EVALUATION BY WICKING TESTS OF CAPILLARY PRESSURE AS FUNCTION OF TEMPERATURE FOR UNCURED RESINS

Pierre-Jacques Liotier[1*], Monica Francesca Pucci[2], Sylvain Drapier[1]

[1]Mechanics and Materials Processing dept, Lab. G. Friedel UMR CNRS 5307, Mines Saint-Étienne, 158 Cours Fauriel 42023 Saint-Étienne, France
[2]C2MA, Mines Alès, 6 Avenue de Clavières 30100 Alès, France
*Corresponding author (liotier@emse.fr)

Keywords: *Liquid Composite Molding – Wetting – Capillary pressure*

Introduction

Test liquids used for permeability characterisation, such as silicone oil, have wetting properties that largely differ from epoxy resins in their liquid state, depending on temperature. This has been proved at room temperature by characterisation of uncured epoxy surface tension and its components [1]. Moreover, some studies have proved the interest of using wicking tests for the characterisation of capillary effects, due to interactions between fluid and fibres [2]. In this 2-steps procedure [2] a totally wetting and dispersive test liquid, commonly n-hexane, is used to characterise the morphology of the preform. Being highly volatile, it cannot be used in temperature. Conversely, silicone oil has the same totally wetting and dispersive character but is stable in temperature, with a very low surface tension.

The present work focuses on wicking tests in carbon fabric reinforcements with different test liquids at several temperatures, following an established procedure [2]. The aim is to evaluate the use of silicone oil as a liquid test for the morphological characterisation of fibrous preforms, prior to evaluating the capillary wicking with resin. This statement based on the theoretical behaviour of silicone oil is logical but as it will be demonstrated, is at least partially, wrong.

Theory

The aim here is to describe wicking of the considered liquid in a fibrous medium compacted in a cylindrical sample holder. The Washburn law, which can be described as equal to the Darcy law allows defining an equivalent capillary pressure P_{cap}. According to those hypotheses, the mass gain m measured by a tensiometer during wicking can be expressed as follows [2]:

$$m^2(t) = \frac{2K\epsilon\rho^2(\pi R^2)^2 P_{cap}}{\eta} t \quad (1)$$

where R is the radius of the cylindrical sample holder, η the viscosity of the liquid and ρ its density, K the permeability in the considered wicking direction [3-4], ε the porosity of the fibrous medium and t the time of flow.
Considering the same mass gain but expressed with the Washburn's equation allows deriving Eq. 2 from Eq. 1:

$$P_{cap} = (c\bar{r})\epsilon \frac{\gamma_L \cos\theta_a}{4K} \quad (2)$$

where c is a parameter related to tortuosity, $\bar{r}$ the mean capillary radius and θ_a the apparent advancing contact angle in the porous medium described by the Washburn law. The squared mass gain should thus be linear as a square root of time, in order to estimate a capillary pressure and validate the assumption of a constant permeability.

Results

Wicking has been performed in a carbon quasi-UD fabric from Hexcel Corp. (reference: 48580), following the method established in previous studies [2]. The silicone oil is the XIAMETER® PMX

200-50 CS and the epoxy resin is the SP106 from GURIT® without hardener. Fig. 1 shows that even if silicone oil is used as a model for liquid resin in permeability measurements, it has a very different behaviour during wicking. Resin shows a behaviour that can be modelled by Darcy and Washburn laws, which means that it is possible to estimate an equivalent capillary pressure and thus permeability. Wicking with silicone oil cannot be described with the same models, this hinders a proper estimation of an equivalent capillary pressure or permeability with this test liquid.

