical characteristics imposed during fabrics manufacturing. However, they reported that external modifications did not affect the local permeability. Furthermore, some works consider coupled flow in zones between the fibers and the channels in order to predict the permeability or to simulate the filling process [82-86]. In a recent work, a 3D-finite- element technique was developed to be applied in fluids flowing through porous volume with fibers inside the microstructure [87], the authors reported satisfactory results using an average criterion of macroscopic permeability for balanced fabrics used as reinforcements. However, for complex 3D structures (three orthogonal fibers), the average criterion to evaluate permeability is not satisfactory. Finally, other relevant mathematical model is a statistical method developed to evaluate chopped strand mats by using digital image processing obtained from several samples

of this cloth and by coupling two permeability measurement techniques: Kozeny–Carman combined with the fiber volume fraction and density probability functions [88].

Theoretical model

There are two theoretical methods [89]: unitary cells and the one derived from Kozeny–Carman equation. In the former, a representative model of the fabric is modeled, boundary conditions are defined and the model is solved using basic conservation laws for the fluid. Although it is a complex method, there are several important contributions in unidirectional and bidirectional preforms [90, 91]. The second method is derived from Kozeny–Carman equation [92, 93]. The most important assumption is that the injected fluid flows through a preform as it was flowing through tortuous capillary tubes (i.e those where the capillary channels do not have a straight and longitudinal orientation). The model calculates the permeability as a function of the fiber's radius and the tortuosity of the preform. The tortuosity of the preform is experimentally determined and depends on the fiber arrangement and their packing level. The results are acceptable when the preform is unidirectional and the fluid flows in the longitudinal directions of the fibers. Besides, some authors studied some preforms with the liquid flowing in the transversal direction of the fibers [12], but the experimental setup used to measure the tortuosity is complex.

Other studies have been carried out by using theoretical permeability in idealized preforms as a perfectly organized package [14, 94] and others, were performed using disordered fiber arrays [76].

Preforms impregnation models

There are important discrepancies in permeability values obtained by theoretical and experimental methods [95]. The differences can be explained because most of the theoretical models do not include the effect of microscopic flow during impregnation. It is known that complex

preforms induce a double-flow behavior: microscopic and macroscopic flow [57, 96-100]. Bubble entrapment in preforms [101] is caused by the imbalances between macroscopic and microscopic flow. Since capillary flow is important in Liquid Composite Molding (LCM) because it is preponderant inside the bundles at high V_f 's, several studies have been focused to the determination of the capillary pressure. The dynamic variation of capillary pressure in LCM [102] and the influence of the capillary pressure on the permeability of fiberglass fabrics have been reported [38] and recently, the capillary effect on fiber's saturation during resin infusion has been modeled [103], by adding a capillary pressure estimated theoretically using a flow simulation.

Permeability according to preforms and preforms arrangement

In many applications of close molding in composite materials, several kind of preforms are located in a cavity, with different orientations. In those cases, the permeability of each layer is different and the permeability analysis more complex than in the case when a single preform is used.

There are several mathematical models to describe the permeability of multi-layers arrangements depending on the permeability of every layer. The first model is called the averaged-permeability [104] and its most important physical assumption is that transversal flow is negligible. Thereof, the results are acceptable only when the permeability of different layers is very similar. By modeling compacting pressure, it is possible to calculate the multi-layer permeability affected by average permeability, the thickness of combinations of preforms and the fiber volume fraction. The thickness of the combination of preforms and the fiber volume fraction are determined by a pressure compaction test in a universal testing machine and by using analytical methods. In the equivalent-thickness method, it is assumed that the preform has an equivalent thickness as

if it was completely solid and made only of the reinforcement fibers. Under this assumption, the permeability is calculated by using the average permeability method.

The effective permeability includes the transverse flow between layers and predicts the flow front in every preform comprising the multi-layer arrangement. In the category of effective permeability models there is a traditional model [105] based on the Darcy law. It is assumed that the pressure in the flow front of every preform changes linearly from the injection pressure to atmospheric pressure (whether the pressure is measured according to manometric pressure).

Another model using multi-layer permeability take into account the transversal flow as well as the longitudinal flow, in the interface among layers [67]. Other studies have analyzed and compared the average permeability models and effective permeability multi-layer and in some cases, when two types or orientations of similar preforms (same thickness) were used, the results of two models were equivalent [9, 67, 105]. Other authors have developed an analytical model to predict the location of the flow front in multi-layer preforms [106]. This model includes the contribution of transverse flow among adjacent layers to total flow through preform. In the model, it is demonstrated that effective permeability (analytically calculated) is more accurate than the average model and it must be added to the model when they are present transverse flows through adjacent layers and when the transverse permeability is high. However, this analytical model underestimates the transverse flow among adjacent layers when it is compared with numerical methods and when the transverse permeability is low.

