# Methodology for permeability coefficients determination using the vacuum infusion technology

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**Abstract.** The article describes a special installation allowing real-time determination of the pressure drops within the impregnation process. The installation working zone is a flat permeameter, where the reinforcing material under study is laid, and liquid is supplied. Ultra-low pressure membrane sensors were used to register pressures along the fluid motion direction. Diagram of the sensors installation is provided, as well as substantiation of selecting a site positioned between two adjacent sensors, where it is advisable to conduct the experimental research. Based on the data obtained, the permeability values coefficients were calculated. The article presents results of assessing the accuracy of the proposed method to determine the permeability coefficient values, which was obtained by the authors in comparing experimental and theoretical duration of the impregnation process.

## 1 Introduction

The polymer composite materials (PCM) application areas are constantly expanding, which is associated with a complex of their unique characteristics [1–4]. Pre-preg technology is traditionally used in the PCM production, but today there appears a tendency to replace the pre-preg technology with the direct molding methods, such as vacuum infusion and pressure impregnation [1, 2]. Impregnation is the key technological operation in these processes. Main advantage of the direct molding methods over the pre-preg technology lies in the low cost of the finished product.

In parts manufacture from PCM using the vacuum infusion technology, as a rule, thermosetting binders are used [5–14], but recently inorganic binders are introduced [15], and composites based on the thermoplastic materials are becoming more widespread [16, 17].

Main disadvantage of the direct molding methods lies in the lack of a quality control technique at the impregnation stage. It is impossible to determine the impregnation quality before curing, which leads to defects, requires additional optimization of the technological process and increases cost of the manufactured parts. To optimize the technological process, experimental methods are most often used to assess the impregnation process kinetics [9-12], or numerical simulation methods are applied. The numerical methods

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accuracy depends on the approximation degree of the initial data to the real values of parameters of the simulated objects and processes.

When simulating impregnation of products made of the polymer composite materials, the permeability tensor is generally such a defining input parameter; in a more particular case, the package permeability components in the impregnation plane are playing this role. For certain materials, manufacturers inform on the permeability values, but reference data on these parameters for various package structures, especially taking into account the material chemical nature, are missing.

This work objective is to develop a technique that makes it possible to experimentally assess the permeability coefficient values depending on the chemical nature of the reinforcing material and its structure.

## 2 Research objects and methods

An installation was developed for experimental study of the impregnation kinetics, i.e. a flat permeameter making it possible to register technological parameters of the impregnation process for application of the Darcy's law in the one-dimensional formulation. To register pressures along the fluid motion direction, the PD100I membrane pressure sensors were used with the upper measurement limit of 6 atmospheres. The selected sensor provides an error of ±0.5% of the upper measurement limit, which corresponds to ±0.03 atm. This measurement accuracy is one of the key factors that ensures repeatability of the experiment.

Sensors used in the work make it possible to withstand pressures of up to 12 atm. However, experience obtained in experimental studies on the developed installation demonstrated that the limiting pressure was 6 atm; therefore, if the pressure on at least one of the sensors approached 6 atm., the experiment was stopped.

Four pressure sensors were used in total in the work; their layout is shown in the Figure. The sensors were located directly under the substrate, where the reinforcing material was laid. The distance between all sensors was the same at 140 mm.

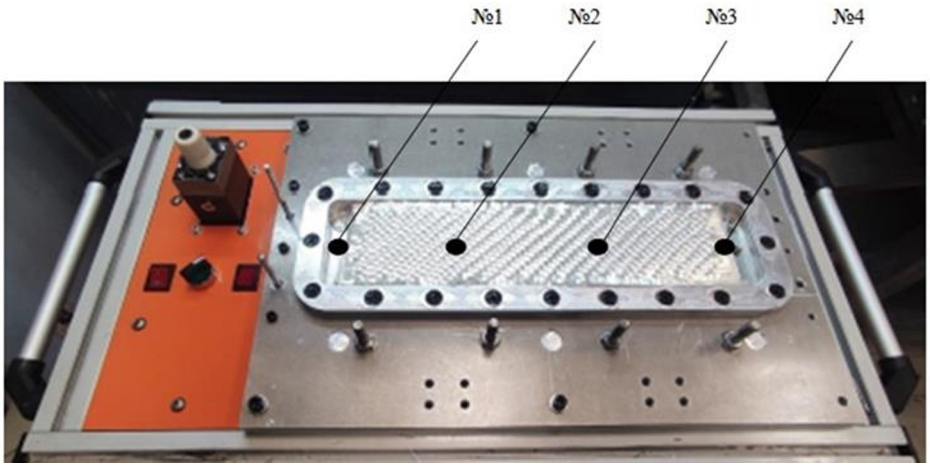


**Fig. 1.** Sensors layout

Analog signals from the pressure sensors were fed to the OVEN MB110 analog-to-digital converter transforming the received signal into digital. After that, the signal was transmitted via the RS485 bus using the Modbus protocol. The bench was connected to a PC via the RS485-USB interface converter.

