NPL REPORT MAT 82

SURVEY OF PROCEDURES IN USE FOR PERMEABILITY MEASUREMENTS IN LIQUID COMPOSITE MOULDING PROCESSES

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MAY 2016

Department for Business Innovation & Skills

FUNDED BY BIS

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ABSTRACT

This report presents the results of an international survey of permeability measurement procedures used to support development and use of liquid moulding processes. The purpose of the survey has been to collate information, identify measurement issues and generate a reference document for future international benchmarking, round-robin and standardisation activities. The survey results are representative and the number of respondents to the survey is satisfactory and of high quality, as sent to specific organisations that are expert in this measurement area.

From the responses, all organisations conduct measurements based on Darcy's law, but using their in-house built test rigs and their own methodologies developed from many years of research in this field. In general, *unsaturated* (by monitoring pressure and flow front) and *saturated* (by monitoring pressure and flow rate) measurements are conducted. Currently, there are no measurement standards, standardised test rigs, standard reinforcement materials, or standard test fluids available. It is noted that model fluids are overwhelmingly used (c.f. resins, catalysed or uncatalysed)

In parallel to this international permeability survey, the National Physical Laboratory and the National Composites Centre will integrate the survey responses and Benchmark procedure into a draft for future standardisation.

ISSN 1754-2979

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Approved on behalf of NPLML by
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CONTENTS

1 INTRODUCTION

A survey of permeability measurement procedures in use has been conducted by National Physical Laboratory (UK) and National Composites Centre (UK) in order to identify;

i) Organisations, worldwide, conducting permeability measurements or having interest,
ii) Measurement rigs, types and working principles,
iii) Measurement techniques,
iv) Test materials (reinforcements and fluids),
v) Issues in measurements
vi) New collaboration opportunities (e.g. benchmark, round-robin exercises)
vii) Standardisation needs

As well as surveying participants in the Benchmark-I [1] and Benchmark-II [2] exercises, a wider international contact list of experts in these techniques was generated to obtain more information. The benchmark exercises [1, 2] and the permeability procedure [3] prepared based on a linear-unsaturated permeability measurement approach are important steps towards future standardisation of in-plane permeability measurements. This report presents information obtained from the organisations listed in Table 1.

The information obtained in the survey represents current best practice and will form guidance and direct input into the future standardisation initiatives. A comprehensive list of references to published work supplied by respondents is included in Section 15.

2 ORGANISATIONS SURVEYED

The responses from 34 organisations, including universities and industrial companies, to this survey are collated in this report. Of the organisations, 90% (Figure 1) stated that they conduct permeability measurements using in-house built test rigs, and the permeability calculations are based on Darcy’s law. In general, the types of measurements include, *linear-saturated*, *linear-unsaturated*, *radial-saturated*, *radial-unsaturated*, *through thickness-saturated* and *through-thickness-unsaturated*. Each organisation reported a different procedure [1-32], but the most commonly used procedures are described in references [1], [2] and [3]. Only two organisations reported an alternative way of measuring in-plane permeability based on continuous measurements using compression [18].

Table-1: Organisations

University of Leoben	Austria	INEGI	Portugal
INTEMA-CONICET	Argentina	*Polytechniqe University of Valencia	Spain
KU Leuven	Belgium	*SICOMP-Swerea	Sweden
Vrije Universiteit Brussel	Belgium	*Ecole Polytechnique Federale de Lausanne	Switzerland
École Polytechnique de Montréal	Canada	EPFL	Switzerland
McGill University	Canada	ETH Zurich	Switzerland
*Ecole des Mines de Douai	France	Khalifa University	UAE
École centrale de Nantes	France	Bombardier Aerospace	UK
*ONERA	France	Formax	UK
*PPE	France	National Composites Centre	UK
Safran	France	National Physical Laboratory	UK
*Universite du Havre	France	Plymouth University	UK
Clausthal University of Technology	Germany	University of Nottingham	UK
IVW GmbH	Germany	Brigham Young University	USA
Technical University Munich	Germany	University of Delaware	USA
*AIRBUS GmbH	Germany	*University of Wisconsin	USA
Technical University of Lodz	Poland		

*Data obtained from Benchmark exercises [1, 2]

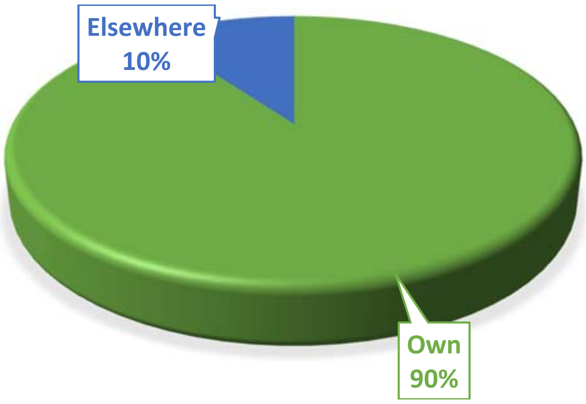

Figure 1: Participant's measurements

3 PERMEABILITY MEASUREMENT TECHNIQUES

There are four main experimental setup configurations for the measurement of in-plane permeability, namely *linear unsaturated*, *linear saturated*, *radial unsaturated*, and *radial saturated*. These configurations are performed in either *constant flow rate* or *constant injection pressure* mode. A Resin Transfer Moulding (RTM) type test rig integrated with a sensing system is used.

