

RESEARCH OF SURFACTANT SOLUTIONS FILTRATION IN HARD ROCKS

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***Aim.** Prospective means of increasing the effectiveness of mining processes is the controlled variation of the properties of rocks with surfactants. When designing the technology of surfactants application, important research problems are the investigation and the forecast of filtration of active solutions in the processed rocks. The aim of the researches is the determination of regularities in surfactant solutions filtration in hard rocks.*

***Research methodology** includes experimental works and theoretical analysis of the processes of active solutions filtration in hard rocks.*

***Research results.** On the basis of experimental works the research has determined that the process of active solutions filtration comes in three stages which differ in the mechanism of active liquids motion in hard rocks. Analytical estimation is introduced of the parameters of the given stages of saturation. It has been shown, that the process of filtration possesses fractal properties and can be investigated from the point of the percolation theory. It has been stated, that in logarithmic coordinates the dependence between the quantity of solution penetrating into rocks on the time is described by the two linear equations which correspond to the stages of filtration saturation and the spontaneously developing process of two-dimensional migration of active solutions.*

***Application area.** The obtained results are used when designing the methods and means of controlling properties and condition of rocks and massifs.*

***Conclusions.** The stated regularities make it possible to forecast the rock massifs saturation by active solutions in various processes of mining. The obtained quantitative assessments (equations) are meant to be the base for statistic modeling (Monte-Carlo method) of rock permeability as a percolation system.*

***Key words:** rocks; surfactants; filtration mechanisms; percolation theory.*

Introduction. One of the prospective means of increasing the effectiveness of mining technology is the controlled variation of rock properties with the help of surfactants. Action of surfactants is based on adsorption decrease of rock strength (*Rehbinder effect*) [1, 2]. Permeating into the fracture process zone, active solutions contribute to rocks softening and fracture development. It is accompanied by a range of positive effects. It has been stated [3], that rock processing with specially selected surfactants is good for the capacity of shafts construction processes and mineral deposits exploitation. Analytical, laboratory, and industrial researches in conditions of the shafts of Severouralsk bauxite deposits and Donbass Horlivka region have shown the following. Action of surfactants, manifesting in adsorption decrease of the surface energy of rocks, fracture growth activation, plastification of intergranular bonds, is accompanied by the regular variation of a complex of mechanical properties of rocks: strength reduction (from 20 to 70%), decrease of the module and limit of elasticity (by 1.3–1.5 times on average); activation (by 1.5–7.0 times) of the processes of creep and relaxation of stresses in a massif, etc.

Such rock properties variation has the following effects. The use of surfactants as drilling fluid increases mechanical drilling speed in boreholes and wells by 1.2–1.4 times, reduces drilling tool wear and blunting by 1.2–1.4 times on average. Surfactant molecules adsorption activity, as well as the improvement of drilling conditions reduces dust content in the mine