The Duplomatic RPC1-0.5/T/43 mechanical fluid flow regulator was used to control the working fluid flow providing flow control of up to 0.5 l/min with an error of ± 2% of 0.5 l/min (0.01 l/min). This was also a key factor in ensuring the experiment repeatability.

Mineral oil with viscosity of $\mu$=0.148 Pa·s was used as the binder prototype, which was close to the typical binder viscosity values. Fluid viscosity was measured using the MCR 702 rheometer. The installation was designed in such a way that the working fluid was pumped into the system at a constant flow rate controlled by a hydraulic device, which main purpose was to maintain the given flow rate regardless of the pressure drop at the start and end stages of the impregnation process. The flow rate was adjusted using a special calibrated knob that changed the gap. The working fluid flow regulator delivered the flow directly to the working cavity ensuring that the fluid flow rate was equal to its filtration rate.

Experimental assessment of the permeability coefficient values consisted of the following stages:

1) Cutting the reinforcing material.
2) Laying the reinforcing material in the installation working cavity.
3) After positioning all the reinforcing material layers, they were fixed with a cover, which was tightened by the threaded connections (as shown in the Figure). If during the impregnation the sample started to move under the working fluid flow action, then the experiment was stopped.
4) Next, the desired flow rate was set on the regulator, and the impregnation process was started (in the testing process, not only the pressure drop of each sensor was controlled, but also the pressure in the liquid pump was monitored; and if it approached the critical level, the experiment was stopped).

According to the Navier-Stokes equations solution (1) for the laminar flow of viscous fluid in the cylindrical channel, i.e. the Poiseuille's law, the pressure drop along the channel depends on the channel geometric parameters, liquid viscosity of fluid and volume flow.

$$\Delta P = \frac{8\eta L Q}{\pi R^4}, \tag{1}$$

where: ΔP is the pressure difference between the points, where they are measured; η is the dynamic viscosity; L is the distance between the points, where the pressure is measured; R is the channel radius; Q is the liquid volumetric flow rate.

The K permeability coefficient value is found from the Darcy's law in the one-dimensional form (2):

$$\eta \frac{Q}{A} = -K \nabla P, \tag{2}$$

where ∇P is the pressure gradient.

Representing pressure gradient in the ΔP/ΔL form and using equations (1) and (2), the following is obtained:

$$K = \frac{\eta \cdot v \cdot \Delta L}{\Delta P}, \tag{3}$$

where v is the fluid flow rate.

Let us assume the following: fluid flow is the stabilizable parameter, difference in the working fluid viscosity and in the real binder viscosity leads to proportional alteration in pressure acting on the impregnated material. The capillary permeability effects associated

with differences in chemical nature of the selected working fluid and of the real binder are assumed to be insignificant, since these effects are significantly manifested only during initial wetting of the capillaries, and the steady-state impregnation regime is considered in the calculations.

To register data from the pressure sensors in the Matlab environment, a program was developed making it possible to connect to the permeameter using the Modbus protocol. Real-time pressure graphing was implemented and allowed the operator to instantly assess correctness of the experiment. The work also implemented the possibility of registering the liquid marks passage on the working surface of the test area (i.e., between each sensor). Registration of marks allowed calculating the flow rate.

The Darcy's law one-dimensional form was used to calculate the permeability coefficient value from the experimental data. It expressed the desired coefficient value and transition from the pressure gradient to the ratio between the difference in pressure sensor readings and the distance between the sensors, i.e. equation (3) was used for calculation.

The calculation was made according to the data of the steady state impregnation. Section, where pressure was calculated, was selected by incrementing measurements in relation to the pressure sensor measurement error. For calculation, let us select the data range corresponding to the steady state flow, where the average increments in pressure values for all the pressure sensors are close to zero with deviation of no more than 25% from the reduced error up and down. Mathematical meaning of this restriction would be considered as equality of the pressure derivative with respect to time to zero with certain tolerance. The derivative equality to zero corresponds to the function constancy, which complies with the stationary mode of the working fluid impregnation process in the reinforcing material under study.

## 3 Results and discussion

Results of the pressure measurements and permeability coefficient values calculated from them for the twill weave fiberglass are presented in Table. 1.
All studies were carried out under the following modes:
- flow rate - 240 ml/min;
- working fluid viscosity - 148.331 mPa·s.

Geometric dimensions of all the studied samples were:
- thickness - 11.5 mm;
- width - 100 mm;
- cross-sectional area - 1150 $mm^2$;
- section length between sensors 1 and 2 - 140 mm;
- section length between sensors 2 and 3 - 140 mm;
- section length between sensors 1 and 3 - 280 mm.