To determine through-thickness permeability, *through-thickness saturated* and *through-thickness unsaturated* measurements are performed using a cylindrical compaction rig integrated with a sensing system.

Between organisations, there are different opinions concerning reliability and repeatability of linear (channel) and radial flow permeability measurements. The disadvantage of linear flow permeability measurement is race tracking, which can be avoided by using an appropriate sealant between the fabric edge and the metallic tool. The disadvantage of the radial flow method is the central hole in the middle of the preform, which requires advanced cutting tools, distorts the fibre architecture and generates handling problems.

Some participants highlighted that saturated flow complies with Darcy's definition, but may not be relevant to processing of fibre reinforced composites. Greater knowledge of surface chemistry and physics and correlation to measured (saturated and unsaturated) permeabilities would be required.

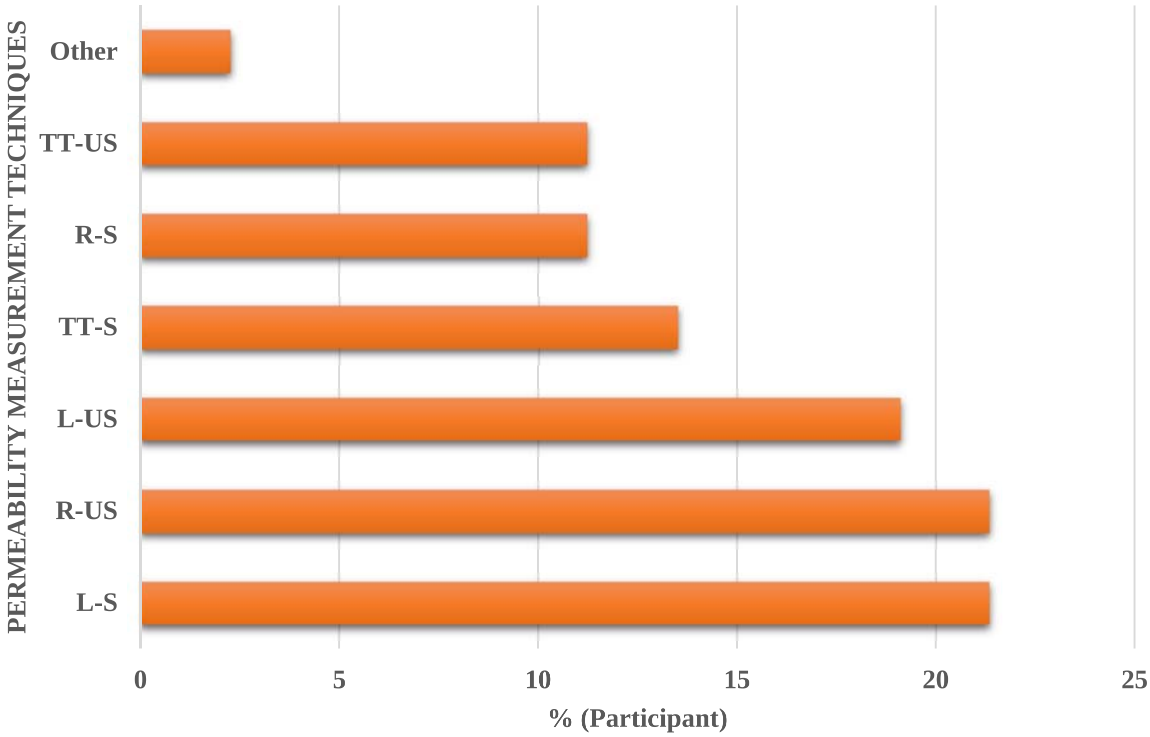

Figure 2: Percentage of respondents using different permeability measurement techniques (L-S: Linear Saturated, L-US: Linear UnSaturated, R-S: Radial Saturated, R-US: Radial UnSaturated, TT-S: Through-Thickness Saturated, TT-US: Through-Thickness Unsaturated)

Linear-saturated, *radial-unsaturated* and *linear-unsaturated* techniques are used more than other permeability measurement techniques. The *through-thickness saturated* permeability measurement technique is more preferable than the *through-thickness unsaturated* measurement technique (Figure 2).

