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Test methods for the experimental characterization of in-plane permeability of fibrous reinforcements for liquid composite moulding

Méthodes d'essais pour la caractérisation expérimentale de la perméabilité dans le plan des renforts fibreux pour le moulage de composites liquides

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Foreword

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Liquid composite moulding (LCM) processes are employed for the manufacture of fibre reinforced polymer composites (FRPC). In all LCM processes, dry fibrous reinforcements are impregnated with a liquid resin system, which is cured following reinforcement impregnation to form the matrix in which the fibres are embedded. Impregnation is driven by positive applied pressure and/or vacuum. LCM is widely applied for the manufacture of lightweight components in the automotive, aerospace, marine, and energy (e.g. blades for wind turbines) industries.

To obtain short cycle times and high component quality in LCM, i.e. fast and complete saturation of the reinforcement with liquid resin, a suitable process design is required, based on knowledge of material properties. Darcy's law relates the phase-averaged flow velocity to the applied pressure gradient, the dynamic resin viscosity, and the reinforcement permeability for fluid flow. The permeability of fibrous structures, such as reinforcements, is generally direction-dependent and is described by a symmetric second-order tensor. Diagonalisation of the tensor leads to three principal permeabilities, which correspond to the flow oriented along three orthogonal axes, two of which describe the in-plane permeability.

This document focuses on the experimental characterization of unsaturated in-plane permeability of reinforcing materials for LCM. As with any kind of experiment, methodological, systematic and statistical errors may arise. In order to minimize methodological errors caused by different experimental methods, this document covers the two most common approaches, linear and radial flow experiments. Systematic errors inherent to these methods are minimized by distinct procedures for preparing and executing the flow experiments as well as for post-processing the acquired measurement data as prescribed in this document. Statistical errors are dominated by variations in material properties, particularly inhomogeneous areal weight and thus, fibre volume fraction of the reinforcing materials. This document covers well known statistical methods, such as multiple experiments at repetitive conditions, in order to estimate the uncertainty associated with the results.

Test methods for the experimental characterization of in-plane permeability of fibrous reinforcements for liquid composite moulding

1 Scope

This document specifies test methods for the experimental characterization of in-plane permeability of fibrous reinforcements for liquid composite moulding. Requirements for test equipment, test methods and data analysis are detailed, to ensure optimal accuracy and reproducibility of the results.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the cited edition applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 286-1:2010+Cor1:2013, *Geometrical product specifications (GPS) — ISO code system for tolerances on linear sizes — Part 1: Basis of tolerances, deviations and fits*

ISO 2555, *Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity using a single cylinder type rotational viscometer method*

ISO 21920-2, *Geometrical product specifications (GPS) — Surface texture: Profile — Part 2: Terms, definitions and surface texture parameters*

ISO 21920-3, *Geometrical product specifications (GPS) — Surface texture: Profile — Part 3: Specification operators*

3 Terms, definitions, symbols and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1.1
in-plane permeability
quantitative material parameter of a fibrous reinforcement (a porous medium), relating the phase-averaged flow velocity of a liquid in the reinforcement to the applied pressure gradient and the dynamic viscosity of the fluid.

Note 1 to entry: During impregnation of a fibrous reinforcement with a fluid, the permeability of the fibrous reinforcement, the permeability tensor, $\boldsymbol{K}$, relates the phase-averaged flow velocity, v, to the applied pressure gradient, $\nabla \boldsymbol{p}$, and the dynamic resin viscosity, μ, as stated in Darcy's law.

$$\boldsymbol{v} = -\left(\frac{\boldsymbol{K}}{\mu}\right) \cdot \nabla \boldsymbol{p}$$

As per this definition, the permeability is given in units of square metres (m^2). Importantly, the permeability of a reinforcement depends on the fibre volume fraction and the geometrical fibre arrangement. Because of the directionality of the fibre arrangement in a reinforcement, the permeability is generally anisotropic. The principal components of the tensor $\boldsymbol{K}$, in its diagonal form are referred to as k_1 and k_2, representing the highest and lowest values of the in-plane permeability, respectively.

Note 2 to entry: Permeability is an equivalent parameter defined at the level of an equivalent homogeneous medium representing an intrinsically heterogeneous material. Darcy's law has been extended to unsaturated flow or transient flow, neglecting the effect of dynamic wetting.

3.1.2
unsaturated flow
dynamic flow of a fluid in a porous medium where initially empty (vacuum) pore spaces are filled or an initially present fluid (e.g. air) is displaced

3.1.3
in-plane anisotropy ratio
characteristic of a material showing different properties in different directions

Note 1 to entry: The in-plane anisotropy ratio, α , is defined here as the ratio of lowest to highest in-plane permeability, i.e. $\alpha = \frac{k_2}{k_1}$.

