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A new ISO standard for the experimental characterization of in-plane permeability of fibrous reinforcements

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Abstract

During Liquid Composite Molding, a fibrous reinforcement is impregnated with liquid resin.

Process design requires knowledge of the reinforcement permeability for fluid flow, but

until recently, there has been no standard available for its measurement. In 2023, following decades of benchmarking activities and a standardization project, an ISO standard for the experimental characterization of in-plane permeability of fibrous reinforcements for liquid composite molding was finally published. It focuses on the experimental characterization of unsaturated in-plane permeability and specifies the requirements for test equipment, methods and data analysis. Given the deficiency of standardized procedures within the composites industry, this paper intends to provide an example of the steps towards standardization and summarizes lessons learned. It illustrates the research milestones that led to the establishment of the standard, promotes the standard by detailing its general content and notable features and finally gives explanations and reasoning behind the developed guidelines.

Keywords: A. Fabrics/textiles; B. permeability; C. Process Modeling, E. Resin transfer molding, E. Resin flow

1. Introduction

Liquid Composite Moulding (LCM) encompasses a broad range of processes used to manufacture fiber reinforced polymer composites (FRPCs) components, which are applied in various industries, including automotive, aerospace, marine, and energy. In LCM, a dry fibrous reinforcement is impregnated with a low-viscosity liquid resin system (viscosity typically $< 1 \text{ Pa}\cdot\text{s}$), which then cures to form the rigid matrix. Impregnation is driven by applied pressure (injection) or a vacuum (infusion) or both. Short cycle times and high robustness, i.e. fast impregnation and minimal risk of incomplete saturation, are typical targets of process design, requiring an understanding of material properties. For such processes, it has been shown that Darcy's law governs the impregnation progress [1]. It relates the phase-averaged flow velocity to the applied pressure gradient, the dynamic resin viscosity, and the reinforcement permeability for fluid flow. As per this definition, the permeability is an equivalent parameter assuming a homogeneous medium representing an intrinsically heterogeneous material. In addition, the permeability is a phenomenological parameter related to the geometry of the reinforcement architecture, with units of m^2 . However, there is no straightforward way of calculating it based on the micro-structure of the medium or through experimental measurement of the flow advancement within the reinforcement. Because of the directionality of the fiber arrangement in a reinforcement, the permeability is generally anisotropic and is described by a symmetric second-order tensor. Diagonalizing the tensor yields three principal permeabilities, corresponding to flow oriented along three orthogonal axes, two of which describe the in-plane permeability. For decades, researchers worldwide have tried to understand the influences behind variations in the measurement of reinforcement permeability. This research field has grown rapidly, with numerous experimental methods reported in the literature [2–4]. Experimental permeability characterization has played a crucial role, enabling systematic studies and

providing input for process simulation, which forms the foundation for process and material optimization. Despite the popularity of LCM processes and the crucial relevance of permeability, no standard for its measurement has ever been available, until recently. Due to the specific nature and architecture of fabrics, none of the existing standards focusing on air, water or vapour permeability of soils, packaging or clothing materials, were applicable or even adaptable. In the last decade, it has become clear that due to their simple working principle, permeability experiments were easy to set-up and conduct, but achieving a high quality and reproducible measurement was quite complex. The values extracted by researchers could differ by orders of magnitude for the same fabric as shown in the first benchmarking activity [5]. Lessons learned from the first benchmark highlighted the importance of the methodology used and identified important checks necessary to obtain consistent and reliable values. This was once again demonstrated by the next two benchmark studies which were conducted using the same fabric by 10-15 different research laboratories [6,7]. In July 2023, after three benchmarking studies and a dedicated standardization project, ISO 4410:2023 was finally published. The ISO standard provides clear guidelines for the experimental characterization of unsaturated in-plane permeability of reinforcing materials for LCM. It specifies the requirements for test equipment, test methods and data analysis to ensure optimal accuracy and reproducibility of results.

This paper is motivated by several factors. Firstly, the lack of standardization in characterizing the processing behavior of semi-finished products used in FRPC production is widespread. This poses a significant obstacle to their standardized and widespread industrial use. By illustrating the research milestones leading to the creation of this standard and the experiences resulting from the benchmarking studies and the standardization project itself, the authors aim to explain the reasons behind the methodology outlined and adopted in the published standard. They also aim to highlight the lessons learned that may be useful for

characterizing heterogeneous materials with an “averaged” homogeneous value. The second major intention is to encourage the adaptation of the new standard, as its true benefit can only be realized from widespread application. Therefore, in this paper, the standard’s general content, methodology guidelines, the rationale behind their adoption, along with notable highlights and potential pitfalls are presented. In addition, a discussion on transferability and usability is included, to point out possible applications in the context of LCM but also for other composite processing technologies. The third major intention is to explain the decisions made during the standard development process. Many of these decisions are not self-explanatory from reviewing the published results of the benchmarking studies alone, and the standard itself does not include any information beyond what is essential for its application.

2. Preliminary work: a brief history of benchmarking

Following the initial publication of Darcy’s work in 1856 [8], permeability of porous structures for fluid flow became an increasingly relevant research field, initially focused on flow in soils. It was only in the mid-1980s, when the works of Adams, Miller, Russel and Rebenfeld [9–14] marked the first milestones of systematic permeability determination within the context of LCM. A Scopus search for the keywords “Liquid Composite Molding” AND “Permeability” shows increasing interest in this field over the last three decades, with 20 to 40 new journal publications appearing every year.