4 FABRIC STRUCTURE

The results show that different fibre types (e.g. glass, carbon, aramid, natural fibre) and fabric formats (e.g. woven, braided, non-crimp multiaxials, nonwoven, chopped fibre mats, distribution media) are used. Respondents reported some unexpected changes in the fabric structure during handling of the fabrics and during the experiments. In addition, further fibre distortions can happen at high injection pressures. Some organisations carry out investigations on shear deformation of fabrics and permeability measurements of sheared specimens.

5 TEST FLUIDS

A wide range of model fluids are used instead of resin systems due to the fact that they are easier to handle since they do not cure and have the advantage of constant viscosity at constant injection conditions. Silicone oil, due to its similar surface characteristics to resins, is the most frequently used test fluid, followed by uncatalysed resin. Corn syrup is the most popular non-toxic test fluid (Figure 3).

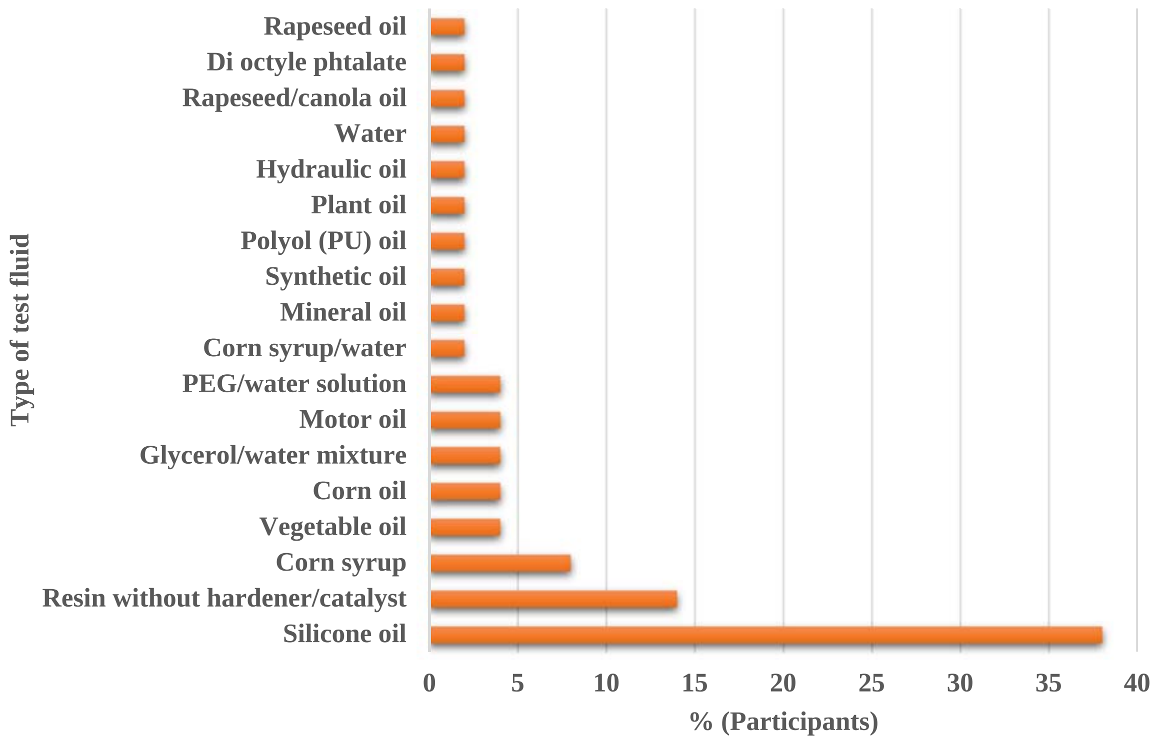

Figure 3: Percentage of respondents using different types of test fluid for permeability measurements

6 NUMBER OF REPEAT MEASUREMENTS

Measurements were found to be repeated between 1 and 3 (38%), 3 and 10 (53%) and more than 20 times (9%). For statistical analysis, the measurements are repeated more than 20 times, but in general they are normally repeated between 3 and 10 (Figure 4).

