

A QUANTITATIVE EVALUATION OF THE UNCERTAINTY OF PERMEABILITY MEASUREMENTS IN CONSTANT THICKNESS FIBRE REINFORCEMENTS (RTM)

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Keywords: Resin flow permeability, uncertainty, liquid composite moulding (LCM)

Abstract

The experimental difficulties and lack of a standard approach for permeability determination, has meant that permeability values determined by laboratories with differing set-ups have produced contradictory data. This fact has led to a number of permeability benchmark exercises worldwide, the latest of which has proposed a jointly developed common experimental method in order to foster data agreement[1,2,3]. Nonetheless, even with these results, some issues still remain regarding our understanding of the fundamentals of variability and uncertainty behind permeability experiments. This study presents an evaluation of the uncertainties produced by measuring instruments and equipment in permeability determination for LCM. The uncertainty measurements were quantified for unsaturated fibres in terms of device accuracy and performance limitations. Preliminary results reveal that both pressure and porosity parameters have significant influence on permeability measurements. It is our view that the proposed quantitative uncertainty analysis has three main contributions to the state of the art: (1) that these results can be used to optimize experimental set-ups and reduce data scattering in future experiments; (2) that the methodology can be used to provide better quality data for stochastic analysis of flow through porous media; (3) that the method can be applied to existing data / experimental set-ups in a retrospective way. Finally, the authors feel that the proposed analysis can allow a better understanding of the reasons that may lead to contradictory results between different laboratories testing the same materials.

1. Introduction

1.1. Permeability

Liquid Composite Moulding (LCM) is a generic name for a family of manufacturing processes for composite parts, in which a stack of dry fibre layers is placed in a mould cavity where a thermoset resin is injected to impregnate preform. During the mould-filling step, resin (wetting fluid) flows to wet individual fibres, fill fibrous preform, and air is pushed (non-wetting fluid) out from the mould cavity. Newton's law governs the relationship between fluid flow and pressure gradient inside the fibrous preform and is manifested as Navier–Stokes or Darcy's (Eq.1) equations:

$$\bar{u} = \frac{K}{\mu} \cdot \nabla p \quad (1)$$

Darcy's equation relates the volume averaged fluid velocity \bar{u} to the pressure gradients ∇p , the fluid viscosity μ , and the preform permeability tensor K . In simulation of mould filling process, the permeability directly affects filling time and flow pattern. An accurate determination of permeability is therefore vital for reliable simulations [4,5,6]. Several empirical models have been proposed to characterize fibrous preform; for example, the Kozeny [7] formed an empirical model by coupling capillary effects and the Darcy's law. Carman [8] defined a variable S , the specific surface exposed to fluid, on Kozeny's equation [9]. However, while these models have been used extensively by researchers (and validated for different porous media) there is a substantial disagreement on the real permeability measurements for media consisting of reinforcement fibres. Also, in their normal form, these models do not convey the stochastic (probabilistic) nature of permeability which may demand a great number of experiments required for the better estimation of permeability (Hoes et al [10]). Permeability is determined through indirect measurements and estimations of cavity thickness, fibre bed porosity, injection pressure, flow front position, etc. Therefore, in addition to the non-deterministic nature of permeability, these other uncertainties also interfere with the permeability results.

Early work on the measurement uncertainty of fibrous preform permeability was done by Dong [11] who determined significant parameters, pressure and viscosity, affecting on 1D-permeability measurement through linear flow experiment. Sriramula et al. [12] and Mesogitis et al. [13] reviewed causes and effects of uncertainty in stochastic analysis and manufacturing process of fibrous composites, respectively. This study aims to extend this by presenting an uncertainty analysis for in-plane permeability as a result of device accuracy and performance limitations. To this end, sources of uncertainty that cause variations in composite quality were identified. The uncertainty analysis of unsaturated measurement was then obtained.