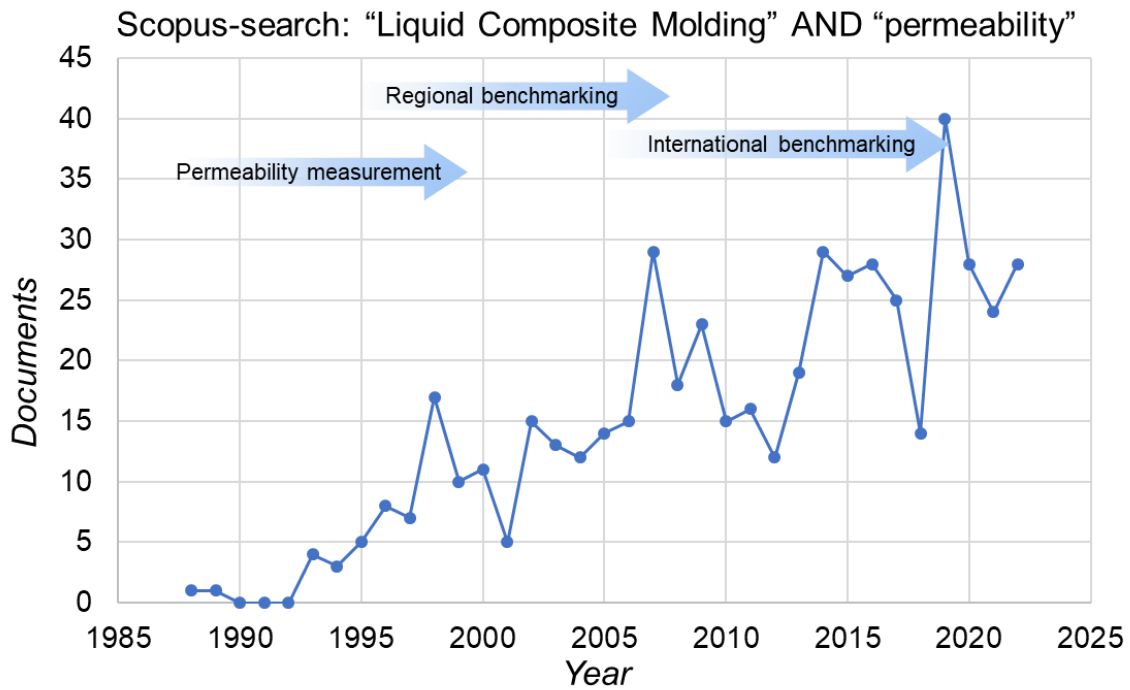


Figure 1: Development of the publication activity and important milestones towards standardization.

Simulation methods for resin transfer molding processes in complex mold geometries were developed in the early 1990s [15,16] fuelling the need for reliable methods of permeability determination. The next significant development occurred in 1995 with the publication of the first regional benchmark study on in-plane permeability [17], quickly followed by subsequent studies [18–20] and initial efforts to establish databases [21]. It became clear that differences in test equipment and methodology led to orders of magnitude variation in results, but the suspected reasons for these differences remained largely unproven. The next milestone occurred in 2006, when the research community of the conference series Flow Processes in Composite Materials (FPCM) organized the first international benchmark study, which initiated the momentum leading to the current ISO standard. In this exercise, initially presented at FPCM-10 [22] and published in 2011 by Arbter et al. [5], all participants investigated the same fabric, but no specifications were defined regarding the measurement method and the test parameters. One advantage of this strategy was that, in the context of technology screening, it provided an overview of all the methods used and

distinguished good practices from the usual pitfalls. One main difference identified was between unsaturated and saturated flow tests. In saturated tests, steady-state flow through the sample is generated and the pressure drop over a certain flow length is measured. In the so-called "unsaturated" linear flow experiment of a fluid into a dry reinforcement sample, one-dimensional flow develops. The resulting flow front advancement can be tracked. It must be noted that the term "unsaturated permeability" is rather unfortunate, as "transient" would be more appropriate to name this on-line measurement, which assumes a sufficiently sharp flow front, and usually neglects the effect of capillary pressure acting at the flow front. However, this terminology was established over decades by the composite community and retained for this reason.

Darcy's law, originally formulated for the saturated case, can be easily adapted to the unsaturated case, but this requires the introduction of several assumptions, either of a sharp flow front where capillary forces may act, or of multiphase flow considering air and fluid phases [23].

The second main difference was between linear and radial flow experiments. In linear flow experiments, a one-dimensional flow is generated. In radial flow experiments, the fluid is injected into a central inlet channel, typically resulting in an elliptical flow front. A scatter of the measured permeability values of more than one order of magnitude was the result of this benchmark. Due to the many technological differences, it was not possible to identify the ultimate causes for the variation.

Based on this experience, a second benchmark was organized, but this time with a mandatory predefined measurement procedure as specified by Alms et al. [24]. The participants were required to apply an unsaturated linear injection method to minimize variance and isolate the sources of variation, and to further investigate this very widely used method among the composite community. The results published in 2014 by Vernet et al. [6]

showed that by defining minimum requirements for the equipment, measurement procedure and analysis, satisfactory reproducibility of data (scatter below 25 %) using different systems could be achieved.

In 2016, United Kingdom's National Physical Laboratory and National Composites Center published a survey of procedures in use for permeability measurements in LCM [25]. In addition to demonstrating significant interest from industry in standardizing permeability measurement, the study also revealed that over 30 institutes were active in this research field, each with varying approaches within their respective groups. However, 2D radial and linear tests were identified as the most common strategies to characterize unsaturated permeability. With this in mind, at the 2016 conference in Kyoto, Japan, the FPCM community decided to launch a third and final international benchmark on in-plane permeability characterization. This time, the benchmark focused only on radial methods, defining even stricter guidelines and included an extensive questionnaire on the applied methods, test equipment and data evaluation techniques used. This allowed deeper insights into the possible sources of variation, e.g. tool cavity deformation was identified as the largest source of error [7]. After three consecutive international benchmark exercises, the community was confident that the time had now come to advance to the next level, i.e. standardization.

3. The ISO standardization project

3.1. Background

Standardization projects are initiated by national bodies. In early 2019, the topic was initially introduced to the German standardization body DIN (Deutsches Institut für Normung) through the DIN Standards Committee Plastics, as the Leibniz-Institut für

Verbundwerkstoffe (IVW), which had organized the last international benchmark exercise, already had a representative within this committee. The DIN committee decided to support the proposal and to approach the ISO committee ISO/TC61/SC13/WG1, which is in charge of 'Reinforcements and reinforcement products'. At this point, a proposal had to be prepared to convince the committee that the maturity level was sufficient and that a standard would bring relevant benefits to both industry and academia. In addition, it had to be proven that a sufficient number of experts worldwide could support the project. Regarding the latter, a list of authors of the three international benchmark exercises was submitted as the list of experts. In addition, a first (rough) draft of the standard had to be prepared, which was a compilation of the existing guidelines of the benchmark exercises II and III, complemented by some initial ideas for additional requirements. This proposal was submitted in late 2019 and had to undergo a ballot. The project's success depended on receiving a favorable vote from at least two-thirds of the ISO committee members, with active support commitments from a minimum of five participating members. In the end, 14 out of 19 members voted in favor, with five members choosing to abstain. Nine members committed to active participation and IVW's ISO delegate, was selected as the project leader. The project was allocated a duration of three years, which is a typical timeframe, and with pre-defined milestones. The project officially began in March 2020 and it is important to note that it was not supported by any dedicated funding, relying entirely on voluntary contributions.

