

Empirical determination of water saturation of porous materials in the process of long duration imbibition

*Volodymyr Yelisieiev*¹, *Vasyl Lutsenko*^{1,*}, *Tetiana Demchenko*¹, and *Vitalii Ruban*¹

¹Institute of Geotechnical Mechanics named by N. Poljakov of National Academy of Sciences of Ukraine, 49005, Dnipro, Simferopolska Str., 2a, Ukraine

Abstract. The paper presents the results of an experimental study of the process of imbibition of porous bodies with a long stay in the impregnating medium. Experiments have confirmed, that at time of the order of 100 min there is a significant slowdown in the rise of the saturation curve, which can be taken as the end of the process. However, for the final completion of the process time is needed in the hundreds of times greater and the water saturation can increase more than twice. Experiments showed that with an increase in porosity, the length of the region of a significant deceleration of the impregnation rate tends to decrease. Regardless of the difference in the structures of the tested samples, it was possible to obtain a general analytical expression for describing the process of water saturation, which indicates similarity in the movement of fluid in the system of pore channels. The approximation of the experimental data by a logarithmic function gives a good result of an analytical representation of the process. It is also shown that the theoretical approaches used make it possible to obtain water saturation curves over long periods with good accuracy.

1 Introduction

Modern technologies for the preparation of various natural and artificial materials for further processing often use the so-called “wet processes”. Most of these materials are porous media, so the study of the saturation processes of porous materials with liquid is important. It is practically impossible to determine the necessary values theoretically, due to the difficulty in determining the structure of pores. To obtain quantitative values, experimentally established data are almost always needed. Theoretical developments, for example, [1, 2] predict only some regularities in the motion of capillary fluids in porous media.

It has now become clear that the question of determining the maximum degree of moisture saturation of various porous materials requires further study. In a review [3], it was shown that a seemingly complete imbibition process at times on the order of 100 minutes, with a longer study time, is further developed. For the final completion of the process, time is needed hundreds or even thousands of times more, and the water absorption can be more than doubled. In the works [3, 4], some features of such a long process are

*Corresponding author: lvi151156@gmail.com

noted.

2 Methods

This paper presents the results of experiments on the imbibition of samples from coal, ceramic bricks and pumice. For these materials, general imbibition patterns were found, despite the different internal structure and capillary properties.

The experiments were carried out with 17 samples, the dry weight of which varied from 0.01 to 0.12 kg. Distilled water was used as a saturating medium. The experiments were carried out at atmospheric pressure. The samples were completely lowered into water so that a layer of water about 0.5 cm high was above the upper surface of the sample. Weighing was carried out on a digital scale with a measurement resolution of 0.01 g. Before each weighing, the samples were removed from the container with water and soaked with soft tissue in order to eliminate surface moisture. The intervals between measurements during the experiment gradually increased from 0.1 to 10000 minutes. The end time of the imbibition process for each sample was determined individually. As a rule, the criterion for the completion of the experiment was the absence of an increase in the weight of the sample in several consecutive measurements for about 7 days. The maximum duration of the experiment for one of the samples was more than 5 months.

The amount of water saturation of the samples in the experiments was determined by the formula

$$w = \frac{m_S - m_D}{m_D}, \quad (1)$$

where m_S – the mass of the sample is saturated with water, m_D – is the mass of the dry sample.

The results of the experiments in the form of graphs of changes in the amount of water saturation with time for some samples are presented in the figures below. At the same time, taking into account the above, in the first two figures the obtained dependences are divided into two parts. The first part depicts the change in water saturation for times on the order of 100 minutes, and the second part shows the change in water saturation during the whole experiment.