1.2. Sources of uncertainty in composite manufacturing process

Permeability is not directly determined but calculated via several other measured values (see e.g. [14]). Hence, the measurement is subjected to uncertainties resulting from the different sources. In this case we must consider four major classes of error sources (1) Instrument uncertainty: this includes viscometer, pressure gage, beaker, spider, ruler, and caliper. (2) The second class of uncertainty originates is the material uncertainty where we considered four

major types of errors sources. (2.1) edge effects or racetracking during 1-D flow experiment, which means resin takes the path of least resistance caused by imperfect fit of the fibrous preform at mould walls. Racetracking can induce errors as high as 100% in permeability measurement. This effect is reduced by using a transparent top mould for visual inspection or flow sensing. (2.2) Second error source results from the entrapment of air: in some cases such air entrapments could introduce very high errors (possibly larger than 50%) in permeability measurement. (2.3) Third, mould deformation due to high fluid pressure could therefore change the preform structure, thickness and architecture: i.e. both permeability and fibre volume fraction are affected by the deformation of the mould. Neglecting this can cause errors in process analysis and uncertainty in measurement. (2.4) A fourth error source was attributed to heterogeneity (different shapes and fibre orientations) of fibrous preform, leading to a large variation in permeability measurement. (3) Environmental uncertainty: error sources can originate in a any number of workplace instabilities, including variation of temperature and humidity, which will affect resin mobility. (4) Human factor related uncertainty. Without a systematic procedures and training, human factors can introduce significant uncertainties can lead to large variations in permeability measurement.

2. Uncertainty analysis

To characterize the uncertainty of a measurand F that is a function of independent variables X_1, \dots, X_n :

$$F(X_1, \dots, X_n) \quad (2)$$

It is combined with a standard uncertainty, $U(F)$, is used, and expressed as:

$$U^2(F) = \sum_{i=1}^n \left[\left(\frac{\partial F}{\partial X_i} \right)^2 u^2(X_i) \right] \quad (3)$$

Where $u(X_i)$ is the standard uncertainty of variable X_i :

$$u^2(X_i) = \frac{s^2(X_i)}{N} + u_e^2(X_i) \quad (4)$$

Where $s^2(X_i)$ is variance (square of standard deviation) of X_i , N is number of measurements of X_i and $u_e(X_i)$ is standard uncertainty of X_i due to measurement system. Standard deviation itself is expressed as:

$$s^2(X_i) = \frac{1}{N-1} \sum_{j=1}^n (X_{ij} - \bar{X}_i)^2 \quad (5)$$

Where X_i is value of each of the N measurements and \bar{X}_i is mean value of the N measurements.

2.1. Uncertainty propagation in a radial injection for permeability determination

Combining an integration form of Dracy’s law and mass conservation equation yields in-plane permeability, K. K is a function of flow time instant (t), fluid flow front position(r) at t, radius of injection tube (r_{inj}), injection pressure(P_{inj}), viscosity(μ), and porosity (φ) :

$$K = \frac{r^2 \left(2 \cdot \ln \frac{r}{r_{inj}} - 1 \right) + r_{inj}^2}{4 \cdot t \cdot P_{inj}} \mu \phi \quad (6)$$

According to equation (3), the combined standard uncertainty of permeability for each instant of time can be written as:

$$U^2(K) = \left(\frac{\partial K}{\partial t} \right)^2 u^2(t) + \left(\frac{\partial K}{\partial r} \right)^2 u^2(r) + \left(\frac{\partial K}{\partial r_{inj}} \right)^2 u^2(r_{inj}) + \left(\frac{\partial K}{\partial P_{inj}} \right)^2 u^2(P_{inj}) + \left(\frac{\partial K}{\partial \mu} \right)^2 u^2(\mu) + \left(\frac{\partial K}{\partial \phi} \right)^2 u^2(\phi) \quad (7)$$

Since all of the variables except porosity are obtained by single measurements, their variances are non-existent. The standard uncertainty of these variables is therefore determined only by the uncertainty due to the measuring system used. Thus equation 4 can be recast as

$$u^2(X_i) = u_e^2(X_i) \quad (8)$$

2.2. An example of the calculation of uncertainty caused by measurement systems

Figure.1 shows the distribution of the permeability measurement results at thicknesses of 2.01, 1.71, and 1.45, from [15] and confirms a large variation / scater in the results.