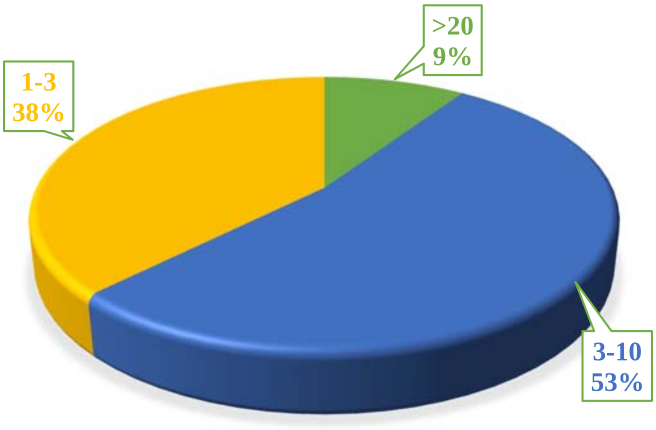

Figure 4: Number of test repeat

7 TEST RESULTS

Figure 5 presents the standard deviation results that are typical for organisations. This varies from 5% to 50%, but can be higher than 50% for some organisations. These variations are due to the type of fabric material and format, homogeneity of fabric structure, human factors (handling, preparation), number of test repeats, type of test procedure and test rig.

It is reported that repeatability of measurement results also depends on the repeatability in the material properties within and between the batches, which is currently uncontrollable by the reinforcement manufacturers. In case of high reproducible material, the standard deviation is found around 10%. However, this also depends on the measurement issues reported in Section 11.

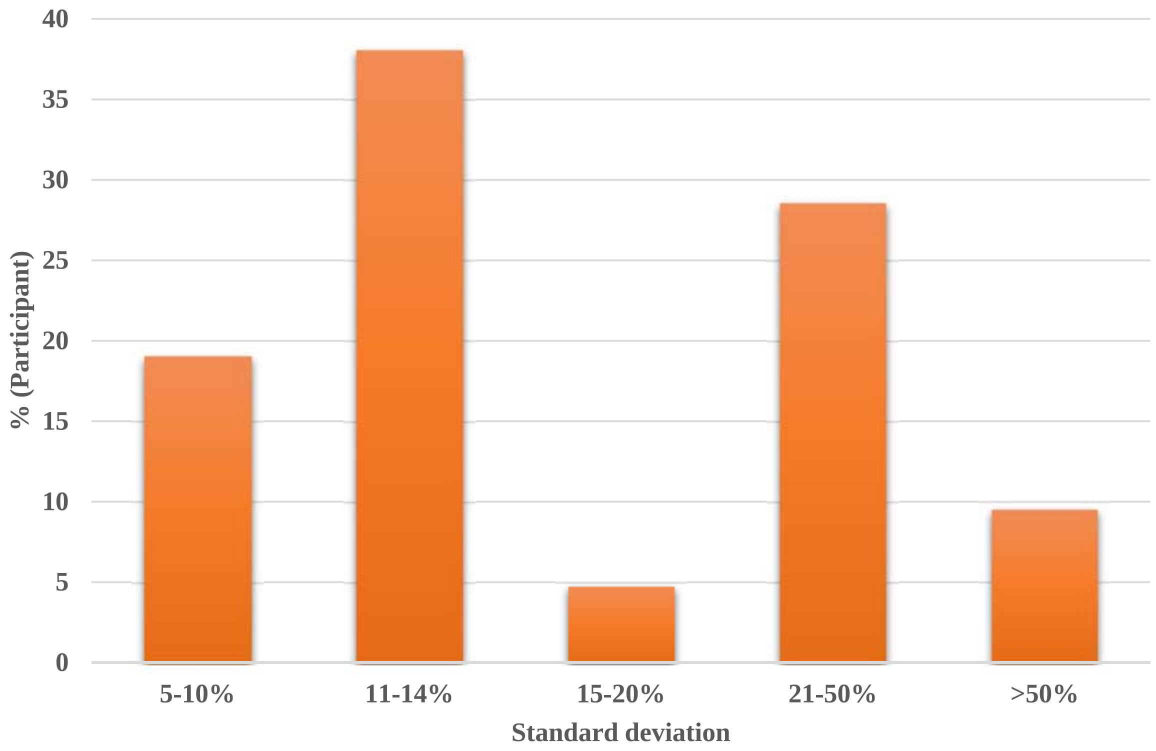

Figure 5: Standard deviation of permeability measurements

8 TEST ENVIRONMENT

The majority of organisations (77%) reported that temperature and humidity are controlled and recorded in the test environment, whereas the remainder reported that they do not control their test environments but record both ambient temperature and humidity during measurements (Figure 6).