3 Experimental results and discussion

Figures 1, 2 and 3 show the time variation of the saturation of two samples of red brick. Both samples were made in the form of parallelepipeds. At the same time, sample 1 had sides of 40×35×30 mm. Sample 2 had dimensions of sides 53×53×25 mm. With each of the samples was carried out on two experiments. From Figure 1 it can be seen that when immersed in water for no more than 5 minutes, both samples acquire a saturation of approximately 0.154. Further, for these samples, the imbibition rate becomes so small that the saturation practically does not change for quite a long time. This is the first imbibition stage. The saturation at this stage practically does not depend on the ratio of sample sizes. After a time of about 500 minutes, the imbibition process is accelerated. Figure 2 shows the entire imbibition process. Due to the large difference in time scales, the first stage at this phase is practically not distinguished. From the consideration of the curves in Figure 2, it can be seen that for a time of 150000 minutes the saturation of the samples reaches values of 0.25. This is more than one and a half times the maximum saturation value at the first stage. For clarity, Figure 3 shows the change in a logarithmic time scale. In this figure, the characteristic region in which the water saturation of the samples remains almost

unchanged is clearly visible.

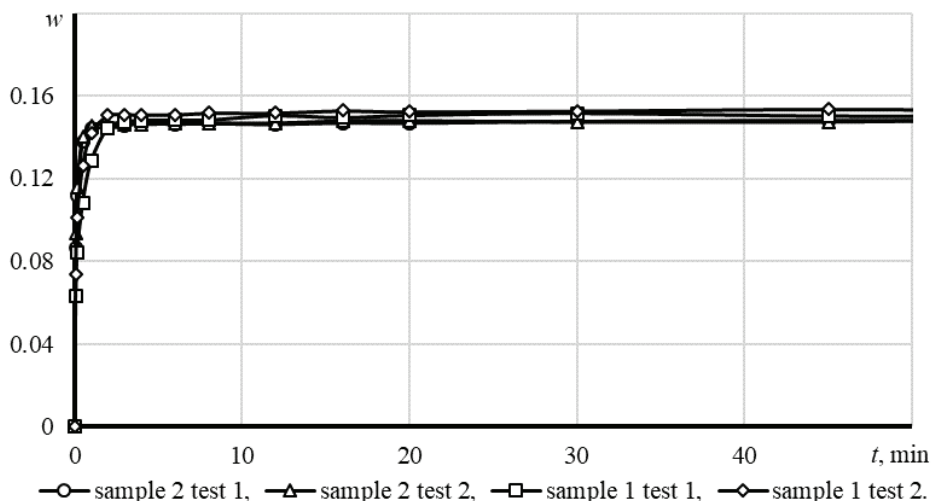


Fig. 1. Change in time of water saturation of red brick samples at the initial stage.

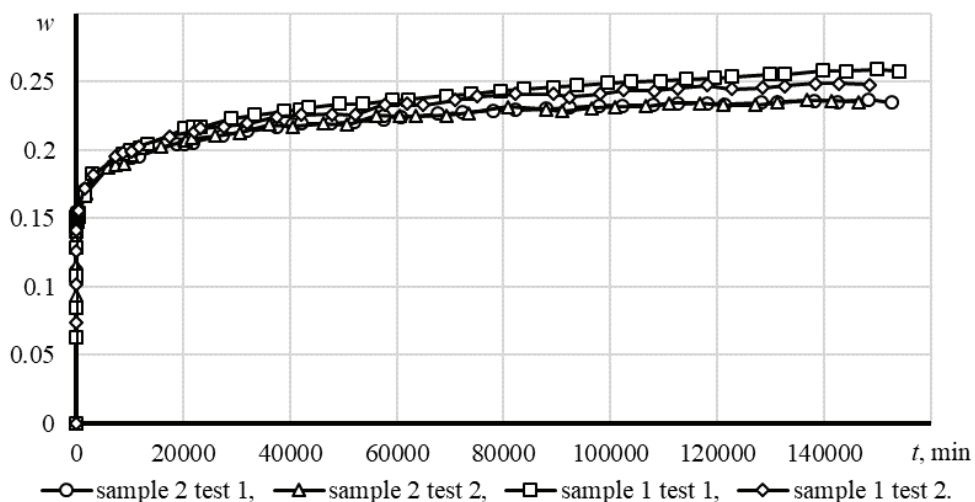


Fig. 2. The change in time of water saturation of samples of red bricks.

