Data measuring system to determine the solvent diffusion coefficient in products from capillary-porous materials

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Abstract
The development of methods and means for non-destructive product testing is the current task of modern instrument-making in the study field of porous materials ability to transport the substances distributed in a solid phase. They allow one to determine the characteristics of mass transfer directly in the finished product, because they do not require the preparation of special samples of a given configuration and size, typical for most of the used methods and devices. The aim of this study is to develop a data measuring system that improves the performance of studies of polar solvents diffusion in porous media. The data measuring system is designed to implement methods of the solvents diffusion coefficient control in fine and bulk products from isotropic and anisotropic porous materials with one-way access to flat surfaces of specified size products. The schematic of the data measuring system, virtual surface for experiment control, the flow chart for the measurement operations of the developed methods, the results of testing the system are given.

Keywords: non-destructive testing, diffusion coefficient, capillary-porous products, data measuring system

1. Introduction
To control the composition and properties of the natural environment, substances, materials and products, non-destructive testing (NDT) methods have become increasingly widespread. They provide not only the safety of products under control, but also higher research productivity. For example, in thermophysical studies high-performance pulse methods for measuring thermal conductivity, thermal diffusivity and heat capacity are widely used. They implement thermal effects on the objects of control in the form of heated lines [1], flat surfaces [2], circles [3], strips [4] and other configurations. In the field of mass transfer characteristics control, such methods and devices implementing them have been less developed. The reasons for this are two features:
- the complexity of measuring the diffusing substance concentrations in localized points of the porous bodies, compared with the local temperature measurement [4];
- the need for individual calibration of the applied local concentration converters for each new porous material and diffusing substance [5]. This completely excludes the efficiency of control, since it is carried out for tens of hours.

Currently, it has been known about several methods and devices providing measurement of the diffusion coefficient in implementing pulse actions by the diffusing substance dose:
1) point pulse action [6], to perform the preliminary calibration of the galvanic converter concentration;
2) point pulse action, to determine the diffusion coefficient in products from fine sheet [7] and bulk [8] isotropic porous materials;
3) linear impulse impact, to determine the desired coefficient in products from fine sheet [9] and bulk [10] anisotropic porous materials.
The last two groups of methods do not require preliminary calibration of the used concentration converter of the diffusing substance, and, therefore, can provide higher test performance. To improve the efficiency of the diffusion coefficient studies in implementing these methods, it is advisable to use an information measuring system (IMS).

2. Problem statement
The task is to develop data measuring system providing automated experiment, measurement and real-time registration of the necessary experimental information and calculation of the desired solvents diffusion coefficient according to the developed algorithms. To achieve the purpose, it is necessary to solve the following tasks:
- to develop a schematic of IMS;
- to develop the algorithm of functioning and the software for the experimental sample of IMS;
- to test the experimental sample of IMS;
- to compare the theoretical estimates of the diffusion coefficient measurement error with the results of experimental studies.

3. Composition of information-measuring system
Information measuring system is equipped with two types of measuring devices [11]:
- No 1 with a point source of solvent dose - to measure the diffusion coefficient in products from fine sheet and bulk isotropic porous materials;
- No 2 with a linear solvent dose source - to measure the diffusion coefficient in products from fine sheet and bulk anisotropic porous materials.

IMS consists of (Fig. 1):
- measuring device 1 with point or linear solvent sources with power W, and electrodes 2, 3 of three pairs of galvanic converters (GC) of local solvent concentration in the porous material 7, located at the distance of 3, 4 and 5 mm from the point or line of pulse action [11];
- connector 4, which is part of the measuring devices 1, to which the electrodes of galvanic converters 2, 3 are connected;
- multi-function data acquisition board 5 PCI-1202H from ISP DAS (Taiwan);
- personal computer 6.

Figure 1. Schematic of information measuring system

Multi-function data acquisition board 5 includes ADC, DAC and discrete output for the formation of the control signal. It is located in the PCI slot of a personal computer.
Analog multiplexer developed by the IMS connects alternately one of the three galvanic converters of the above measuring devices to the board amplifier 5. ADC generates digital equivalents of the input signals, which are smoothed, filtered and processed in accordance with the developed algorithm. The program of experiment control, experimental data processing and visualization has been developed by National Instruments technology in LabView environment.