3.2. Approach to develop guidelines for the ISO review process

Shortly after the start of the project, the 19th European Conference on Composite Materials was scheduled to take place in Nantes, France where a workshop on the topic was planned to take place. However, due to the Covid-19 pandemic in 2020, this was postponed, and a digital substitute program was put together by the organizers. Within this framework, a kick-

off workshop was organized, to promote the project and to attract collaborators. Prior to this, working groups (WGs) and their respective leaders were identified (Figure 2). During the workshop, the working groups were introduced and their content discussed, with over 90 participants invited to join. In the following weeks, these groups established themselves and began to conduct studies in order to find answers to open questions, decide on guidelines and finally elaborate on the standard content.

The group leaders and some experienced researchers formed a core team, which met quarterly, to coordinate work, deal with overlapping contents and set-up joint meetings of two or more groups. This core team also took final decisions, whenever disagreements could not be resolved within a group and compromises had to be found. Contributions of the different working groups were regularly merged into a unified standard draft document.

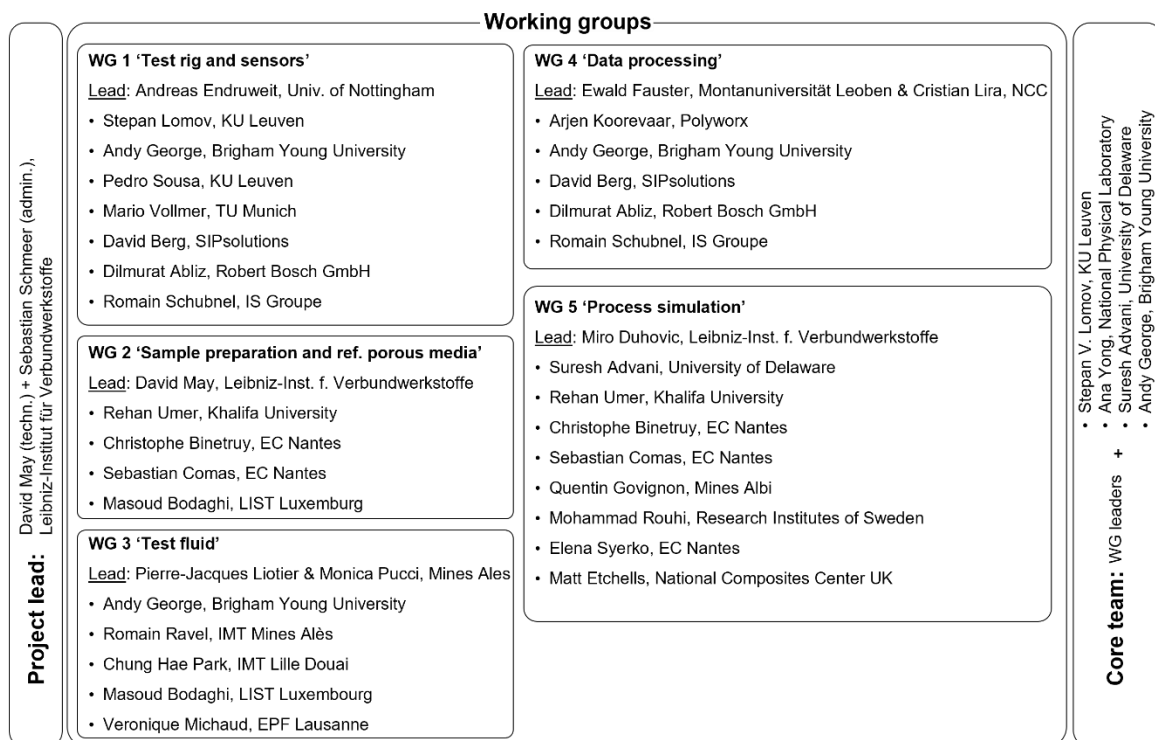


Figure 2: Organizational structure of the working groups for the ISO standardization project

The topics addressed within the working groups were as follows:

- WG1 ‘test rig & sensors’ dealt with the test rig as a physical system, i.e. sample size, surface quality, procedures for deflection control and several other questions of practical relevance when building a testing device. A particular focus was on type, position and number of sensors and prerequisites for their accuracy etc.
- WG2 ‘sample preparation and reference porous media’ prepared procedures for cutting, stacking, weighing, storing and possibly sealing of samples. Furthermore, potential candidates for reference materials were evaluated, which could allow plausibility checks when constructing new test rigs based on the standard.
- WG 3 ‘test fluid’: For permeability determination in the context of LCM, Darcy’s law has been extended to unsaturated flow or transient flow, neglecting the effect of dynamic wetting. The task of this WG was to discuss how the errors caused by this simplification can be minimized e.g. by selection of an adequate test fluid. Numerous studies on potential fluid candidates were performed.
- WG4 ‘data processing’ turned out to be the most work-intensive topic, as many other topics, such as definition of legitimate sensor arrangements and sample sizes strongly depend on the requirements defined for amount and quality of data to be captured during a test. The tasks also involved procedures for data evaluation and segmentation, validity criteria for tests as well as selection/definition of strategies and algorithms for permeability calculations based on the captured test data. Furthermore, guidelines for reporting and averaging of permeability values were the focus.
- WG5 ‘process simulation’: Permeability is often determined to generate input for process simulations. The task in this WG was to discuss and help set the basic guidelines/rules of thumb/best practices for the recording and use of experimentally

measured in-plane permeability data for the numerical simulation of resin injection or infusion processes.

The initially submitted draft was revised extensively in the first year of the project and submitted to the committee in April 2021. The national ISO experts provided their input and comments on the draft, which were addressed with a response letter, including comments on all changes made to the document. The committee after examining the revised document voted and suggested further changes. Following ISOs standard project scheme, the second draft was submitted in April 2022 and after further edits, the final draft was submitted in April 2023. The complete project with all relevant dates and milestones and can be found here: <https://www.iso.org/standard/79944.html>

The first draft was more concerned about the technical content, whereas the latter drafts focused more on formal correctness, e.g. if the specific “standard language” was used. The technical changes required specifying details. For example, many validity criteria, such as the maximum variation in injection pressure within a test, were already formally described, but not quantified by a fixed number. Eventually, the standard was passed from the committee and sent to the ISO secretariat for final checking. At this point, no more changes to the technical content could be made.