The following Figures 4 and 5 show the experimental data with samples made from artificial pumice. Ten samples of different weights and sizes were tested. The porosity of this material is much higher than the porosity of red bricks. Therefore, the water saturation values were much higher. This helped to increase the accuracy of measurements. The maximum values of water saturation in the experiments with samples made of pumice approached one, and for the most highly porous sample 10 was much higher than this value. In Figure 4 and 5 it can be seen that in the time range of about 100 minutes the impregnation rate drops noticeably, or even becomes close to zero, which can be perceived as the end of the process. This area is not as long as in Figure 3 for red bricks. Similar behavior patterns of saturation curves were also noted in experiments with other samples. In [3], it was shown that this region is an intermediate asymptotic, then the process continues, and the saturation gradually increases. From the analysis of the curves in Figures

3, 4 and 5, it can be assumed that with increasing porosity, the length of the above zone tends to decrease.

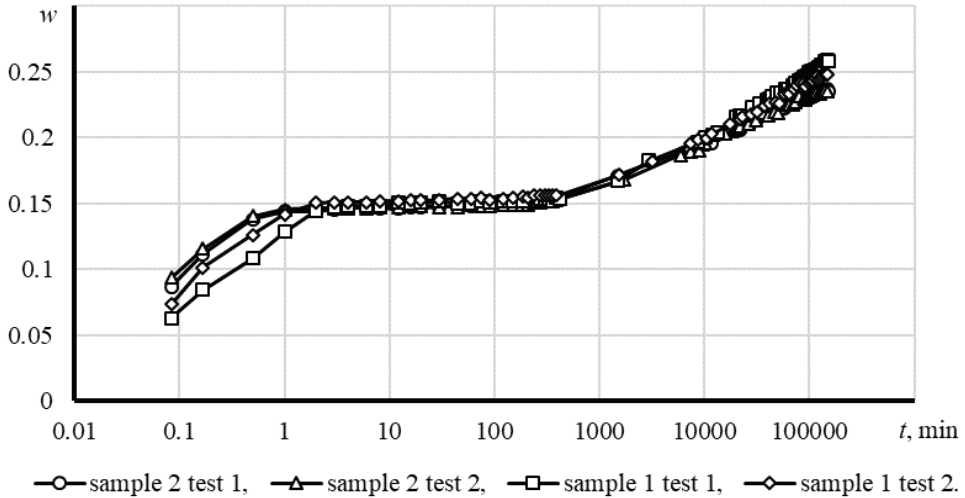


Fig. 3. Change in time of water saturation of red brick samples in logarithmic coordinates.

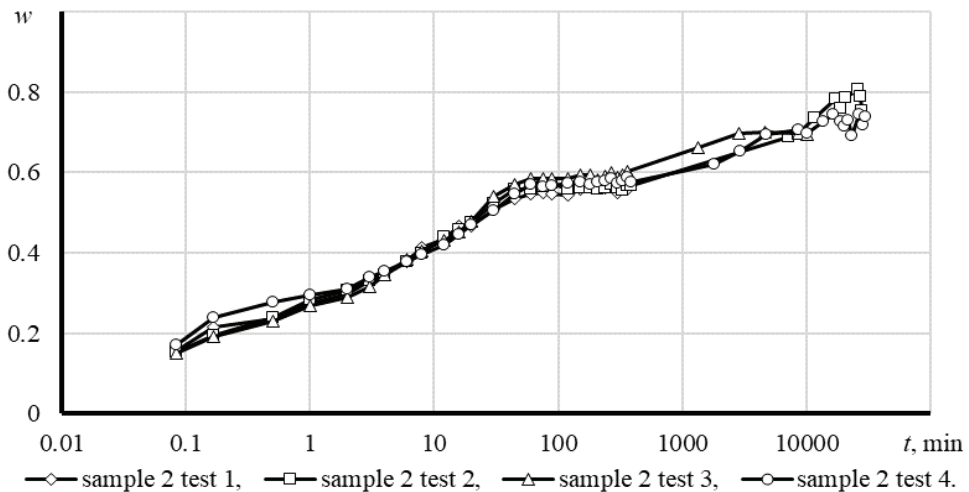


Fig. 4. The change in time of water saturation of sample 2 from artificial pumice.