4. Algorithm of IMS functioning
The sequence of the developed methods measuring operations includes the preparatory part (performed before the study of products from a new class of materials) and the main one (in the implementation of
inline inspection of products from the selected class of materials). The preparatory part includes the following operations:

1. to determine the sample density of the considered material class \( \rho_0 \) in the dry state.

2. to exposure the sample of the considered material class in an atmosphere of saturated solvent vapors at a given control temperature. To determine the equilibrium concentration of the solvent \( U \) and the maximum signal of the galvanic converter \( E \) corresponding to the solvent concentration in GC.

The sequence of the developed methods measuring operations for non-destructive testing of the products batch is given in Table 1.

| No of operation | The content of the operation |
|-----------------|-----------------------------|
| 1               | To exposure the product at a given elasticity of the solvent vapor to create the required initial solvent distribution in it \( U_0 \) for a given time. When using a "dry" material (in the absence of a distributed solvent) – without pre-exposure. |
| 2               | To select and connect a measuring device \( \# 1 \) that implements point pulse action, or \( \# 2 \) that implements the linear impulse action |
| 3               | To enter initial parameters \( \rho_0, U_p, E_p, r_0 \) or \( x_i \) – respectively, the distance to the point or line of the pulse application \( (i=1, 2, 3) \), etc., necessary for the IMS operation. To calculate the required solvent dose for a point source, to control the diffusion coefficient in fine or bulk products from isotropic porous materials according to [7, 8]. To calculate the required dose for a linear source, to measure the desired coefficient in fine or bulk products from anisotropic porous materials according to [9,10]. |
| 4               | To place the device with a point pulse or linear mass source on the surface of the controlled product in the case of bulk products studying. When using fine-sheet products, pre-placement of the product on an insulating substrate. |
| 5               | To measure the initial values of the electromotive force (EMF) of galvanic converters. |
| 6               | To "moisture" pulse point or linear the test material surface by a given dose of the solvent from the dispenser with simultaneous IMS start. |
| 7               | To determine the time of reaching the maximum EMF of the galvanic converter at a distance \( r_0 \) or \( x_c \) from the source and to calculate the desired diffusion coefficient. |
| 8               | To inform of the experiment end in the form of text and sound message. |

The program, implementing the method of the experiment and processing experimental data, was developed in the LabView environment and based on the use of the Standard State Machine template and the Case structure from the function library. This configuration of the program allows one to carry out serial measuring operations presented in Table 1 and implemented by subprograms placed on the tabs of the above structure.

At the first stage of the experiment, meeting to the first tab of the Case structure, the choice of the studied item type is made: from fine or bulk CPM, the choice of the pulse action type, the initialization of the used variables and the enter of constants, as well as the configuration of the measuring channels of the data acquisition board, connecting three primary measuring converters (sensors) to the analog inputs through the matching devices. Figure 2 shows the control panel of the experiment at the first stage. At this stage of the experiment, the operator enters the following information: 1) surname, name and patronymic of the researcher (field "Researcher"); name of the material ("Material"), distances "r0 sensor_1", "r0 sensor_2" and "r0 sensor_3" from the sensors to the point or line of solvent dose exposure on the sample (see Fig. 2), the density of the test material \( \rho_0 \) in the dry state, the equilibrium concentration of the solvent \( U_p \) and the maximum signal of the galvanic converter \( E_p \) corresponding to the concentration of the solvent in the porous material. Date and time information is determined automatically, but the operator can adjust these parameters determined by the program.

After entering the data, the program calculates the rational solvent dose to be introduced as a point or linear mass pulse for each sensor. The solvent dose value for the selected sensor (with a specified \( r_0 \) or \( x_c \) value) is selected. The required amount of solvent is poured into the dispenser. The preparation to the active part of the experiment is over.
To start the experiment, a pulse with a dose of solvent should be applied to a given point or along a straight line on the surface of the test product and the "Start" button is pressed. Then the structure of the Case program will go to the tab "2" and one can observe the real time change of the sensor signal on the screen of the graphical indicator. At this time, the measurement information is recorded in a two-dimensional array. Each row contains three values of sensor EMF and one value of time. Moreover, the EMF of the sensors is represented in relative units to the maximum signal of the galvanic converter $E_p$. When the sensor EMF begins decreasing steadily, one should stop the experiment with the "Stop" button. This causes an automatic transition to the Tab Control of the front panel "Calculation" and the tab "3" of the Case structure. This tab contains a subprogram for processing experimental data and calculating the diffusion coefficient of the test material for each sensor. Data processing includes determining the status of a possible sensor saturation, i.e., the threshold excess of sensor sensitivity by the concentration at the control point, or finding time to reach maximum if saturation does not occur. Saturation control is based on the use of the condition:

$$\frac{\partial E_i(\tau)}{\partial \tau} \leq \varepsilon,$$

where $E_i$ is EMF of the $i$-th sensor, $\tau$ is time, $\varepsilon$ is threshold, the value of which meets to the minimum derivative $\frac{\partial E_i(\tau)}{\partial \tau}$ typical for the beginning of saturation. The diffusion coefficient of the test material is calculated if sensor saturation does not occur.

After that, the operator presses the "Stop" button and then the program offers either to start a new experiment or exit the program. Figure 3 presents the control panel after the experiment.

If there is no preliminary information on the values $p_0$ and $U_p$, the solvent dose is selected by the operator by carrying out trial experiments. Based on the changes in the signals of all sensors, a decision is made on the subsequent change of the introduced solvent dose, to achieve the maximum EMF of the selected galvanic converter in the operating range of its static characteristic.
5. Experimental results
The information measuring system equipped with two types of measuring devices was tested on a number of porous products, including those with explicitly implied anisotropy of properties. The results of twenty-fold measurements of the ethanol diffusion coefficient in foamed gypsum with a density in the dry state of 600 kg/m³ at \( r_0 = 4 \) mm, obtained using a measuring device No 1, equipped with a point pulse device, for isotropic porous materials are presented in Figure 4 as an example.

![Figure 4. Results of the ethanol diffusion coefficient control in foamed gypsum](image)

The results of twenty-fold measurements of the moisture diffusion coefficient in the machine and cross directions of "Tissue" type paper with the thickness of \( \sim 0.1 \) mm at \( x_c = 4 \) mm, obtained using the measuring device No 2, equipped with a linear pulse device, are presented in Figure 5, as an example.

At present, it is not possible to estimate the systematic component of the error due to the lack of reference materials for porous material–solvent systems in question, so the authors estimated the random component of the error. The relative error of the measurement result was determined as follows:

\[
\delta_{\text{rel}} = \frac{t_{\alpha,n}S_n}{\sqrt{D}},
\]

where \( S_n = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (D_i - \overline{D})^2} \) is the standard error of individual measurement, \( \overline{D} \) is the mathematical expectation of a random variable; \( t_{\alpha,n} \) is Student's coefficient at a confidence probability \( \alpha \) and the number of measurements \( n \).

![Figure 5. Results of the paper moisture diffusion coefficient control](image)
6. Results and discussion

The experimental studies showed that the random uncertainty in determining the diffusion coefficient at a confidence probability of 0.95 did not exceed 6 - 9% for isotropic and 8 - 13% for anisotropic materials. The duration of the experiment is about 12-70 minutes. At the same time, the experimentally received values of the random uncertainty in determining the desired characteristic did not exceed the previously obtained theoretical estimates [7-10]. The estimation of comparability of the study results with the developed IMS and data on the desired diffusion coefficient, obtained by other tested methods [12], revealed a discrepancy in the range of 8 – 17%.

7. Conclusion

The given information measuring system can be used for non-destructive testing of the diffusion coefficient in 4 types of products from porous materials having a sufficient flat section of the structure for the placement of the measuring device of the corresponding type:
1) bulk with isotropic structure (for example, building panels from gypsum and composites, flat porous heat insulators, block porous sorbents, etc.);
2) bulk with anisotropic structure (for example, composite panels with oriented fibrous structure, etc.);
3) fine sheet with isotropic structure (for example, different types of paper, fabrics, porous sheet sorbents);
4) in different directions of fine products with anisotropic structure (for example, with oriented fibrous structure).

The structure of IMS, algorithm and software allow one to reconfigure quickly the system, to study each type of product. The developed IMS algorithms, ensuring the operation of the applied galvanic converter in a rational range of its static characteristics, quickly calculate the dose of the introduced by the pulsed action solvent. IMS is tested on a number of products from porous materials, including those with explicitly implied anisotropy of properties. It provides a relatively rapid determination of the desired diffusion coefficient without destroying the tested products.

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