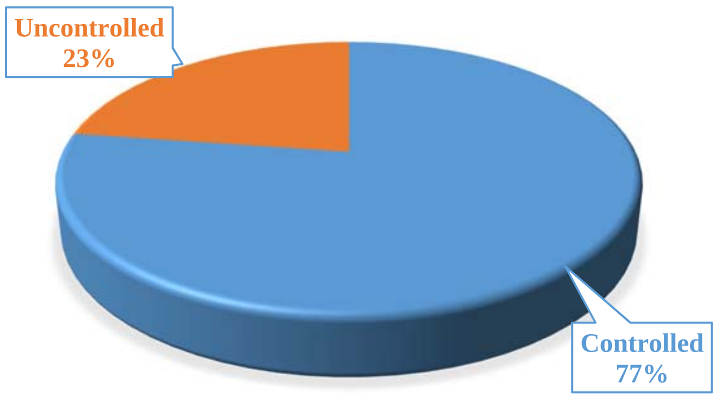

Figure 6: Controlled or uncontrolled test environment during permeability measurements

9 TEMPERATURE MEASUREMENT LOCATIONS

Nearly 35% of respondents measure the temperature of the test fluid in the fluid pot. Other temperature measurement locations are as follows: mould (27%), inlet (14%), vent (8%), reinforcement (5%) and pressure pot (5%). Some respondents (5%) do not conduct temperature measurements, but record ambient temperature during measurements (Figure 7). Organisations highlighted that their reinforcements, test fluids and test rigs are kept at the room temperature before and during measurements.

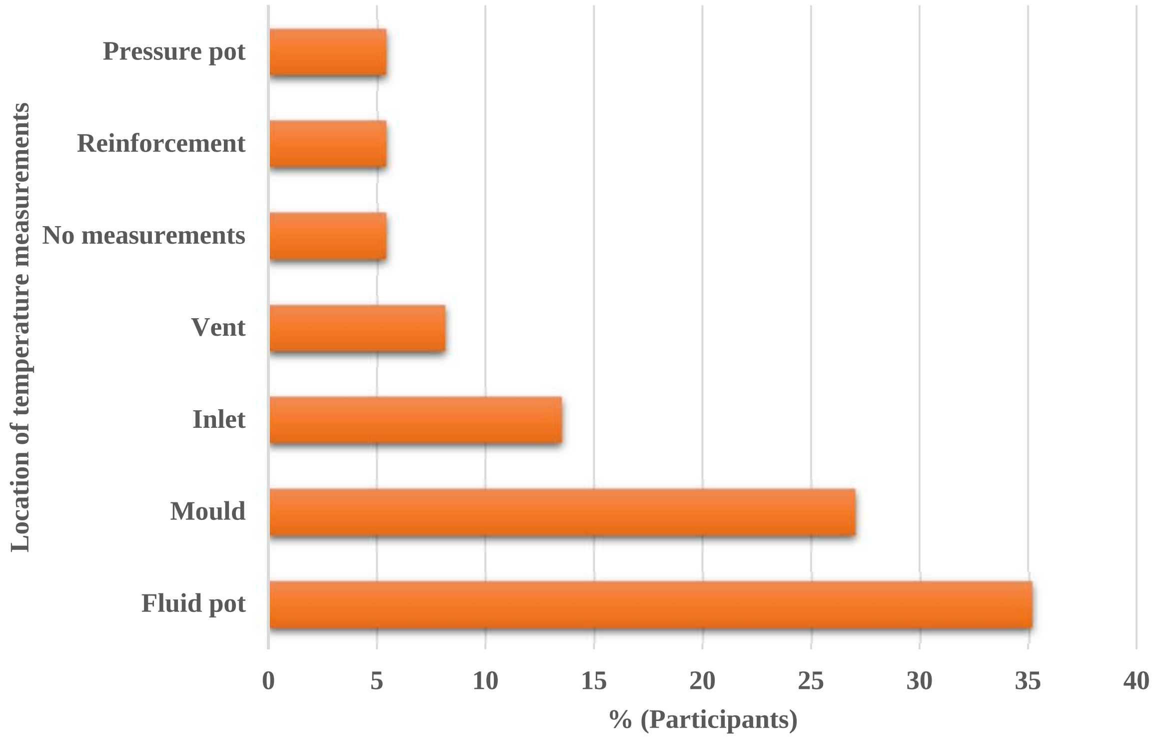

Figure 7: Location of temperature measurements during permeability measurements by percentage of respondents

10 PROPERTY MEASUREMENTS

Type of property measurements that aid permeability measurements and support process simulations were also surveyed. Figure 8 presents property measurements that are conducted by organisations. Preform compressibility (22%) and fluid viscosity (18%) measurements are performed more than other measurements. Only one organisation stated that they conduct hydrodynamic compaction experiments.

