EXPERIMENTAL INVESTIGATION OF CAPILLARY FLOW POROMETRY FOR CHARACTERIZATION OF DUAL SCALE POROSITY IN FIBROUS REINFORCEMENTS

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Introduction

Engineering textiles used in composite materials can be considered as porous materials because of the open spaces between the fibers in a bundle and between the woven bundles themselves. These spaces are commonly referred to as microscopic and mesoscopic pores. The dual scale porosity of fibrous reinforcements is of great importance when studying *Liquid Composite Molding* (LCM) processes because the pore size distribution directly influences the resin flow and fabric impregnation during the injection stage. For example, Vernet and Trochu [1] used optical microscopy to investigate experimentally the mesoscopic pore size distribution in 3D interlock fabrics in order to validate a predictive permeability model. Though theoretically possible, applying such an approach to characterize the microscopic pore size distribution would represent a daunting task. In contrast, capillary flow porometry is a relatively simple characterization technique widely used up to now to study simple scale porous media such as membranes, filtration media, paper, etc... [2, 3]. In the present study, this method will be applied in a novel way to characterize engineering textiles used in high performance composites. The main objective is to demonstrate its potential to quantify the dual scale pore size distribution of anisotropic fibrous reinforcements.

Apparatus

The experiments are carried out with the 3Gz porometer of Quantachrome Instruments Inc. The control module of this equipment can be connected to different sample holders to conduct porometry tests in different directions. Firstly, through-thickness experiments were carried out with a cylindrical sample holder. This device is one of the standard accessories commercially available and its diameter (25 mm) is larger than the representative elementary volume of the fabrics considered in this study. With this through-thickness configuration, the sample is placed between a porous support plate (bottom) and an O-ring (top). The main drawback of this configuration is the lack of control of the fiber volume fraction since only the sample edge is compressed by the O-ring. Therefore, through-thickness measurement aims only at evaluating the pore size distribution for the natural thickness of the fabric without considering the influence of fabric compaction and different fiber volume fractions. However, after thorough evaluation of this new approach, a new sample holder with thickness control can be devised.

In most LCM manufacturing processes, mold filling is achieved by a simple in-plane resin flow through the fiber bed. Depending on the textile architecture, fibrous reinforcements possess different properties in the weft and warp directions. As a matter of fact, a standard sample holder based on radial flow appears to be limited for in-plane testing of non-isotropic porous structures. A new sample holder was then devised to carry out unidirectional flow experiments. This new sample holder possesses a rectangular cavity and allows measurements in a specified direction depending on how the sample is cut. The device contains shims of calibrated thickness for accurate control of fiber volume fraction.

Methodology

The principle of the test is identical for both the through-thickness and in-plane measurements. The porous sample is first soaked in a special wetting liquid with low surface tension called Porofil®. All the pores are filled up so that the air/solid interface is replaced by a full liquid/solid interface. This filling

process happens spontaneously. As a matter of fact, the free energy of the system decreases because the liquid/solid interfacial free energy is smaller than that of the air/solid interface [2]. The sample is then placed in the device and air pressure is progressively applied by the porometer on the top of the sample to expel the liquid. Two tests are conducted consecutively during which the porometer measures the air flow through the sample as a function of pressure. The first test corresponds to the "wet run" when the liquid is forced out of the sample. Then, when the sample is completely dry, the same range of pressure is applied again during the so called "dry run". Dividing the values obtained during the wet run by the dry ones gives the cumulative flow as a function of pressure. The expel pressure is linked to the pore diameter by assuming an equivalent porous medium made out of cylindrical pores. The equivalent pore diameter is determined by Laplace equation [3]:

$$D = \frac{4\,\gamma \cos\theta}{P} \tag{1}$$

where γ is liquid surface tension, θ is the contact angle of the liquid and P is the pressure applied. For highly wetting liquids, $cos\ \theta = 1$ because the contact angle is zero. Finally, the pore size distribution is obtained by simple differentiation of the cumulative flow data.

Preliminary results

The first experiments carried out on both carbon and glass fiber reinforcements confirmed that the percentage of flow passing through the sample depends on the pore size. The derivative curves reveal a bimodal distribution with two peaks corresponding to the micropores and the mesopores. For the example of Figure 1, the mesopores average size is 150 microns and the micropores one is 25 microns. Note that the measurements are repetitive. In conclusion, capillary flow porometry based on liquid expulsion is a promising technique to characterize the double size porous structure of engineering textiles. In future work, quantitative results obtained by porometry will be compared with the information obtained by a very different approach based on X-ray.

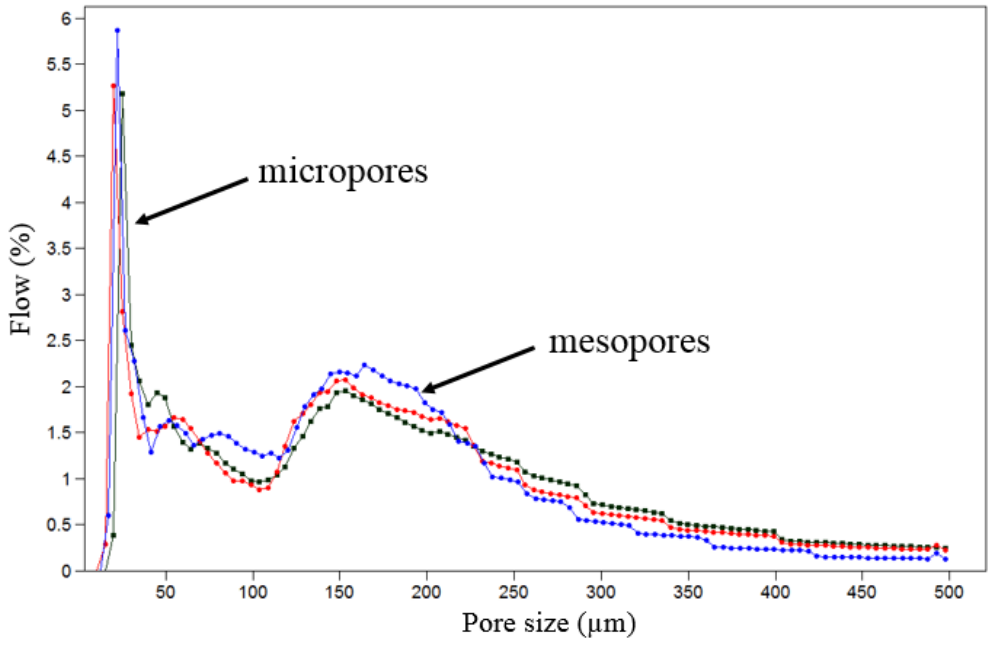

Figure 1: *Through-thickness pore size distribution of three samples from a Sigmatex SC6227270 carbon fabric.*

References

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