4. Contents of the standard

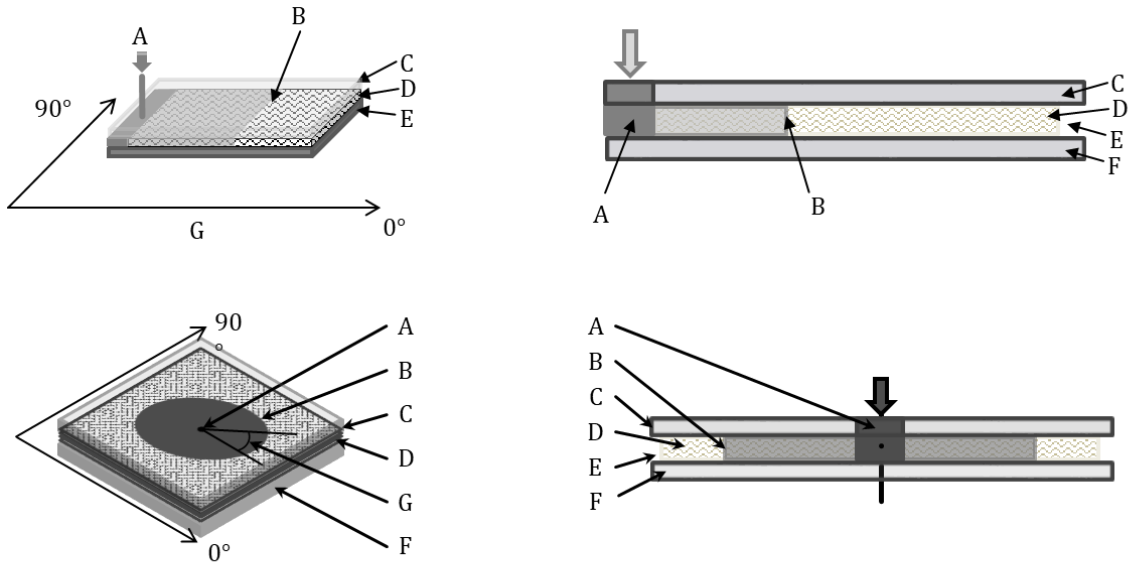
It is not intended to reproduce the detailed contents of the standard here. However, as the standard itself does not provide an opportunity to explain the many decisions made to develop the final content of the document, insights and highlights of particular interest from a scientific point of view are summarized in the following sections.

4.1. Sources of variation and their management

The standard focuses on the experimental characterization of in-plane permeability of reinforcing materials for LCM. During such experiments, three general sources of variation arise: **methodological errors**, **systematic errors** and finally **statistical variation** which is a nothing to be prevented but rather a material property to be characterized.

4.1.1. Methodological errors

If many experimental methods are approved by the standard, minimizing methodological errors caused by different experimental methods becomes more and more difficult. Therefore, restricting to preferably one valid method becomes highly meaningful, though it naturally causes disagreement over which method is the best choice. The decision to choose the radial and linear injection approaches, depicted schematically in Figure 3, was based on their relevance and central role in the benchmarking activities [25], which provided the required experience and data.



- A Fluid inlet
- B Flow front tracked
- C Top plate
- D Specimen
- E Vent region
- F Bottom plate
- G Orientation angle

Figure 3: Schematic illustration of the linear (top) and radial (bottom) injection approaches, selected by the standard

For similar reasons and given that in LCM processes the resin enters an unsaturated mold and saturates the reinforcement, there is interest in understanding the permeability of the fabric during this saturation process. Therefore, it was decided to focus on unsaturated testing conditions, which will be further discussed in Section 4.3. From the start of the project, it was intensely discussed if the standard should focus on radial or linear injection only, but this idea was eventually discarded as both methods provide unique benefits and have the potential to show high reproducibility as proven in the benchmark exercises. Unsaturated testing requires flow front monitoring and many different technologies have been used for this in the past. The standard is not restrictive in terms of the sensor technology used. However, it defines the number of data points to be captured and also the required accuracy of the sensors. As optical flow front tracking is wide-spread and the obligatory transparent molds are known to be deflection-prone, a distinct procedure for deflection measurement and a maximum allowed deflection were defined. Adequate flow front tracking also requires that the flow front has fully formed and that it is tracked over sufficient time and flow length. Therefore, requirements on the sample size were defined. To minimize wall effects, a minimum cavity height and number of reinforcement layers are also specified.

4.1.2. Systematic errors

Systematic errors inherent in these methods are minimized by introducing well-defined procedures for preparing and executing the flow experiments as well as for post-processing the acquired measurement data. For example, the benchmark exercises have shown that results can depend on where the injection pressure is measured, but also how it is used in the calculation for data reduction. Due to the direct effect on the calculated permeability, the standard now exactly defines how to cut off run-in effects and to time-average the effective injection pressure and temperature. Furthermore, it is well-known that the injection pressure

can cause variations, e.g. through different amounts of fiber movements. Therefore, a maximum injection pressure was defined. A further example is how the temperature-dependency of the viscosity is included in calculations, i.e. constant based on a time-averaged temperature or adapted to each time step's temperature.

4.1.3. Statistical variation

Statistical variations are dominated by (local) variations in material properties, particularly inhomogeneous areal weight distribution and thus, fiber volume fraction of the reinforcing materials. The standard therefore defines how data should be reported, e.g. how to average results from multiple experiments at repetitive conditions or how to approximate a dependency on the fiber volume content via the Kozeny-Carman [26] phenomenological equation. Mandatory information on the material, the sample, and the test design is defined, so that further usability of results reported in literature is ensured.

4.2. Highlights

4.2.1. Validity criteria

The standard was developed to be widely applicable. Therefore, it is generally not restrictive in terms of the fiber materials that can be investigated. Of course, some fibrous reinforcements might cause unwanted effects, e.g. unacceptable flow front distortion due to structural heterogeneity. To address this issue, a set of validity criteria was developed to check whether the application of the prescribed mathematical algorithms for permeability calculation is appropriate. This included thresholds for the maximum allowable variation of pressure and temperature within the test range (Figure 4, left). One of the most important criteria is to consider the validity of flow front geometry for measurements. For example, during linear injection, higher or lower permeability at the edges of the preform parallel to the flow direction

causes the flow front to speedup (race-track) or slow down along the edges [27–29]. This usually causes over- or under-estimation of the measured permeability. At the time the standard was prepared, no clear pass-fail criterion for a permeability test affected by the effect of race-tracking was available, i.e. it was not clear how to decide when race-tracking has caused too much error. As part of the project, a task force was appointed to address this issue. Hundreds of flow simulations were carried out, varying the sample geometry, the edge conditions and many more parameters. By comparing the respective measured and true permeability (which is known as it is a simulation input) several candidates for a pass-fail criteria were compared [30]. Finally, a simple comparison between different flow front positions (Figure 4, right) was used as the basis for a model to predict the measurement error as a function of a small number of relatively easily accessible experimental parameters. The error is evaluated for each time measurement and compared to a critical threshold that is again linked to specimen length and permeability values.