Experiments with samples of natural pumice, which are shown in Figure 6, do not fit into the picture described above. In this figure, the region of slow growth of saturation is completely absent. Perhaps this is due to the peculiarities of the internal structure of the pores. Additional research is needed to resolve this issue. Additional studies of materials with very high porosity are also needed.

Figure 7 shows the time variation of the saturation of the coal samples. There is a significant variation in experimental data. This is due to the fact that coal has a significantly lower porosity compared with previous materials. Also, the intermediate asymptote of saturation is not observed as it was in the previous cases. In general, for all the experiments conducted with various samples of coal, it can be noted that the maximum water saturation was in the range from 1.8 % to 6 %.

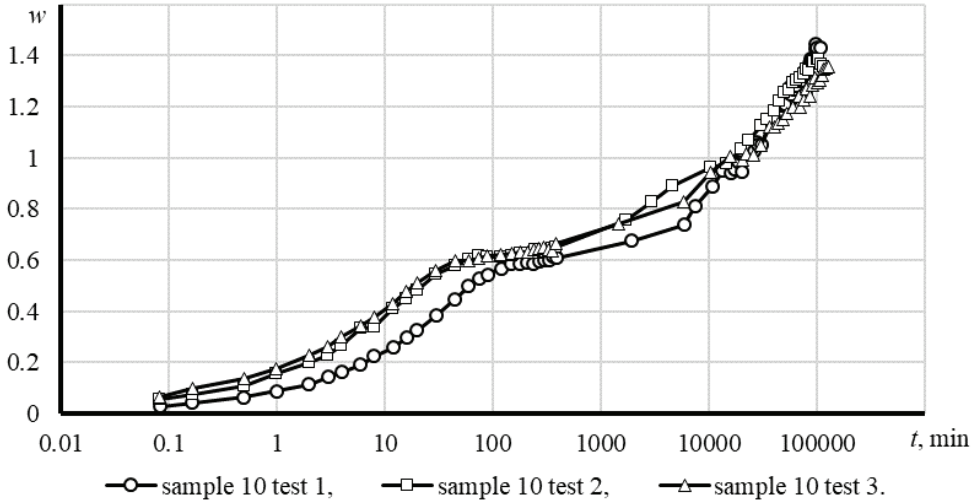


Fig. 5. The change in time of water saturation of the sample 10 from artificial pumice of high porosity.