A pioneer work was performed about permeability measurements using a preform arrangement. In that work, the behavior of fibrous reinforced preforms is analyzed with average permeability models in multi-layer arrangement [31]. Later on, another work proposed multi-layer arrangement permeability model called effective permeability.

This model is an enhanced method compared with average permeability model and its more important contribution is the prediction of flow front for every layer of the arrangement, even if the their geometries are considerably different [105].

In case of dual scale preforms, where it is important to distinguish the flow in the channels and flow inside fibers [29, 107-111], some researches had been done, in order to calculate the micro-permeability of fibers and the total permeability of preform from pressure profile measurement at mold inlet [96] and by using a model based in the physics of flow derived from Darcy's law [112, 113]. There are available models in the literature treating the total permeability of the real micro-structure of preforms, as fractal geometric models [114, 115].

Since permeability has an important variation depending on geometry preform (diameter and length of fibers), some researches have studied those parameters and they proved that when larger diameters and longer fibers are used, the permeability is increased [116].

Conclusions

The permeability is the most important property to study the impregnation phenomenon in fiber reinforced preforms used in Liquid Composite Molding (LCM) and its correct measurement is crucial in order to perform numerical simulations of the flow front advancement in the filling of molds. In spite of the impregnation phenomenon is highly dependent of the material properties of the resin and the fiber (viscosity, contact angle, superficial tension, among others) and of the architecture of the reinforcement during the injection, most authors agree that permeability, as a property, depends only on the last parameter. Taking into account that premise and the assumption of a darcian flow impregnating the fibrous reinforcement, there have been proposed several methods to determine the permeability of fiber preforms.

In general, as it was exposed in the present work, the permeability methods can be classified into non-experimental and experimental ones. In the non-experimental methods, some theoretical methods are used to estimate the permeability taking into account a fully saturated domain (Unitary cell and Kozeny Carman); other works, that account for imbalances between the macroflow and microflow in dual scale preforms and its influence in the effective permeability, have also been proposed. Nevertheless, the experimental methods are by far the most used ones and they comprise an experimental setup and a mathematical model. In this regard, the experimental setup of each method results from a combination of three types of classifications: classification according to capillary effects (saturated and unsaturated), according to the flow regime (constant pressure and constant flow) and according to the direction of flow (unidirectional and radial); then, the mathematical model of the method is established taking into account such a combination. After the survey conducted in the present work, they can be highlighted three experimental methods (two by their frequency of use by many authors and one, by their accuracy), with their corresponding mathematical models, namely: the Unidirectional unsaturated CPMP+Ellipse-iterative fitting, Radial unsaturated + Weitzenbock and Radial Sensorized PIERS+MNET.

Other key issues mentioned in this review were: the influence of the liquid in the permeability measurements and the determination of the filling patterns in arrangements where the layers have different permeabilities. Regarding to the first topic, it shall be mentioned that, in spite of several researches have been carried out to analyze the influence of the liquid in the permeability of the preforms, this is not yet a scientific closed matter due to the complexity of the relationship between the capillary and viscous phenomena with the different architecture of the preforms. The topic of the impregnation of multilayer arrangement has been mainly tackled using averaged-permeability and effective permeability models, but a deeper

insight into the phenomena that arise along the interfaces among the layers is still required in order to obtain more reliable models that accounts for such a phenomena considering the in-planar, transverse and inter-layer permeabilities of each layer.

Finally, it is relevant to mention that, in spite of the notorious developments in the measurement of permeability of fibrous reinforced preforms employed in LCM processes, important aspects shall be still considered in order to improve the agreement with real situations of filling of molds. Many of those aspects are referred to the dual-scale nature of many reinforced fabrics and some of them are: capillary effects, imbalances of flow inside the preform, formation of voids by mechanical entrapment, among others; those aspects cause that the global permeability determined by some traditional methods mentioned here does not produced the actual infiltration pattern in many applications. Thus, future works of permeability determination will be addressed to the study of those effects in dual-scale preforms in order to improve the characterization of the permeability of that kind of fibrous reinforcements.

Acknowledgement

The authors acknowledge to the Research Center of INSTITUTO TECNOLÓGICO METROPOLITANO of Medellín, Colombia for its financial support to the researchers involved in this work.

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