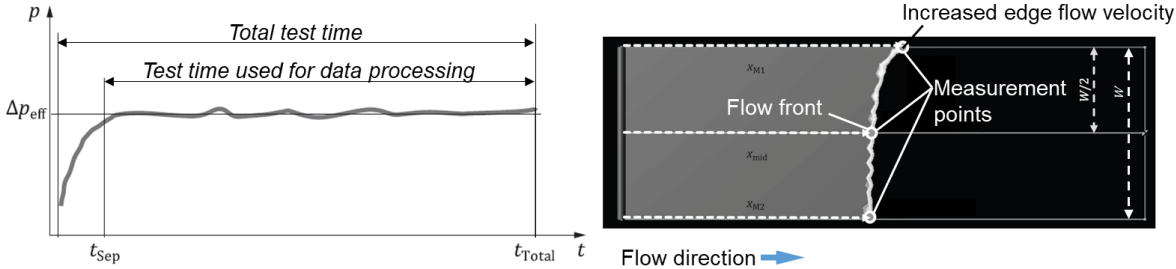


Figure 4: Typical characteristics of fluid injection pressure as a function of time (left) and averaged effective pressure Δp_{eff} to be extracted, example for flow front location measurement to calculate extent of race or slow tracking (right)

For radial injection, race tracking is less of a problem but still the quality of permeability calculation is very much dependent on the adherence of the flow front shape to the elliptical shape assumed by the algorithm. In this case, measurement validity is checked by evaluating the fitting quality of the elliptical paraboloid fitted to the entire flow front data over time using a unified data processing approach.

4.2.2. Unified data processing

Reliable and accurate data capture and appropriate selection of the data subset for further processing are key for minimizing variance. However, even two sets of the exact same data can give very different permeability values, if different calculation approaches are used. This includes the flow front approximation, the algorithms for calculating the permeability [9,10,12,13,31–33] and the general strategy with which the algorithm is applied to the data (e.g. elementary, reference time step, single step and global method according to [34]). To minimize any possible variation connected to data processing, a universal processing strategy was defined. This was straightforward for the linear injection method, where the entire permeability can be calculated based on the slope of the squared flow front progression over time, Figure 5 (left), given that a constant injection pressure is applied.

For radial injection, there is even greater potential for variation due to the complex shape of the flow front, needing more detailed mathematical considerations. As shown in Figure 5 (middle), different sensor technologies, which are in principle all allowed by the standard, give very different data sets. Here, the vertical axis corresponds to the time, horizontal x and y axis represent the flow front progression. A unified approach must be capable of processing all types of data. Eventually, an algorithm based on the fitting of an elliptical paraboloid to the complete data set in the (x,y,t) space was chosen due to its high degree of robustness (see Figure 5, right).

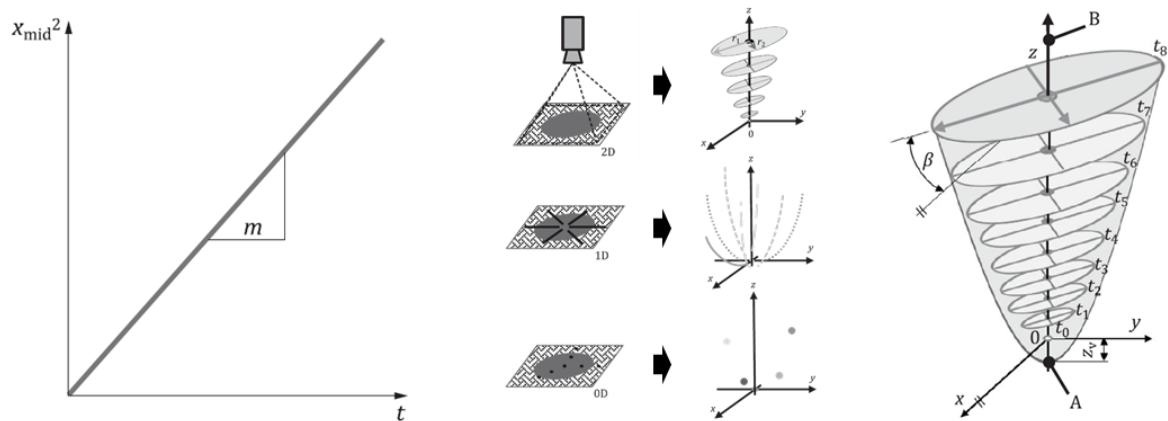


Figure 5: Squared flow front over time as basis for the permeability calculation from linear injection (left); different types of flow front capturing technologies and corresponding data sets (middle); universally applicable elliptical paraboloid fitting to the entire flow front measurement data (right)

4.2.3. Plausibility checks

The authors of the standard hope that the new standard will be widely accepted and that it will lead to an increased number of test systems in use worldwide. Those already engaged in research in this field are well aware of the numerous challenges that must be addressed before a reliable system can be achieved. In all benchmark exercises, outliers were found. The extreme deviation in the results of the remaining research groups indicated that one or more small but critical errors still remained undiscovered. This could simply be a straightforward error in unit conversion. To support those developing new systems or modifying existing ones to meet standard requirements, provisions for plausibility checks were incorporated. The foundation for this is a reference porous media concept, an idea which has been revisited consistently since the beginning of benchmarking activities. Textiles [21,35–38] as well as 3D-printed structures [39,40] were considered as reference materials. Using ISO’s relatively new feature to include permanent links to a data storage, CAD-files for a 3D-printable structure were included (<https://standards.iso.org/iso/4410/ed-1/en/>). The structure, presented by Bodaghi et al. [41], mimics a textile in relevant features

and can be easily adapted to the specific size of the respective test rig. Depending on the printing method used, precision close to the benchmark requirement can be achieved [41,42], but even simpler 3D-printing methods can be used to at least check for plausibility of the values.

4.3. Explanation and reasoning behind the ISO guidelines

Table 1 gives brief explanations on the most relevant decisions that were made through-out the standardization project. In general, the project team was committed to two simple, yet important guidelines:

- The build-up and use of systems adhering to the standard should be cost-efficient and achievable with a minimum of training, to pave the way for broad use of the standard. Every decision striving for minimization of the above-mentioned types of errors must be balanced with this objective.
- The broader the technological variety, the greater the risk of variation and the more extensive and less user-friendly the standard will be. Wherever possible, variability must therefore be reduced.