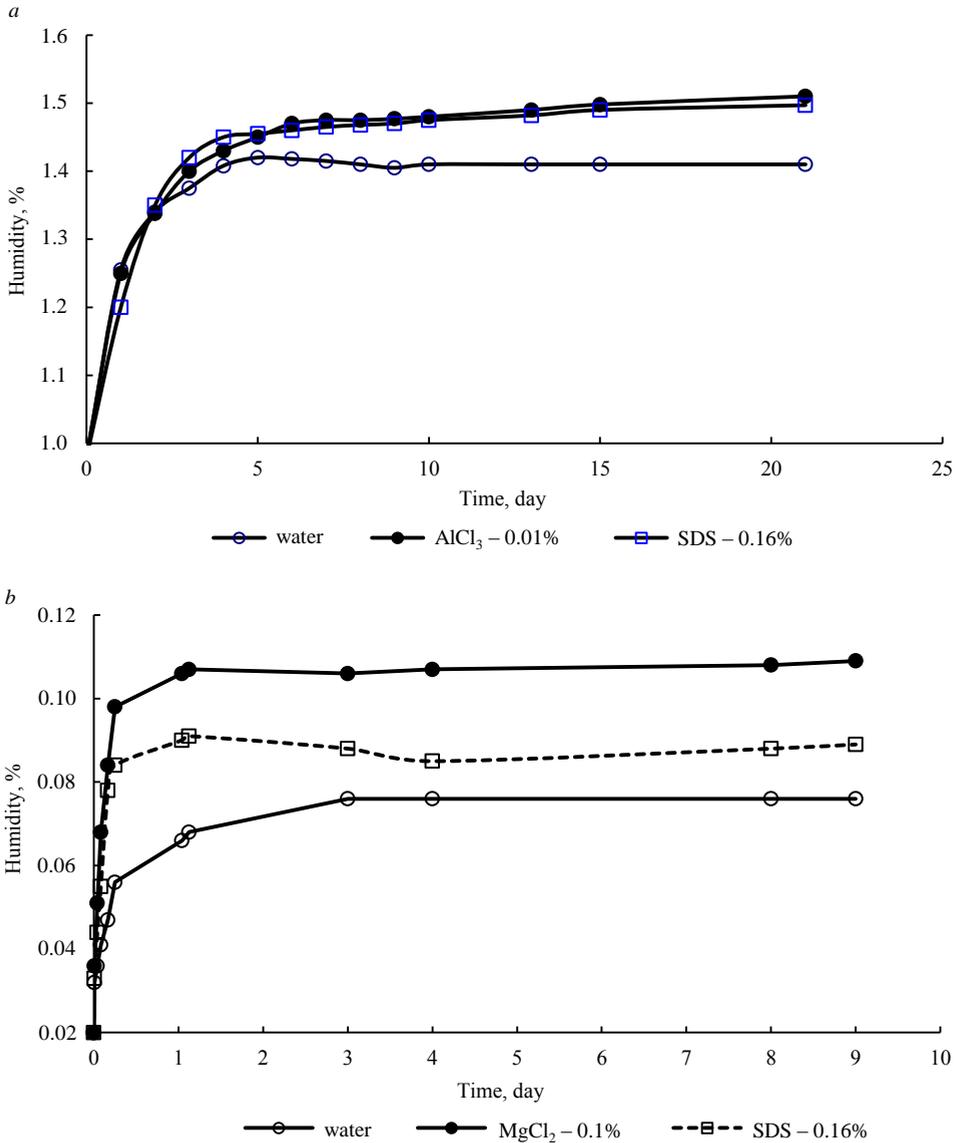


Fig. 1. Kinetics of saturation:
 a – porphyrite; b – limestone
 Рис. 1. Кинетика насыщения:
 а – порфирита; б – известняка

atmosphere by 1.5–3.7 times. Rock weakening in surfactants is accompanied by the lowering of specific consumption of explosives during blasting operations by 1.4 times on average. The use of surfactants to protect from rock bumps makes it possible to increase productivity of relieve slots and wells drilling, reduce their quantity, bring rocks into a condition free of bump hazard, and reduce stress in the marginal massif.

The indicated effects provide for surfactants delivery to the rock massif. That is why the *research problem* of the given work is study of the kinetics of rock saturation with active solutions.

Research results. Experimental works were performed in a laboratory environment with the rock samples of and under solutions delivery into the rock massif. It has been stated [4] that in conditions of solid rocks the mode of multigraded repeated surfactant solutions delivery under low pressure head is the most efficient. In this case, the filtration mode serves only for the delivery of active solution along the existing macro fissures in the depth of the massif. Its further saturation with the solution is performed by means of the mechanisms of capillary condensation and adsorption migration of active molecules along the micro fissures and bonds of mineral grains. These mechanisms play a pivotal role at the surfactants influence upon rocks.

According to the character of filtration processes, all the pores and fissures in a rock massif can be classified as hypercapillary, capillary, and subcapillary pores. In subcapillary fissures the molecules of water are tightly bound to the surface of pores and cannot travel. Penetration and motion of fluid in capillary pores is defined by the physics of the phenomenon of wetting. For this reason, fluid filtration in the rock massif properly depends on the character of its macrostructure, i. e. the concentration and geometry of hypercapillary fissures.