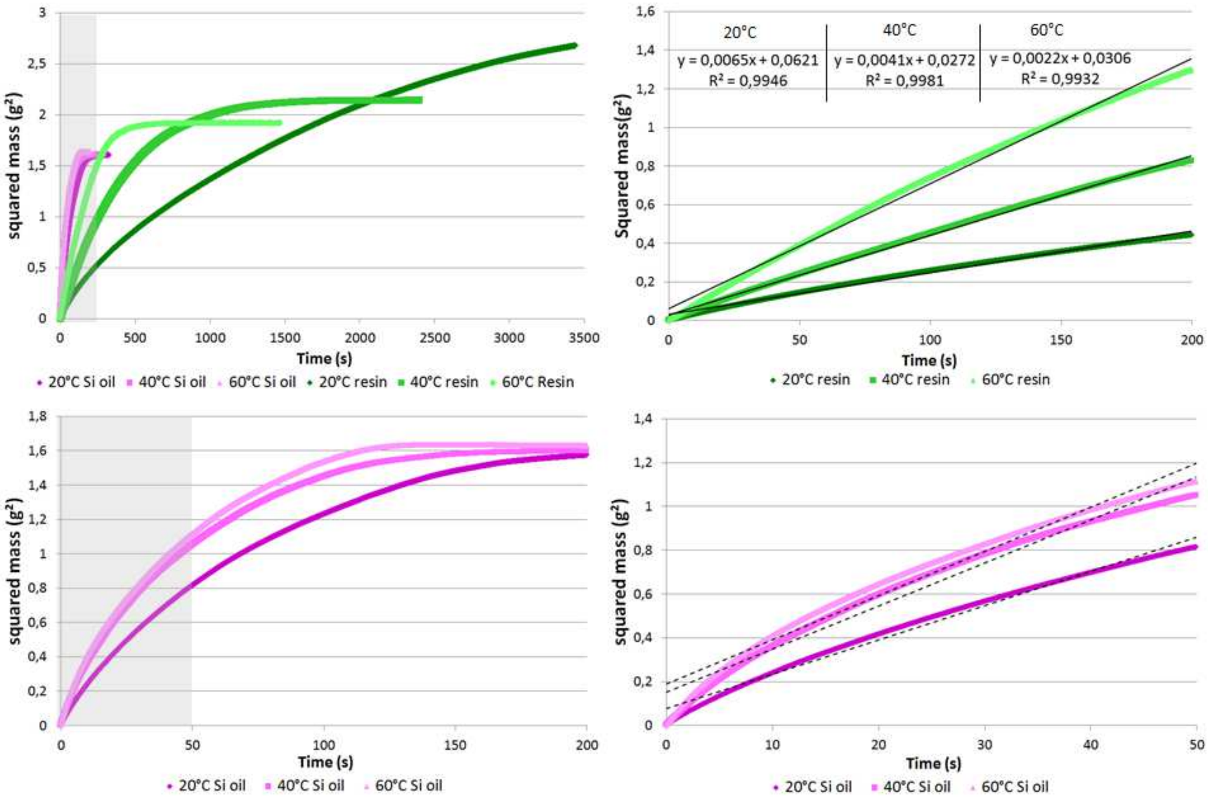

Figure 1: *Wicking in quasi-UD carbon fabric along the fibres main direction with silicon oil and epoxy resin.*

Conclusion

A lot of interesting conclusions can be drawn from Fig. 1 but the main one is that wicking of silicone oil into carbon reinforcements cannot be described with the common equations used for permeability estimation. Since no pressure gradient is applied in this test, the physicochemical differences between resin and silicone oil make the comparison impossible. It is also impossible to replace n-hexane by silicone oil in temperature, even if the physicochemical properties of those liquids are theoretically similar. These results raise questions about the validity of using silicon oil as a test liquid for unsaturated permeability estimation, or on the meaning of unsaturated permeability itself.

References

[1] M.F. Pucci, P.J. Liotier, and S. Drapier. Tensiometric method to reliably assess wetting properties of single fibers with resins: Validation on cellulosic reinforcements for composites. Colloids and Surfaces A : Physicochemical and Engineering Aspects, 512 :26–33, 2017.

[2] M.F. Pucci, P.J. Liotier, and S. Drapier. Capillary wicking in a fibrous reinforcement - orthotropic issues to determine the capillary pressure components. Composites Part A : Applied Science and Manufacturing, 77 :133–141, 2015.

[3] R. Arbter, J.M. Beraud, C. Binetruy, et al. Experimental determination of the permeability of textiles: a benchmark exercise. Compos Part A 2011; 42: 1157–1168.

[4] Lundström T. S., Stenberg R., Bergström R., Partanen, H., Birkeland P. A.: In-plane permeability measurements: A nordic round-robin study. Composites Part A:Applied Science and Manufacturing, 31, 29–43 (2000).