Table 1: Explanations of decisions made in the standard development

Topic	Specification	Explanation / Outlook
Context of permeability measurement	The standard is focused on permeability measurement in the context of LCM	Permeability is relevant for various composite manufacturing processes. However, the material and process conditions can be very different from LCM. Permeability in the context of LCM is certainly the most advanced research field in polymer composites manufacturing and focusing on LCM has allowed one to draw from this extensive expertise as well as the benchmark studies. Nevertheless, this standard can be very useful for other process groups, as further discussed in section 5.
Injection method	Measurements are based on unsaturated flow conditions, i.e. a	It is well-known that most textiles processed in LCM provide a distinct dual-scale nature, i.e. flow within the fiber bundles differs strongly from flow between the bundles. This is a result of the very different sizes of

<p>progressing flow front.</p>	<p>the flow channels between the fibers. During unsaturated flow, depending on the value of the capillary number, flow separation takes place, with either flow leading in the tows, if capillary forces dominate, or flow leading between the tows, if hydrodynamic forces dominate [23]. Therefore, capillary pressure may affect the flow propagation [43–48]. Measurements under saturated flow conditions bypass these effects and thus represent indeed the geometric permeability of the porous medium. However, LCM is affected by these effects, and many have developed the so-called unsaturated permeability measurement as a practical representation of the current practice and potential flow separation effect. The standard was intended specifically for this case, and to respect the common practice already established in the composite community. Therefore, bypassing them for the sake of measurement accuracy was regarded as the less attractive option. Furthermore, the benchmarking studies, which partially involved tests of both types, have shown that by proper selection of fluid pressure and viscosity the influence of capillary forces can be reduced to a negligible effect. Several other studies have highlighted that since permeability of textiles in composite processes is dominated by the presence of the larger spaces between the tows, the so-called unsaturated permeability is close to the saturated one if measurement conditions lead to hydrodynamic forces leading flow, so a sufficient flow velocity. Therefore, it was decided to focus on unsaturated test conditions, but define ranges for injection pressure and viscosity. Also, it was defined how the pressure is to be measured and controlled, to make sure these conditions are comfortably met. Integrating both, saturated and unsaturated methods, in the standard was discarded at a very early stage, simply to reduce complexity.</p>
<p>Radial and linear injection</p>	<p>Radial and linear injection have shown to be by far the most popular methods for in-plane permeability, according to a study conducted in 2016 [25]. Therefore, the last two benchmarking studies focused on these methods and the results did not indicate that any other methods could bring relevant improvements. Both methods show unique advantages and for both high repeatability could be proven by benchmarks and therefore it was decided to include both of them in the standard. 3D injection methods are occasionally reported in literature [49–52]. While the very limited data of the benchmarks show that they can give equal results when implemented properly, they are generally more sophisticated and therefore less suitable for a standard targeting easy and robust implementation for labs with limited expertise and industry.</p>

	Constant injection pressure	The mentioned survey [25] also revealed that most systems use constant injection pressure throughout the test. Very few systems have been built on the principle of constant flow rate and in the last benchmark study only one such system participated. The results were not conclusive as there is simply not enough data to make a statement about the implications. This method was therefore omitted for the sake of simplicity.
Definition of terms	Terms such as in-plane permeability, unsaturated flow, in-plane anisotropy ratio, linear/radial injection, race-/slow tracking as well as orientation angle are defined	Some frequently used terms have been defined quite differently in the literature, e.g. an exact definition of in-plane anisotropy and race-tracking is hard to find. A benefit of the standard is that it provides definitions and it was decided to do so for all terms relevant for the standard. The definition of permeability in particular was discussed controversially. Darcy's law originally describes saturated flows in very homogenous porous materials. For LCM it is adapted to unsaturated flows in porous media with dual-scale nature, known to be possibly affected by the capillary number. Our definition of permeability is accompanied by notes, that point out that this means wetting effects are neglected and that our permeability is defined at the level of an equivalent homogenous medium representing an intrinsically heterogeneous material. The standard prescribes boundary conditions intended to ensure that these assumptions are meaningful. The possibility of using alternative terms like "effective" or "apparent" permeability was discussed, but ultimately dismissed. Permeability is a widely used term and used for all kinds of different concepts, many of them neglecting effects that were not originally considered. Including "effective" will not make this any clearer. A permeability measured according to the standard is a permeability according to the definition given in this standard.
Fiber materials and textile types	The standard is non-restrictive in terms of fiber sizing or type as long as no relevant interaction with the test fluid (e.g. fiber swelling) is expected	Darcy's permeability does not allow for any interactions between fluid and fiber, e.g. capillary forces are neglected and fiber swelling would change the fiber volume content throughout the measurement. The standard defines no measure for when interaction becomes relevant. There is simply not enough information to make such a statement. Yet, the standard defines limitations for inlet pressure as well as flow front variation and as long as these are met, the standard is applicable. There is currently no solution for cases where this simplification cannot be met. It was decided that for the moment, raising awareness that such effects could occur is the most meaningful statement. Another challenge is that textiles can be very different. For example 3D-fabrics, i.e. fabrics with rather complex three-dimensional geometries such as I-profiles, will have locally strongly varying

		<p>permeability. Application of this standard is basically only possible for flat, quasi-homogeneous regions of these fabrics, while localized geometrical features will have severe influence on the overall flow front pattern. Further research on these specific materials might make additional specifications necessary in future, leading to an update in the version of the standard.</p>
Minimum number of layers	A minimum number of four layers is defined.	<p>Due to nesting effects, flow between textile layers differs from flow between the sample and the mold wall [53]. Therefore, the number of layers can have an influence which is presumably negligible if the number of layers is not too small [54]. Naturally, this threshold number is very material-specific. Selecting a very high value to be on the safe side was not an option, due to practicality. The value of four, chosen in the end is a compromise, based on the experience of those involved. It results in a ratio of at least 3:2, textile/textile flow regions to textile/wall flow regions.</p>
Fluid injection pressure	A maximum injection pressure of 0.3 MPa is defined.	<p>The higher the injection pressure, the greater the risk of relevant tool deflection and textile deformation [55]. At the other end, excessively low pressures could make the tests very slow and inefficient and, even worse, lead to relevant influence of capillary effects [45,46,56,57]. The chosen value is based on the experience of those involved and the results of the benchmarking studies. No minimal injection pressure was defined, but the possible influence of capillary effects at low injection pressures is emphasized in the standard. In order to reduce the variances introduced by capillary and wetting effects, it is essential to gain a more comprehensive grasp of these effects in the future.</p>
Test fluid	Test fluid must be quasi-Newtonian and incompressible with a viscosity between 70 and 200 mPas.	<p>Contrary to the initial intention and despite extensive studies [56,57], no final decision could be made on a standard test fluid. Several candidates were identified and evaluated, but none were fully convincing or universal. The idea of a universal test fluid generally seems questionable, as the wetting characteristics will not only depend on the fluid but also on the fiber sizing composition. In the benchmarking studies there was either no specification or silicone oil was specified, which, however, is known to have wetting properties very different from those of typical thermoset resins. Some examples of frequently used fluids are given in a note in the standard, to support possible newcomers to the field. In the end, only a viscosity range was defined, based on the experience of those involved and based on the results of the benchmark. One could argue that given this lack of information the more honest solution would have been to go without any restrictions, but this ultimately would have meant that even extremely high or low viscosity test fluids would</p>