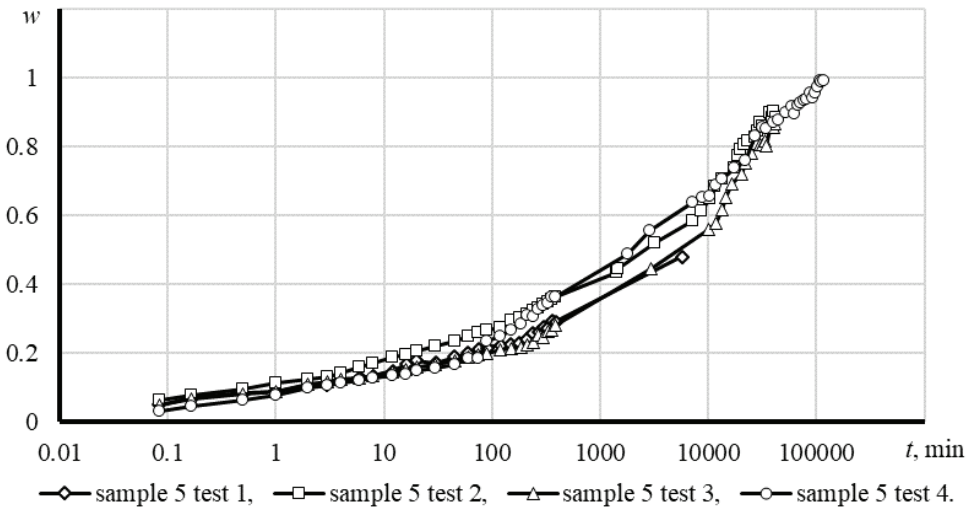


Fig. 6. Change in time of water saturation of sample 5 from natural pumice.

Separately, experiments were carried out to determine the water saturation of coal samples after their treatment with 3 % hydrogen peroxide solution. These experiments consisted of four stages. At the first stage, the experiments described above were carried out to determine water saturation. Then the samples were dried to constant weight. After that, they immersed in a solution of hydrogen peroxide for a long time. Then again dried to constant weight. After drying, the experiment to determine water absorption in distilled water was repeated two times. Some of these experiments are also shown in Figure 7. The figure shows that the treatment with hydrogen peroxide significantly reduces the water saturation of coal. Subsequently, water saturation is restored to values close to the original level.

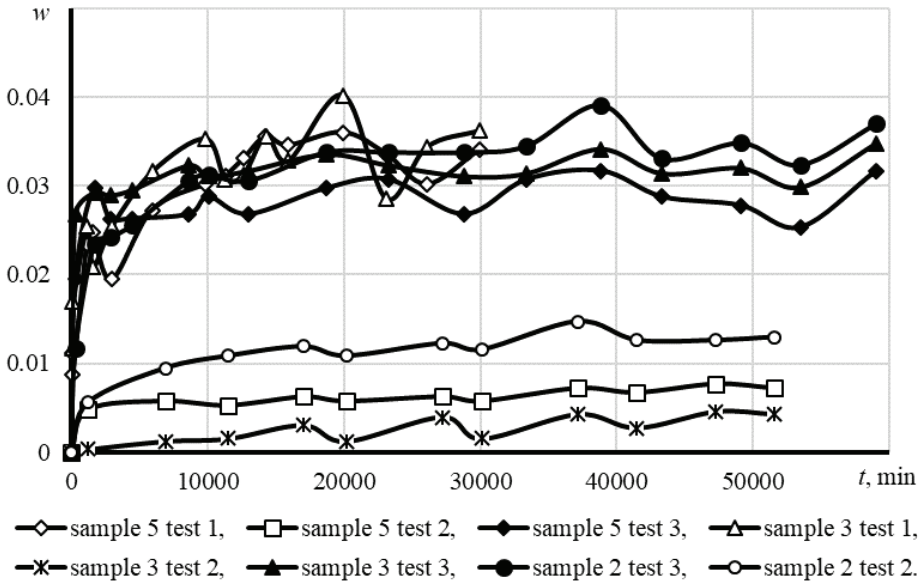


Fig. 7. Change in time of water saturation of samples from coal.

Analysis of the obtained curves showed a very important detail, which is also noted in the cited review. It indicates that in a number of works the impregnation curves are close to the curve of the form: $\gamma \cdot \ln t$. We attempted to approximate experimental curves in the form

$$\bar{w} = 1 + (\bar{w}_N - 1) \frac{\ln\left[\frac{(a+t)/(a+t_C)}{(a+t_N)/(a+t_C)}\right]}{\ln\left[\frac{(a+t_N)/(a+t_C)}{(a+t_N)/(a+t_C)}\right]}, \quad (2)$$

where t_N – the first point from which the approximation curve is drawn (in our case in all variants for pumice and brick $t_N = 30$ min, for coal $t_N = 26$ min); \bar{w}_N – saturation corresponding to this point; t_C – end point of the time interval; a – fitting value. In this formula $\bar{w} = w/w_C$, and w_C – finite water saturation.