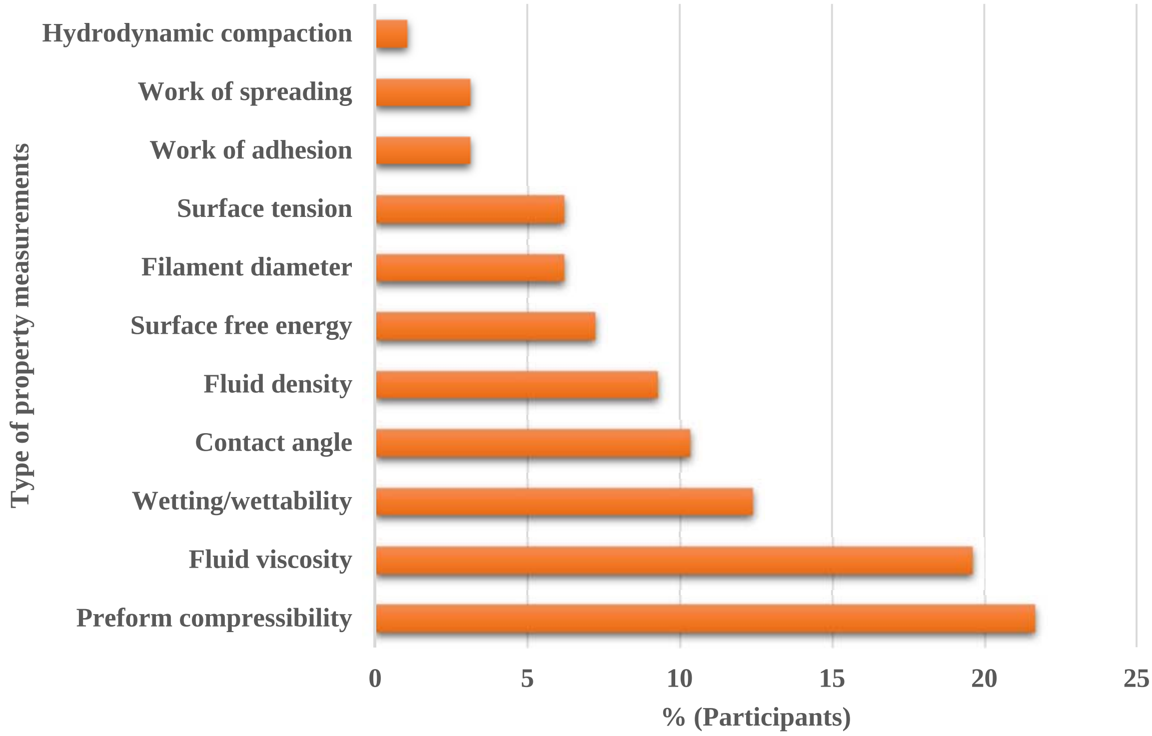

Figure 8: Property measurements conducted in conjunction with permeability measurements by percentage of respondents

11 CRITICAL ISSUES

Material selection and design of measurement rig, fluid injection system, type, location and performance of sensing system, integration of sensing system, and geometrical tolerances and factors are important for permeability measurements. Mould deflection (29%) was reported as the most critical issue for permeability measurements, followed by test rig design and sensor issues (Figure 9).

As shown in Figure 10, there are various issues related to measurement procedures, materials, human errors and ambient conditions. It can be seen that race tracking (14%) and preparation and handling of materials (~12%) are the most common issues. In the survey, it is mentioned that using an appropriate sealant at the edge of reinforcements can avoid race tracking, which is normally not a problem for the radial flow measurements. From here, it can be said that preparation and handling of materials is the most important factor because race tracking is specific to the channel flow measurements. Other important factors are preform structure, type of fabric material, nesting, dual-scale effects, capillary effects particularly in unsaturated injection, deformation of the reinforcement, and ratio of capillary pressure and injection pressure.