		<p>be eligible. This, however, would definitely introduce more sources of variation and undetermined implications. Concerning the wetting characteristics, dedicated studies are still ongoing to discriminate the relative influence. The pressure fluid viscosity, and geometry conditions are such that the dual-scale flow should remain minimal to neglect these effects. However, care should be taken when running the experiments to note the presence of dual-scale flow, related to both potential capillary and viscous effects, and keep its potential effect in mind.</p>
Cavity deflection	Cavity deflection is limited to 2% of the target mold height.	<p>The last benchmark showed that tool deflection is most likely the single most relevant source of variation. Therefore, it must be minimized, keeping in mind that a too strict limitation will strongly increase the effort and cost of setting up a permeability measurement system. With the 2% threshold originally defined in the benchmark, an acceptable comparability of results was reached. About half of the participants managed to adhere to this limitation so that it also seems a realistic target. The effect of deflection is mainly a result of the fiber volume content changes it induces. Therefore, the threshold was defined relative to the total mold height instead of using an absolute value.</p>
Surface roughness	All surfaces in contact with the sample must at least correspond to IT10 tolerance in the ISO 286 international tolerance classification.	<p>This value was chosen after discussion with LCM tooling experts from industry, which named this as a typical requirement for RTM tools.</p>
Sensor accuracy and positioning	For pressure and temperature measurement the position and accuracy is defined.	<p>The benchmark studies revealed that sensors, if used at all, are often positioned in a manner that introduces significant deviations from the actual value of interest. For example, the pressure sensor was often positioned either at the pressure vessel or in a tube, some distance away from the inlet, where the exact value needs to be determined. Pressure losses within the tube were calculated for some cases and based on this it was decided to make its positioning with a T-fixture directly at the inlet mandatory, to avoid any measurement errors. The accuracy was defined after reviewing the market availability of sensors and taking into account the pressure range to be covered.</p>
Specimen dimensions	Linear: Length between 300 and 600 mm, width between 100 and 200 mm (preferably 150 mm)	<p>Linear: The range of allowed dimensions affects the reproducibility of tests performed with the standard. Larger samples have a higher risk of deflection, very narrow molds would have undetermined edge effects. In any case, the sample must be long enough so that a straight flow line will emerge. The extensive simulation study performed to define validity criteria</p>

		(see section 4.2.1) was also used to investigate the effect of sample size and influence of race/slow tracking. Within the given range the study shows that the validity criteria deliver meaningful results. For all other values, the effect could be significant and unpredictable.
	Radial: Edge length > 30 times the inlet channel radii, possibly more for very small values of $\alpha = \frac{K_2}{K_1}$.	For practical reasons the inlet in radial injection is always circular, but due to anisotropy most tested materials will eventually have an elliptical flow front shape. This shape must be fully developed before evaluation can start and a meaningful testing area must be covered. The standard defines minimum ratios of sample edge length to inlet radii, depending on the materials permeability anisotropy. For typical anisotropies this will lead to sample sizes far below 500 x 500 mm. All systems used in the last benchmark were within this range, reinforcing the argument that this is a practical and realistic requirement.
Data segmentation	Data from the initial pressure build-up phase is excluded from the evaluation.	The constant injection pressure set-up is known to have a run-in phase where the pressure builds up. As in many experiments, this phase is not representative of the conditions required to determine the correct permeability. Therefore, it is excluded from the evaluation.
Data sampling	Linear: At least 10 flow front positions, and corresponding temperature and pressure measurements. Radial: At least 9 flow front positions, and corresponding temperature and pressure measurements.	It is obvious that a minimum number of sampling points must be included in the standard in order to account for statistical variations and increase test robustness. Consideration was given to the fact that, aside from optical test methods, each sampling point adds to the sensor costs. The chosen numbers are based on experience and statistical considerations.
Documentation	Mandatory information is defined, that must be given in scientific publications or any other forms of publications in order to present results according to the standard.	Despite all the limitations and restrictive guidelines the standard imposes, relevant variations are of course possible. Therefore, the standard defines which information is to be reported so that possible reasons for variation can be identified and are traceable. The standard also includes an optional section on how to document repeat measurements. This provides guidance for the typical use case of a number of measurements at different fiber volume contents, where an interpolation is intended to correlate permeability and the fiber volume content. The Kozeny-Carman model is recommended for this purpose, based on the reasoning provided in [26].

All these decisions were made based on the available know-how existing at the time, balancing the desire for scientific accuracy with the practical need for an easily implementable standard. Naturally, discussions were extensive, often controversial and sometimes simply the minimum that could be agreed upon. With this standard as a basis we hope that permeability measurement as a research field will continue to grow and lead to further insights. From the standard's life cycle (<https://www.iso.org/standard/79944.html>) one can see that its publication does not mark the end of the work on the standardization. The next stage would be a review. This would typically occur after many years in use, allowing practical application experiences to be incorporated into the standard. Until then, the document can be used to deepen understanding of LCM impregnation phenomena in order to improve the standard in areas, which are currently left unaddressed due to a current lack of information.

5. Discussion on usability and transferability

The main application for the standard in the context of LCM will be firstly initial permeability determination for material selection and secondly input generation for process simulation. Of course, higher permeability can allow for faster processes, and if all other properties are similar, it will maximize process efficiency. On the other hand, in industrial applications textile layers are mostly stacked to multi-layer laminates, almost always with different orientations and often combining different materials. Here, a homogenous permeability distribution is needed to impede excessive differences in flow velocity, that can result in voids. Process simulations can be used to optimize process design and material selection and permeability is one of its key inputs. The standardization project team had a working group focusing solely on the question of specific requirements that might result from the use of the permeability values in simulations. As a result, the standard generally allows for single

materials testing and virtual stacking in a multi-layer simulation, but also for 2D shell simulations based on tests of the complete multi-material/orientation stack. Validity criteria must still be met to ensure that the data processing remains accurate and the homogenized permeability is truly representative of the reinforcement stack.