Practically all the curves obtained in our experiments are well described by this law, despite the different sizes of samples and different materials.

For example, Figures 8–10, which show approximation variants (curves 2) for samples of pumice stone, brick and coal. As can be seen from these figures, the approximation by a logarithmic function of the form (2) is quite good (this is done for almost all samples). Thus, it can be noted that the expression obtained is valid for describing the impregnation of porous and fractured porous materials.

The same figures of curve 3 show the analytical dependence of the displacement of a gas bubble from the capillary, obtained in [5]:

$$\bar{w} = 1 + (\bar{w}_N - 1) \cdot \exp\left\{-\left[(\bar{w} - \bar{w}_N) + b(t - t_N)\right]\right\}, \quad (3)$$

where $b = (gT/8vH_C) \cdot R_C^2$ – fitting complex (T – time scale; v – kinematic viscosity coefficient; g – acceleration of a freely falling body; H_C – capillary length; R_C – pore channel radius).

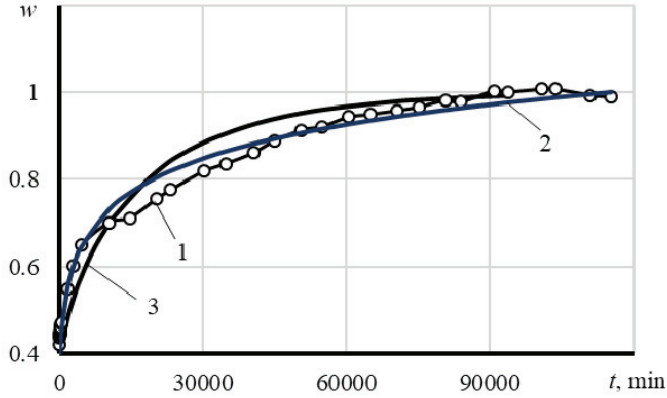


Fig. 8. Approximation of the saturation curve for pumice: 1 – experimental curve (sample 10 test 2), 2 – approximation curve by the formula (2), 3 – approximation curve by the formula (3).

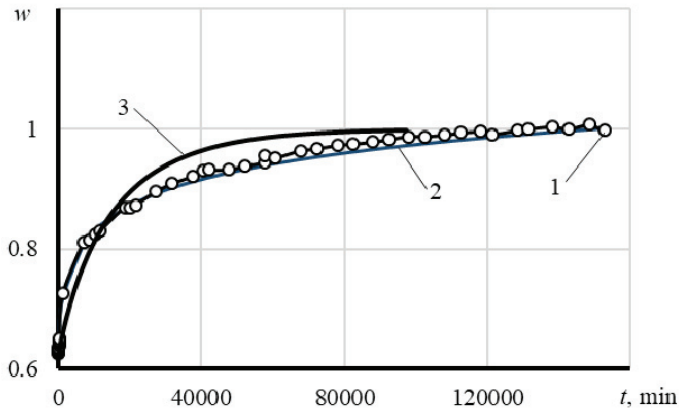


Fig. 9. Approximation of the saturation curve for red bricks: 1 – experimental curve (sample 2 test 1), 2 – approximation curve by the formula (2), 3 – approximation curve by the formula (3).

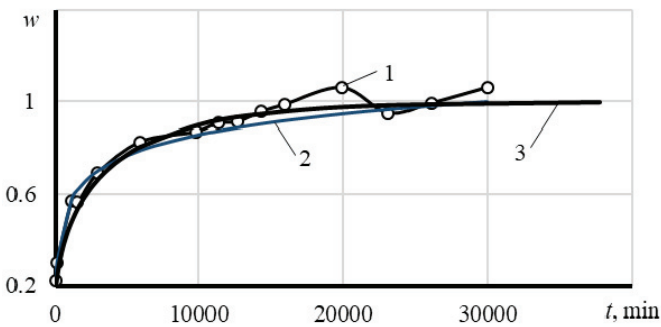


Fig. 10. Approximation of saturation curve for coal: 1 – experimental curve (sample 2 test 3), 2 – approximation curve by the formula (2), 3 – approximation curve by the formula (3).