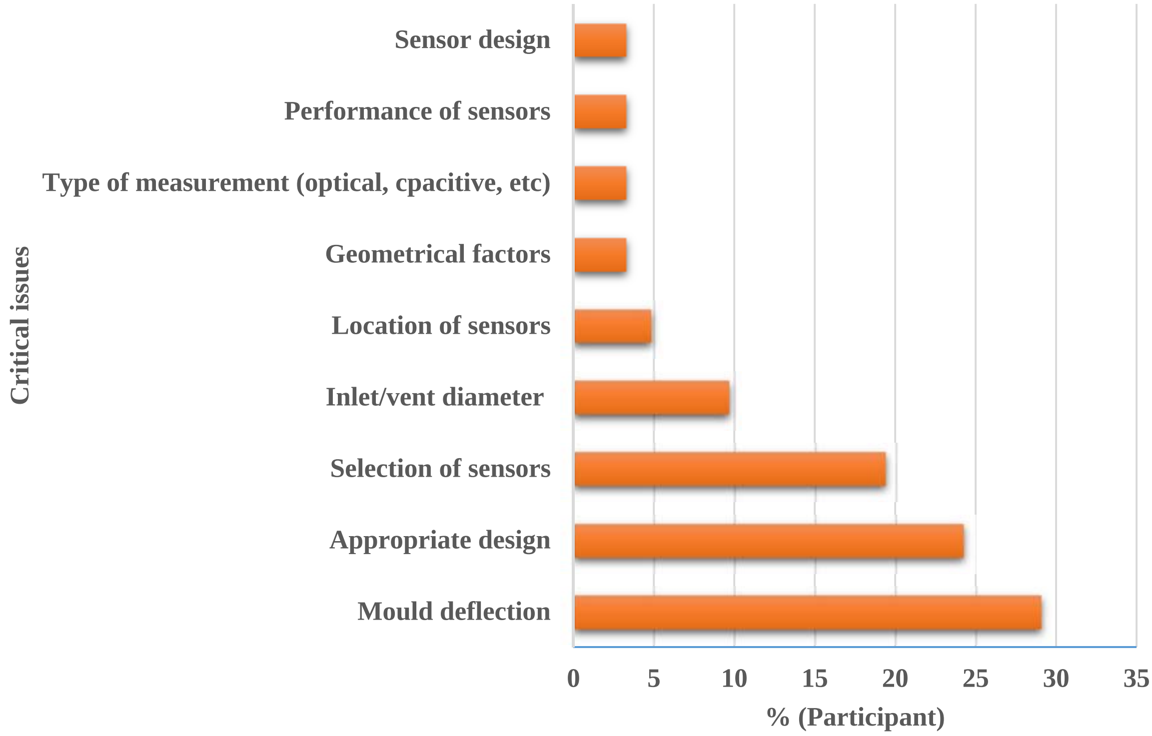

Figure 9: Critical issues related to test rig

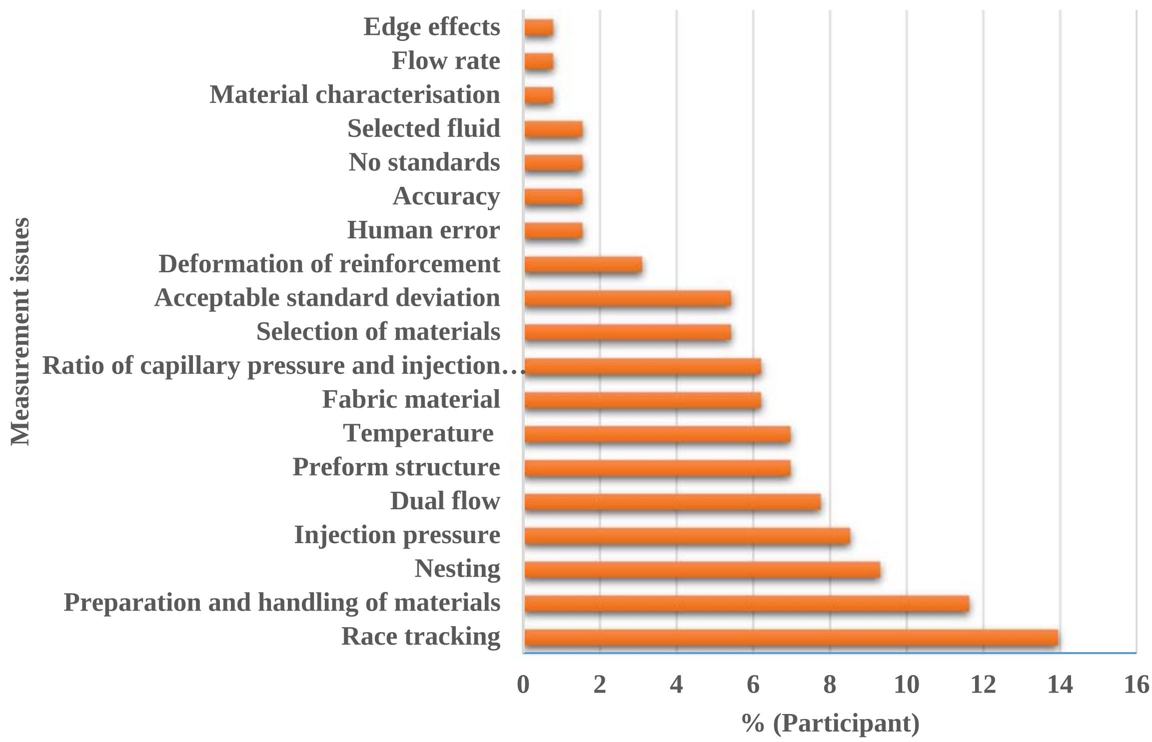

Figure 10: Issues affecting permeability measurements

12 TIMELINESS OF STANDARDISATION

From the results, the timeliness of this standardisation activity is a current need for 44%, overdue for 48% and needed in the future (+5 years) for 8% (Figure 11). The standardisation priorities shown in Figure 12 are very close to each other. However, l*inear-unsaturated* (21%) and *radial-unsaturated* (18%) received slightly more attention than others.