3.1.4
linear injection
injection of fluid into a porous medium along one short edge of a rectangular geometry, resulting in a flow along the long edge, with velocity vectors oriented primarily in one direction

3.1.5
radial injection
injection of fluid into a porous medium through a central injection gate, resulting in a flow with velocity vectors extending radially outward from the gate, in all in-plane directions

3.1.6
race-tracking
locally increased flow velocity in gaps between specimen and mould

3.1.7
slowtracking
locally decreased flow velocity caused by over-compaction of the specimen along the mould edges

3.1.8
orientation angle
angle, β, between the direction of highest flow velocity, k_1, and a reference direction, which is commonly the production direction of the material

Note 1 to entry: See Figure 2.

3.2 Symbols and abbreviated terms

Symbol	Unit	Meaning
A_s	m^2	Specimen area (i.e. l_s multiplied with w_s for rectangular specimens)
$C_{1...4}$	m^2	Constants for the calculation of the principal permeabilities
CV	%	Coefficient of variation
$\boldsymbol{D}$		Matrix containing the (x, y, z) data sets of an experiment
$\boldsymbol{e}$		Eigenvector
$\boldsymbol{f}_1, \boldsymbol{f}_2$		Auxiliary functional terms
FS		Full scale

Symbol	Unit	Meaning
h	m	Height of the reinforcement specimen
i		Counting variable indicating the time step
J		Counting variable for experimental configurations of mould height and number of layers
k		Counting variable indicating the measurement data set
$k_{C,1}$ and $k_{C,2}$	m^2	Kozeny constants
$\bar{k}_e$	m^2	Average of experimentally measured permeability
k_e	m^2	Experimentally determined permeability
k_e^0	m^2	Experimentally determined permeability in the defined reference direction
k_e^{45}	m^2	Experimentally determined permeability orientated at an angle of 45° to the defined reference direction
k_e^{90}	m^2	Experimentally determined permeability perpendicular to the defined reference direction
k_e^{-45}	m^2	Experimentally determined permeability orientated at an angle of -45° to the defined reference direction
k_x	m^2	Permeability in flow direction
k_y	m^2	Permeability perpendicular (in-plane) to flow direction
k_1	m^2	Highest in-plane permeability
k_2	m^2	Lowest in-plane permeability
k_3	m^2	Out-of-plane permeability
$k_{1,a}$	m^2	Highest in-plane permeability, adjusted according to the actual fibre volume fraction
$k_{2,a}$	m^2	Lowest in-plane permeability, adjusted according to the actual fibre volume fraction
$\boldsymbol{K}$	m^2	Permeability tensor
l_s	m	Specimen length
LCM		Liquid composite moulding
m		Slope of the trend line correlating ${x_{mid}}^2$ and t
M_s	kg	Specimen mass (dry)
n		Number of measurement data sets in an experiment
n_L		Number of layers of a fibrous reinforcement in a specimen
n_T		Number of sampled data sets in a linear injection experiment
N		Number of experiments in a set
p	Pa	Array of experimental pressure values
$\nabla \boldsymbol{p}$	Pa	Pressure gradient applied across the specimen, i.e. the gauge pressure applied
ΔP	Pa	Pressure drop
ΔP_{eff}	Pa	Time-averaged pressure drop
q		Coefficient in paraboloid matrix
$\boldsymbol{q}$		Array of coefficients from paraboloid matrix
$\hat{q}$		Coefficient in rotated matrix
Q	m^3/s	Volume flow rate
$\mathbf{Q}$		Matrix of paraboloid coefficients