The reason for considering imbibition as the process of displacing a gas bubble is that when a sample is introduced into water, the internal gas filling the pore space is surrounded by water on all sides. As shown in [6], the internal gas volume behaves in approximately

the same way as a bubble in an unlimited volume of liquid. This is clearly seen in experiments at the very beginning of the body's immersion in water. This dynamic phase of oscillation of the internal bubble quickly ends and another phase begins - the phase of gas hydraulic displacement due to the difference in hydrostatic pressure on both sides of the sample. As calculations in [5] show, this phase largely depends on the geometrical characteristics of the pore channel. In reality, the behavior of the displacing fluid more complex [7]. The process is practically completely random in nature and is associated with an ordinal wetting hysteresis [8]. In this phase, the interaction of the surface of the pore channels with the liquid begins to play a role: adsorption, gas dissolution in dead-end pores, hydration of polymer inclusions, etc. [9]. All these processes are not amenable to mathematical modeling with a sufficient degree of accuracy.

Returning to Figures 8 – 10, it can be noted that formula (3) describes saturation curves somewhat worse than approximation curve (2), but, in general, it is satisfactory.

Conclusions

The approximation (2) and the theoretical curve (3) describe almost the entire imbibition curve, not counting small compared to the whole process of the initial section, which can be attributed to the dynamic stage of the process. Approximation formula (2) is a kind of formal mathematical processing, in which the kinetic side of the process is hidden, however, it has a high accuracy of the description of the process as a whole. Advantage of the theoretical description expressed by formula (3) is that it clearly involves important parameters that characterize both the physical parameters of the fluid and the geometric features of the pore space, for example, the effective channel radius, which characterizes the scale of a pore channel of the impregnated body.

References

1. Barenblat, G.I., Entov, V.M., Ryzhik, V.M. (1984). *Dvizhenie zhidkostey i gazov v prirodnykh plastakh*. Moskva: Nedra
2. Moshinskiy, A.I. (2009). Mathematical model of impregnation and extraction in the case of bidisperse porous material. *Teoreticheskie osnovy khimicheskoy tekhnologii*, 43, 4, 401–407
3. Barabanov, V.L. (2014). Empirical parameters of the model of countercurrent capillary impregnation of rocks. *Geofizicheskie issledovaniya*, 15, 1, 27–52
4. Barabanov, V.L., Lyubushin, A.A. (2013). Experience in the study of fractal properties of capillary impregnation of rocks. *Inzhenerno-fizicheskiy zhurnal*, 86, 1, 3–13
5. Yeliseyev, V.I., Lutsenko, V.I. (2017). Liquid displacement of a gas bubble from a capillary. *Geotekhnichna Mekhanika* [Geo-Technical Mechanics], 134, 95–104
6. Yeliseyev, V.I., Lutsenko, V.I. (2017). The influence of the wetting angle on the initial stage of impregnation of the body with a homogeneous porous structure. *Geotekhnichna Mekhanika* [Geo-Technical Mechanics], 132, 51–61
7. Bulat, A.F., Naduty, V.P., Yeliseyev, V.I., Lutsenko, V.I. (2017). *Kapillyarnye efekty v dinamicheskikh protsessakh obezvozhvaniya izmelchennoy gornoy massy*. Dnipro: Porohy
8. Yeliseyev, V.I., Lutsenko, V.I. (2006). The phenomena of a static hysteresis in capillaries, *Geotekhnichna Mekhanika* [Geo-Technical Mechanics], 66, 157–163
9. Lykov, A.V. (1954). *Yavleniya perenosy v kapillyarno-poristykh telakh*. Moskva: Gosizdat