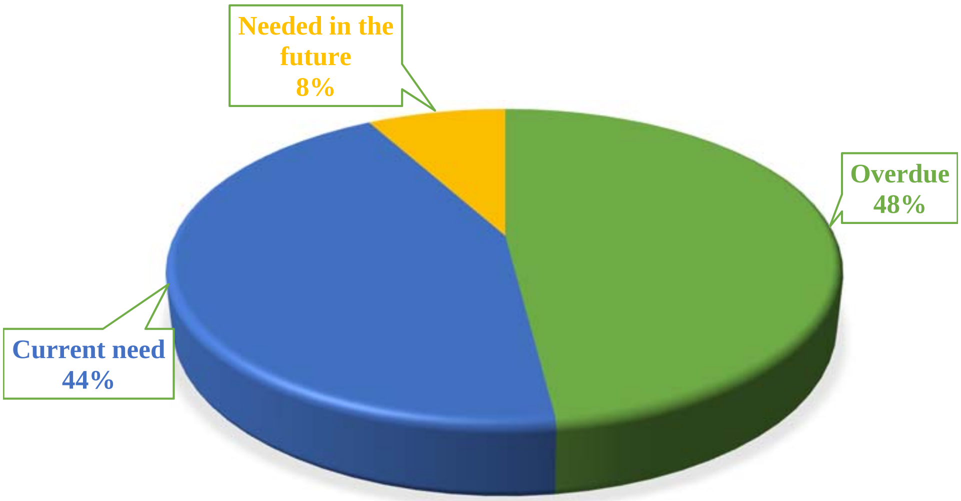

Figure 11: Timeliness of standardisation

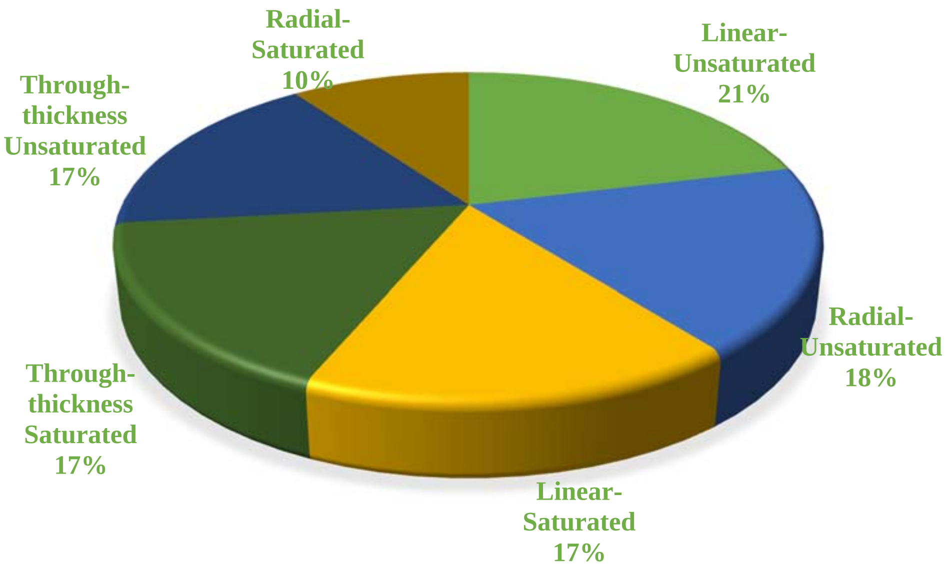

Figure 12: Standardisation priority

13 CONCLUDING COMMENTS

This report presents the results of an international survey of permeability measurement procedures used to support development and use of Liquid Composite Moulding processes. The purpose of the survey has been to collate information, identify measurement issues and generate a reference document for future international benchmarking, round-robin and standardisation activities. The survey results are representative and the number of respondents to the survey is satisfactory and of high quality, as sent to specific organisations that are expert in this measurement area.

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From the comments, it can be seen that there is a good level of communication between organisations (e.g. international collaborative activities and knowledge transfer). Recently, two international benchmarking exercises have been organised based on in-plane permeability measurements. Benchmark-I [1] compared all types of in-plane permeability measurement techniques, and Benchmark-II [2] focused on the linear-unsaturated permeability measurement technique by using the procedure developed in Reference [3].

In the survey, it is mentioned that more international collaborative exercises, like Benchmark-II, are needed on other techniques (*linear-saturated*, *radial-unsaturated*, *radial-saturated*, *through-thickness saturated* and *through-thickness unsaturated*).

In parallel to this international permeability survey, the National Physical Laboratory and the National Composites Centre will use the survey responses and Benchmark procedure to develop a draft for future standardisation.

14 ACKNOWLEDGEMENTS

The authors gratefully acknowledge the support and collaboration of the respondents to the survey. This survey is undertaken within a project investigating metrology challenges in composites processing funded by the National Measurement Office of the UK Department of Business, Innovation and Skills under the Materials 2014-2017 programme.

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