**Table 1.** Results of experimental studies

| Experiment No. | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|
| Pressure drop at section, | | | | | | | | | |
| atm | 2.256 | 1.19 | 1.32 | 1.93 | 1.73 | 0.67 | 1.42 | 1.07 | 0.68 |
| 1-2 | 1.355 | 2 | 1 | 6 | 7 | 1 | 1 | 1 | 3 |
| 2-3 | 4.91 | 1.41 | 1.21 | 1.33 | 1.18 | 0.97 | 0.91 | 0.92 | 0.95 |
| 1-3 | | 2.57 | 5 | 1 | 3 | 2 | 2 | 8 | 7 |
| | | | 2.53 | 3.26 | 2.92 | 1.34 | 2.23 | 1.59 | 1.34 |
| | | | 6 | 7 | 1 | 3 | 3 | 9 | |

| Permeability coefficient, Darcy | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | 320.17 | 605. | 546. | 373. | 415. | 1076 | 508, | 674. | 144 |
| 1-2 | 572.06 | 86 | 95 | 11 | 76 | .8 | 17 | 04 | 9.0 |
| 2-3 | 994.22 | 512. | 594. | 542. | 610. | 874. | 890. | 868. | 806. |
| 1-3 | | 27 | 49 | 76 | 47 | 4 | 09 | 0 | 2 |
| | | 562. | 569. | 442. | 494. | 1075 | 646. | 903. | 147 |
| | | 11 | 73 | 22 | 65 | .6 | 97 | 11 | 7.6 |

Analysis of the data obtained makes it possible to draw the following conclusions:
- values of pressure drops in the section between sensors 1 and 2 for 9 measurements taken changes from 0.671 atm (sample No. 6) up to 2, 256 atm (sample No. 1), i.e. more than by 3 times. Similarly, pressure changes in the section 1-3, where for different samples the pressure drop magnitude differs by 3.6 times. At the same time, the scatter in results of experimental studies of the pressure drop values in section 2-3 differs from each other by less than one and a half times;
- the permeability coefficient values obtained on the basis of the pressure drop data in sections 1-2 and 1-3 for different samples differ by 3 or more times; while the data obtained on the basis of results in section 2-3 is changing by no more than 40%.

Thus, to calculate the permeability coefficient values, it is necessary to use only one section located between sensors 2 and 3. For sections 1-2 and 3-4, the edge effects are characteristic, which leads to such a serious scatter in the experimental data in the pressure values.

Using similar technique, the permeability coefficient values for carbon and basalt fabrics with different types of weaving were estimated. It was established that with other factors being equal (porosity, density and type of weaving), glass and basalt fabrics were having higher permeability compared to the carbon fabrics. With an increase in the surface density, the permeability coefficient values were growing.

Accuracy of the proposed method for determining the permeability coefficient values were assessed by comparing the impregnation process duration, which was determined experimentally and theoretically using equation (4) []. The data obtained are provided in Table. 2.

$$\tau = \frac{\eta L^2}{2K\Delta P}, \tag{4}$$

where τ is the impregnation process duration.

**Table 2.** Duration of the glass fiber impregnation process

| No. | Impregnation process duration, s | |
|---|---|---|
| | Theoretical | Experimental |
| 1 | 63.57 | 67 |
| 2 | 68.92 | 70 |
| 3 | 67.35 | 70 |
| 4 | 65.02 | 69 |
| 5 | 66.34 | 0 |
| 6 | 47.34 | 51 |
| 7 | 42.78 | 46 |
| 8 | 44.63 | 46 |

| | | |
|---|---|---|
| 9 | 45.82 | 48 |
| average | 56.86 | 51.88 |

Analysis of the obtained results demonstrated extremely high accuracy of the proposed method. The error between experimental and theoretical values did not exceed 1%. For all the studied samples, the experimentally determined values of the impregnation process duration exceeded those found theoretically.

## 4 Conclusion

An installation was designed and developed, which made it possible to determine the permeability coefficient values for glass, carbon and basalt fabrics. High accuracy is the advantage of the proposed method.

It was established that to assess the permeability coefficient values, it was necessary to use not the entire area of the reinforcing material, but only its limited section located between sensors 2 and 3. Edge effects associated with start and end of the impregnation process were completely missing in this section.

As a result of the conducted experimental studies, the coefficient values were determined. Accuracy of the results obtained was determined by comparing the total impregnation process duration found theoretically and experimentally. It was established that for all the samples under study, the experimentally determined values of the impregnation process duration exceeded those found experimentally by more than 1%.

Results of the experimental studies would establish the basis for a database making it possible to create digital passports for the fibrous reinforcing materials under study.

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