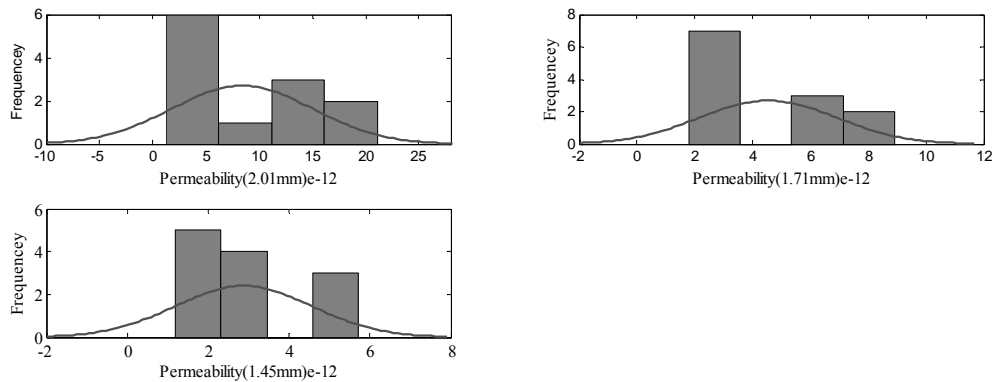


Figure 1. Distribution of permeability measurement at different thicknesses.

Considering the uncertainty propagation discussed above one can study the different contributions to this scatter from its constituents:

2.2.1 Time instant, t

t was measured by Spider 8-30 multichannel system. The system has an error of 0.05%. Therefore, the uncertainty is:

$$u_e(t) = t \cdot \frac{0.05}{100} (s) \quad (9)$$

2.2.2. Flow front position, r

The ruler that was used for r measurement has an uncertainty equals to half of the smallest division (1mm) on:

$$u_e(r) = \frac{0.001}{2} (m) = 5 \times 10^{-4} m \quad (10)$$

2.2.3. Radius of injection tube, r_{inj}

In measuring r_{inj} , a caliper with 0.02mm least count was used. Therefore, the uncertainty is:

$$u_e(r_{inj}) = \frac{2 \times 10^{-5}}{2} (m) = 1 \times 10^{-5} m \quad (11)$$

2.2.4. Injection Pressure, P_{inj}

The injection pressure used in reference [15] was 4.2×10^5 Pa. Measurement instruments for the pressure were Spider 8-30 multichannel system with an error of 0.05% and sensors Keller PR-21S with an error of 1%. Therefore, the uncertainty is:

$$u_e(P_{inj}) = 4.2 \times 10^5 \times \frac{1 + 0.05}{100} (Pa) = 4.4 \times 10^3 Pa \quad (12)$$

2.2.5. Fluid viscosity, μ

A viscometer with resolution of 0.1 mPa.s was used [15]. Therefore, the uncertainty is:

$$u_e(\mu) = \frac{1 \times 10^{-4}}{2} (Pa.s) = 5 \times 10^{-5} Pa.s \quad (13)$$

2.2.6. Standard uncertainty of porosity, ϕ

ϕ is a function that can be formulated as

$$\phi = 1 - V_f = 1 - \frac{M_f / \rho_f}{V_{cav}} = 1 - \frac{M_f}{\pi(r_{cav}^2 - r_{inj}^2)h_{esp}\rho_f} \quad (14)$$

Where M_f is the reinforcement mass, ρ_f the fibre density, r_{cav} is radius of mould cavity, r_{inj} is radius of injection tube and h_{esp} is height of the spacers. As for the standard uncertainty of the porosity, this quantity is not measured but calculated indirectly. From the propagation of uncertainty principal, the squared combined standard uncertainty of ϕ can be expressed:

$$u^2(\phi) = \left(\frac{\partial\phi}{M_f}\right)^2 u^2(M_f) + \left(\frac{\partial\phi}{\rho_f}\right)^2 u^2(\rho_f) + \left(\frac{\partial\phi}{r_{cav}}\right)^2 u^2(r_{cav}) + \left(\frac{\partial\phi}{r_{inj}}\right)^2 u^2(r_{inj}) + \left(\frac{\partial\phi}{h_{esp}}\right)^2 u^2(h_{esp}) \quad (15)$$

2.3. Standard combined uncertainty of permeability, K

To quantify the uncertainty of the permeability, first each of the partial derivatives given in equation (7) is obtained and then the values of the partial derivatives are substituted in equation (7).