Symbol	Unit	Meaning
$\mathbf{Q}_{33}$		3x3 Submatrix of paraboloid coefficients
$\hat{\mathbf{Q}}$		Matrix of rotated paraboloid coefficients
r_s	m	Specimen radius
r_1	m	Major radial extension of flow ellipse
r_2	m	Minor radial extension of flow ellipse
E_{RMS_f}	m	Root mean square error of fitting the elliptic paraboloid
E_{RMS_p}	Pa	Root mean square error of the pressure
s_{N-1}	m^2	Permeability standard deviation
$\boldsymbol{S}$		Scatter matrix
SVD		Singular value decomposition
t	s	Time
$\boldsymbol{t}$	s	Array of experimental timestamp values
T_{eff}	°C	Time-averaged temperature
$\boldsymbol{v}$	m/s	Darcy velocity vector
V		Coefficient of variation
V_f	%	Fibre volume fraction
w_s	m	Specimen width
x	m	Spatial coordinate in the reference direction of the coordinate frame of the test rig
x_{mid}	m	Shortest distance between the inlet region and the flow front position at the midpoint along the specimen width
x_{M1}	m	Shortest distance between the inlet region and the flow front position along the upper edge of the specimen in linear injection
x_{M2}	m	Shortest distance between the inlet region and the flow front position along the bottom edge of the specimen in linear injection
y	m	Spatial coordinate perpendicular (in-plane) to the reference direction of the coordinate frame of the test rig
z	s	Experimental time
α		Anisotropy ratio (k_2 / k_1)
β	degrees	Angle indicating orientation of K_1 with respect to the defined reference direction of the considered fibrous reinforcement
δ	°	Relative angle between the long cutting edge of a specimen for linear flow experiments reinforcement and the defined reference direction of the considered fibrous reinforcement
ε	m^2	Root mean square error of the flow front location x
ε_{crit}		Critical threshold for the race-tracking error
ε_K	m^2	Measurement error
ε_R		Race-tracking error
μ	Pa·s	Dynamic viscosity of fluid
λ		Eigenvalue
$\xi_{0\ldots2}$		Auxiliary quantities
ρ_f	kg/m^3	Material density
ϕ	%	Porosity of reinforcement

Symbol	Unit	Meaning
w_A	kg/m^2	Areal density of a fibrous reinforcement layer (grammage)
ω	°	Smallest of the three relative angles δ selected for testing

4 Principle

A specimen of the fibrous reinforcement is compressed between two impermeable, parallel plates at a defined and uniform thickness. Then, a test fluid with known viscosity is injected at constant injection pressure through a defined inlet region, either a linear injection gate along one specimen edge or a radial injection gate in the centre of the specimen. This results in a one- or two-dimensional (i.e. linear or elliptical) flow pattern. While the reinforcement is impregnated, the flow front propagation is tracked to determine the directional flow front velocity. Data reduction schemes based on Darcy's law are applied to calculate in-plane permeability from the flow front velocity, the applied pressure gradient, the fluid viscosity, and the reinforcement porosity.

5 Design of experiments

5.1 Selection of injection method

The linear and radial test methods are equally applicable to the majority of reinforcements.

NOTE In special cases, each of the methods provides relevant specific advantages and disadvantages resulting from the different injection strategies:

— In the linear flow method, resin flows along the specimen edges. Gaps between the specimen and the mould walls can induce race-tracking, causing locally increased flow velocity compared to the bulk material. In the radial flow method, flow takes place within the specimen. However, if k_1 >> k_2, the flow front in the k_1 -direction may reach the specimen edges before the minimum distance to the inlet (to obtain a stabilised flow front shape) is reached in the k_2 -direction. This would cause a calculation error as the resulting change in the pressure distribution is not considered during data processing.

— Radial injection methods allow for determination of the full in-plane permeability tensor in a single test, whereas it requires three tests in the linear injection method.

— Radial injection methods typically employ more expensive tooling than linear injection, due to the greater propensity for mould deflection owing to the larger specimen surface area.

Details on specific sources of scatter are described in detail in the publications on the results of international benchmark studies (see References [1] to [3]).

5.2 Number of repeat tests

The flow experiments defined in this document for characterization of unsaturated in-plane permeability are destructive by nature as the reinforcement specimen is irreversibly saturated with the test fluid. Therefore, repeat tests shall not be performed on a previously tested specimen. In repeat tests, new specimens with identical specifications, preferably from the same material roll/sheet, are tested at identical target conditions.

For statistical evaluation, at least five repeats shall be performed for each test condition.

NOTE Depending on the material, five repeats might still not be enough to reach a confidence interval required for certain statistical evaluations, e.g. calculation of standard deviation (see Reference [4]).

5.3 Setting the fibre volume fraction

The dependence of the in-plane permeability values on the fibre volume fraction is often of major interest. Varying the fibre volume fraction for a given reinforcement may either be done by adapting the mould cavity height, the number of layers present in the mould, or both, according to Formula (1):

$$V_f = \frac{n_L \cdot w_A}{\rho_f \cdot h} \tag{1}$$

All tests at a particular value of V_f shall be performed with the same number of layers, n_L. To minimize the differences in flow conditions near the mould bottom or top, and in the middle of the specimen, all tests shall be performed with the minimum values $n_{L,min} = 4$ and $h_{min} = 2$ mm, respectively.

The uncompressed thickness of a specimen shall be greater than the anticipated mould height for the test, to ensure tight packing of the specimen against the top mould surface.

Formula (1) assumes that the reinforcement consists of one type of fibre. If more than one fibre material is used, or if additional materials are present in the reinforcement, such as polymeric powder binder or stitching yarns, this shall be considered by calculating separate volume fractions for each material component, from their respective areal densities and material densities, and then adding up the component volume fractions. In this case, one should use the term “solid volume fraction” (the sum of volume fractions of all components) to point out that the solid volume consists of more than one type of reinforcing fibres or other solids.