In general, it is important to note that the saturated, macroscopic level Darcy-based modelling of flow, neglecting capillary effects, as used to extract permeability in the standard method is a practicality-driven approach. Literature reports fluid flow models, relying on a more complete approach to permeability, that are, from a scientific perspective, clearly superior especially when it comes to accurate depiction of the dual-scale nature of porosity [58–61]. Two main approaches have been described in the LCM literature. First, a direct solution of the multiphase flow equations can be established. For this, it needs to be known how the permeability varies with fluid saturation and how saturation varies with pressure [23,61,62]. Secondly, solution of saturated flow but with the introduction of sink terms has been reported, to take into account the two scales of impregnations, but this requires knowledge of the flow kinetics within the tows [61]. Corresponding tools to simulate multiphase/multiscale LCM flow are already being developed [58,63]. Such models are key for the improvement of the physical understanding of LCM processes. Nevertheless, the convenience correlated with the conventional saturated Darcy-based modelling led to it currently being clearly the most dominant approach in use. However, neglecting sink effects in tows causes an inherent error, which must be critically reflected upon when applying values generated following the standard, in particular if the fabric has large separation of scales [64], or if the flow is clearly dominated by capillary effects [65]. The guidelines defined by the standard minimize errors in the determination of the unsaturated permeability as a geometric parameter. However, in the case of significant resin cure simultaneous to the considered flow and/or when non-isothermal flow is given, the dual-scale nature implies effects that the conventional model can

simply not cope with. This must always be kept in mind when applying unsaturated permeability values for flow simulation in LCM.

With the standard published, a natural next step would be to start collecting permeability values within a database. Corresponding initiatives have been started in the past but the lack of comparability was always the main hurdle. This hurdle has now been cleared, and with such a database in place, LCM simulation could gain popularity, potentially boosting the adoption of LCM processes in industry.

While application of the permeability values in the context of LCM is rather straight-forward, it is not so clear for other composite manufacturing processes. Permeability is a material parameter, which does not depend on a certain process. However, there are some obstacles for its direct use in other fields:

- Thermoset impregnation, as given in LCM, is also present in autoclave processes or LCM variants with only one-sided rigid tooling, such as vacuum infusion. The permeability measured with the standard is not different from what is of interest here, but it is not sufficient to fully describe the textile behavior in the process. This is because of significant textile deformation. To make proper use of the permeability the exact fiber volume content at different locations with respect to time needs to be known, which requires sophisticated compaction models and separate characterization.
- Pultrusion and wet winding are processes which are typically thermoset-based and involve corresponding impregnation flows. Besides the fact that the aforementioned deformation effects are an issue here as well, in both processes fiber bundles are directly processed. The standard was developed for flat, areal materials and not for strand-like materials and therefore offers no solutions on how to measure them.

- In principle the permeability values are also valid when impregnating textiles with thermoplastics. However, due to the orders of magnitude higher viscosity, some physical effects such as reinforcement deformation will be even more pronounced. A further challenge inherent to thermoplastics is extensive shear-thinning, thus the assumption of quasi-Newtonian behavior typical to thermosets is no longer valid. Therefore, understanding permeability alone leaves researchers with many unknown variables, although significant differences in impregnation behavior can certainly be explained by very different permeabilities.

Finally, permeability is of course a concept used in many more fields than just composite manufacturing. For some of these fields, standards have already been developed. For the rest, the standard could provide a useful reference. Participants of the benchmarks have reported use of their permeability test rigs e.g. to test impregnation of clothing for laundry machine simulation or flow in battery materials. It is not possible to make a general statement about the usability of the standard for such cases, but whenever in-plane flow of incompressible fluids in textile(-like) fabrics is given, it seems a very viable option.

6. Summary and conclusions

Several closely related benchmarking activities are currently ongoing: Two rounds of international benchmarks have been conducted on out-of-plane permeability, compressibility and also virtual permeability prediction in the last years. In each case, the first rounds of results have already been published [66–68] while the second rounds have been completed but have not yet been published. In all three cases, it is to be decided what the next steps will be. In this section, we would like to share the following insights to support the scientific community in these and other future standardization efforts.

- A successful benchmarking process is essential to initiate the standardisation project. The benchmarking progresses through three stages: Initially, measurements are conducted using various procedures defined by the participants themselves. This typically yields widely scattered results, but provides a screening of available methods. Next, normalization rules are proposed and demonstrated to ensure consistent measurements. Finally, a round with strict rules confirms that these can serve as the foundation for the standard.
- It is important to accept that decisions must be taken, and compromises must be made. Not every question will be answered to the full satisfaction of everyone involved and sometimes a best guess is better than no value at all. The boundaries for the viscosity are a good example. To some extent, the selection of a specific maximum and minimum value is somewhat arbitrary. However, not defining limits would mean that the standard would e. g. allow for permeability tests to be performed with extremely high or low viscosity test fluids. In this context, our strategy of working groups and an additional core team for final decisions proved to be very effective.
- In addition, it is important to aim for a practically feasible minimization of variability. If requirements become too strict, the application of the standard might become impractical because the required expertise and financial investment become too high, or each individual test becomes too costly. Acceptance in the community would be compromised, thus falling short of the standard's initial objective.
- It is not expected in a standard to justify or critically evaluate the selection of methods. It is more like a recipe, containing only the normative content needed to prepare it. There are only very limited possibilities to provide informative content. We tried to use these opportunities to inform users of the standard about helpful best

practices, which are not mandatory but can support newcomers to permeability measurements.

- A specific style of language is used in the standard to clarify which aspects of the described procedures are mandatory and which are only recommendations.

7. Acknowledgements

The project has benefited significantly from direct contacts to the DIN and ISO committees. We were researchers navigating the uncharted, with practically no previous ISO experience. The first-hand information and the many years of standardization experience of our official project leader Sebastian Schmeer were a valuable support, even though his research background is on mechanical testing and not permeability. This allowed us to concentrate solely on the technical aspects of the standard.

The standardization project only brought to an end what other researchers started many years ago and have helped drive forward over several decades. The authors would like to acknowledge all organizers and participants of the three international benchmark studies on in-plane permeability determination and especially the scientific community of the conference series Flow Processes in Composite Materials (FPCM).

8. Literature

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