3. Significant parameters

To determine which of the parameters have significant influence on accuracy of permeability measurements, the relative standard deviations of variables are calculated by recasting equation (7):

$$U_K^2 = \left(r \frac{\partial K}{\partial r}\right) \left(\frac{U_r}{r}\right)^2 + \left(r_{inj} \frac{\partial K}{\partial r_{inj}}\right) \left(\frac{U_{r_{inj}}}{r_{inj}}\right)^2 + \left(\mu \frac{\partial K}{\partial \mu}\right) \left(\frac{U_\mu}{\mu}\right)^2 + \left(P_{inj} \frac{\partial K}{\partial P_{inj}}\right) \left(\frac{U_{P_{inj}}}{P_{inj}}\right)^2 + \left(\phi \frac{\partial K}{\partial \phi}\right) \left(\frac{U_\phi}{\phi}\right)^2 + \left(t \frac{\partial K}{\partial t}\right) \left(\frac{U_t}{t}\right)^2 \quad (16)$$

And since

$$\left(r \frac{\partial K}{\partial r}\right) + \left(r_{inj} \frac{\partial K}{\partial r_{inj}}\right) = \left(\mu \frac{\partial K}{\partial \mu}\right) = \left(P_{inj} \frac{\partial K}{\partial P_{inj}}\right) = \left(\phi \frac{\partial K}{\partial \phi}\right) = \left(t \frac{\partial K}{\partial t}\right) = K \quad (17)$$

The relative standard deviation can be expressed:

$$U_K^{*2} = U_r^{*2} + U_{r_{inj}}^{*2} + U_\mu^{*2} + U_{P_{inj}}^{*2} + U_\phi^{*2} + U_t^{*2} \quad (18)$$

The relative standard deviation of the parameters is shown in figure.2. From this, it is revealed that pressure and porosity measurement errors account for 96% of the relative standard uncertainty on average. The uncertainties of pressure and porosity measurement therefore require attention so that the in-plane permeability results are improved.

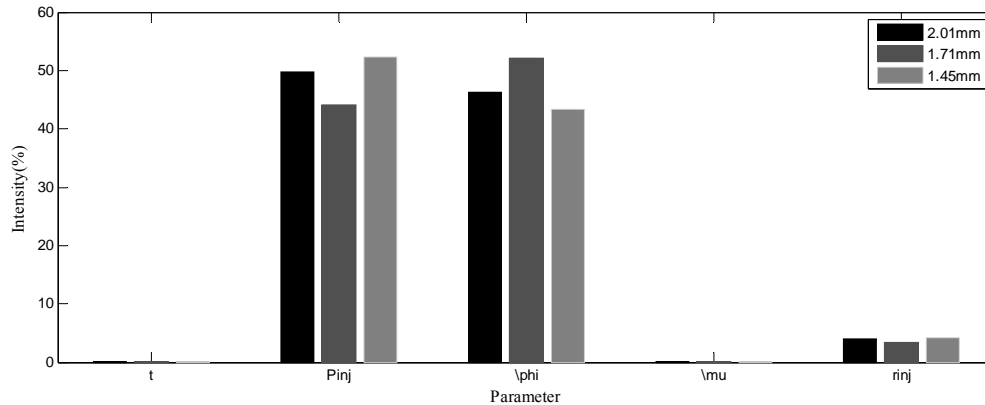


Figure 2. Relative standard deviation of parameters at different spacer thicknesses for permeability.

As pressure is directly measured by a pressure meter, uncertainty will be reduced by using a pressure meter with higher accuracy. Regarding porosity, by evaluating relative standard uncertainty, it was found out that radius of mould cavity (r_{cav}) is a significant parameter in porosity calculation. A precise measurement of r_{cav} should be done by precision sensors.

4. Conclusion

This study presented an uncertainty analysis for 1D in-plane flow permeability measurement. The sources of uncertainty were discussed and individual parameter uncertainties were estimated versus device accuracy and performance limitations. Their influence on the permeability uncertainty was analysed by uncertainty propagation. The relative uncertainty was also calculated to identify the significant parameters. By evaluating uncertainty of in-plane permeability results, both pressure and porosity were identified to have the biggest influences on combined standard uncertainty of permeability. To decrease variability in permeability results, a great attention should be paid to the significant parameters. It should be noted that porosity is a function of some variables, radius of mould cavity (r_{cav}) among which introduced the highest degree of uncertainty. This uncertainty evaluation approach can be applied to the determination of the quality of permeability measurements obtained by different experimental setups. Furthermore, the proposed method can be utilized in many aspects of composite manufacturing processes, such as providing inputs (e.g. edge effects or racetracking, mould deformation, air entrapment, curing and fibre compaction) for stochastic simulation.

Acknowledgements

The authors would like to acknowledge support of the Portuguese Innovation Agency, within the frame of Project newFACE, contract N. 23213 of the Program of Incentives to Technological Research & Development. The authors would also like to acknowledge support from the Portuguese Foundation for Science and Technology, under the research Grant SFRH/BD/51578/2012 and the MIT-Portugal Program.

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