5.4 Selecting the fluid injection pressure

The injection pressure shall not exceed 0,3 MPa.

This document does not define a minimum pressure. Yet, it is emphasized that, with decreasing injection pressure, the measurement error caused by neglecting the capillary pressure and wetting effects increases. This capillary pressure depends on the fluid/reinforcement interaction and can reach a sufficient value to have an influence on the calculated permeability. It is thus recommended to estimate the capillary pressure for the combination of test fluid and reinforcement, and to use a pressure gradient significantly higher than the estimated value (at least two times higher). A corresponding test method is, for example, described in Reference [5]. In any case, it is important to consider the wetting properties of the test fluid to evaluate the validity of the permeability value calculated. For common test fluids and fibre structures, depending on their fibre volume fraction and orientation, capillary pressures from 1 kPa to 40 kPa have been found.

NOTE The injection pressure can influence the permeability measurement. Darcy’s law assumes a rigid porous media. However, reinforcements and the mould can both deform under high fluid pressure.

5.5 Temperature conditions

The test shall be performed under isothermal conditions.

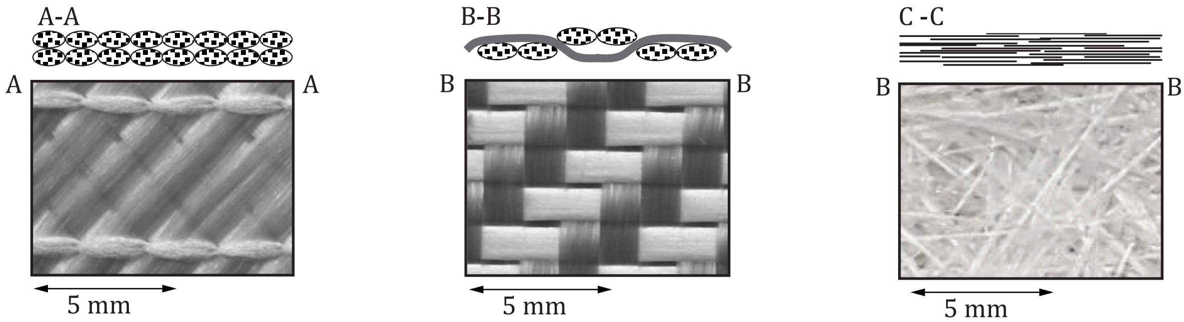
5.6 Plausibility checks

After initial set-up of a new test apparatus, a basic plausibility check on the results should be performed. This can be done by performing measurements on a reference porous media such as the one described in Reference [6]. The geometrical data of this structure (including a CAD-model), as well as information on its permeability characteristics can be downloaded from: https://standards.iso.org/iso/4410/ed-1/en.

6 Test specimen and specimen preparation

6.1 General information

The types of reinforcement tested by the methods described in this document may be woven fabrics, non-crimp fabrics, braids, knits or non-wovens, but can also include fibre structures prepared by dry fibre placement, tailored fibre placement or similar processes. In general, this document is applicable to characterization of specimens with a quasi-homogeneous structure. Figure 1 shows examples of typical reinforcements.



a) Non-crimp fabric **b) Woven fabric** **c) Non-woven**

Figure 1 — Examples of reinforcement types

In the context of composite manufacturing, reinforcements are frequently made from carbon, glass or aramid fibres. Other fibre types, e.g. synthetic fibres such as polyethylene-based fibres or natural fibres such as hemp or flax, may be used. In general, this document is not restrictive in terms of the fibre material to be investigated nor its sizing, as long as there is no interaction with the fluid, such as fibre swelling due to moisture absorption or sizing dissolution.

6.2 Specimen cutting

Methods, which allow high cutting accuracy to be obtained and minimize specimen deformation shall be applied. The use of computerized numerical controlled (CNC) machines is recommended. Other cutting methods can be applied if a high degree of geometrical accuracy can be obtained.

NOTE Dimensional inaccuracies and unwanted deformation induced by cutting and handling, cause errors in the calculation of the fibre volume fraction, and can contribute to unwanted race-tracking, uneven nesting and local slowtracking effects.

6.3 Specimen stacking

A reference direction and reference side shall be defined for every fibrous reinforcement to be tested. For roll materials, the production direction and top side, as illustrated in Figure 2, shall be used as the reference direction and side, respectively. When intending to measure the permeability of one specific fibrous reinforcement, all layers stacked to form the specimen shall have aligned reference directions and the reference side on top (see Figure 2).