For the comparative analysis of filtration kinetics, apart from surfactant solutions, the process of rock saturation with distilled water was studied. As a criteria characteristic, humidity of rocks was measured as a relation of the mass of rocks at various stages of saturation to the mass of the mineral matrix. As an example, fig. 1 introduces the saturation graphs for porphyrite and limestone which significantly differ in porosity index. Electrolyte solutions and sodium dodecyl sulphate (SDS) were used as surfactants, showing greater effectiveness in the drilling processes.

Saturation of all the familiar rocks with the solutions of various surfactants possesses similar character – rapid growth of humidity in the beginning of the process with further slowdown of the rate of rock saturation with the solutions. Only graphs scales are different. Thus, for thick homogenous limestone with the initial (natural) humidity $W_n = 0.02\%$ maximum adsorption of solutions do not exceed 0.12% (fig. 1, *b*). For more porous porphyrite with $W_n = 0.98\%$ saturation limit is 1.5% (fig. 1, *a*). It will be noted that maximum humidification of rocks exceeds their total porosity, obtained during the experiment with dry rocks, i. e. before humidification. It is explained by active solutions penetration along the bonds of mineral grains omitting pores, and by the increase of porosity (rock jointing) during the actual process of saturation.

Experimentally obtained data, exhibited as points on the graphs, cannot be described by any simple algebraic function. This is because there are three various sections, which are clearly distinguished on the graphs of saturation, corresponding to various stages or phases of saturation.

The first stage – filtration of solutions along the capillary channels. The given stage is defined by free penetration of solutions along adjacent pores and fissures. Theoretical calculations and experience are evidence of the fact that inside fissures (pores) with opening more than 1 mm capillary forces scarcely act. On the other hand, under the use of surfactants, the layer of adsorbed molecules of liquid with rough thickness of $\delta = 10^{-7}$ m develops on the fissures hydrophilic surface, laws of motion of the layer are no longer defined by capillary forces, but by the mechanism of two-dimensional migration. Thus, for surfactant solutions, fissures (pores) with the width of opening $R = 10^{-3} - 10^{-7}$ m should be considered capillary.

Kinetics of solutions in this case is described by the laws of viscous flow of fluid along the capillaries [5, 6]. As far as it penetrates the capillaries, the speed of fluid motion decreases nonlinearly and can be described by an equation:

$$\dot{h} = \frac{dh}{dt} = \frac{R\gamma_{ls} \cos \Theta}{2\eta h},$$

where h – the length of the head of liquid in a capillary at the time point t ; R – fissure opening rate (capillary radius); γ_{ls} – interfacial surface tension between the liquid and rock; Θ – contact angle; η – dynamic viscosity coefficient.

It follows from equation that

$$h = \sqrt{\frac{R\gamma_{ls} \cos \Theta}{2\eta} t}.$$

From general considerations it can be accepted that h will be proportional to wetting W of rock at the given stage of filtration.

Then

$$W_1 = k_1 \sqrt{t},$$

where

$$k_1 \approx \sqrt{\frac{R\gamma_{ls} \cos \Theta}{2\eta}}. \quad (1)$$

Calculation of proportionality factor k_1 at the last formula for real rocks may serve only as approximate assessment of the process. This is conditioned by both of the assumptions, and the fact that the indicated equations are deduced for the single capillary with circular section with constant radius R . In real rocks there are plenty of capillary fissures (pores), and their section is variable. That is why the value of k_1 should be determined experimentally. Processing of experimental data has shown that for various solutions the value of the coefficient varies within the limits $k_1 = (4.9-6.5) \cdot 10^{-6} \text{ s}^{-1/2}$. Average value of $k_1 = 5.6 \cdot 10^{-6} \text{ s}^{-1/2}$. At that, for surfactant solutions the capillary rate saturation k_1 is significantly higher than that of the clear water. This is due to the change by means of active molecules both of interfacial surface tension between the liquid and the rock γ_{ls} , and the contact angle Θ . At the given stage of researches it is impossible to divide the influence of these factors. Simultaneous estimation can be fulfilled according to the formula (1) as:

$$\gamma_{ls} \cos \Theta = \frac{k_1 2\eta}{R}. \quad (2)$$

Accepting that in single set of experiments the alteration of parameters η , γ_{ls} and R is negligible, formula (2) can be reduced to the ratio:

$$\frac{\cos \Theta_{\text{sur}}}{\cos \Theta_0} = \left[\frac{k_{1(\text{sur})}}{k_{1(0)}} \right]^2.$$

In particular, for limestone the addition of active molecules MgCl_2 increases the cosine of the contact angle by 1.27 times.

The second stage – two-dimensional migration of active solutions. For the familiar solid rocks determinative is the second stage of saturation connected with two-

dimensional migration of active molecules along the surface of micro fissures and bonds of mineral grains. There, penetration of solution into the rock is connected with the collective action of several mechanisms. In correspondence with the theory of surface phenomena [7] solution motion is determined by the phenomenon of wetting as well as the active force appearing through the difference between the concentrations of adsorbing molecules of the solution.

There, the role of surfactant molecules is most noticeable. The graphs of saturation (fig. 1) demonstrate significant exceedance of the rock humidity in surfactant solutions as compared to their saturation with water. This is due to the alteration affected by active molecules of the contact angle, greater adsorption capacity of surfactants, as well as inclusion of solvate liquid layers into the fissures by a moving monomolecular layer of surfactant molecules.

Analysis of the deduced functions shows the following. For semi-porous limestones the given stage finishes within first 6 hours of the samples exposition in a solution, and for porphyrite which are more porous, 6 days. Rate of saturation for the second stage is described by the equation:

$$W_2 = k_2 \ln t.$$

Correlation coefficients of logarithmic function in all cases exceed 0.9 which testifies to their statistic significance.

Angular coefficients k_2 can be considered as the rock saturation rate with various solutions. For the familiar rocks its value changes significantly. For porphyrite under water saturation $k_2 = 0.109$, under saturation with the solution AlCl_3 $k_2 = 0.124$, i. e. there occurs increase by 14%. For limestones this value is significantly lower (0.011 and 0.026 correspondingly), but the relative intensity of saturation with the surfactant solution is almost 2.5 times higher than that of the distilled water. It has been stated that such difference is determined by the features of rocks mineral composition. Intergranular bonds and fissures of limestones jointings are made of clay-like chloritized material, content of which amounts to 5–10% of the rock volume. Greater adsorption capacity of such material significantly activates the process of limestones saturation with surfactant solutions.

The third stage – transport of surfactants along the developing fissures. The third stage of saturation is observed only for surfactant solutions. Indeed, when the process of rock saturation with water stops, humidity of samples in surfactant solutions continues growing (fig. 1). It is connected with the development of rock jointing under the effect of surfactants.

The moment of the beginning of the third stage is clearly recorded at saturation graphs and corresponds to the transition of the obviously nonlinear function $W = f(t)$ into the linear one. Indeed, for all the familiar rocks, the best approximation to the experimental data is the equation of the form $W_3 = k_3 t$. Correlation analysis of the experimental data has shown, that for all functions the correlation coefficient is not lower than 0.93. The parameters of the equation: for porphyrite in the solution AlCl_3 – $W_2 = 1.454\%$; $k_3 = 2.74 \cdot 10^{-3}1/\text{day}$, in the solution SDS – $W_2 = 1.448 \%$; $k_3 = 2.51 \cdot 10^{-3}1/\text{day}$; for limestone in the solution MgCl_2 – $W_2 = 0.105\%$; $k_3 = 0.42 \cdot 10^{-3}1/\text{day}$; in the solution SDS – $W_2 = 0.085\%$; $k_3 = 0.35 \cdot 10^{-3}1/\text{day}$.

Thus, the deduced equations make it possible to quantitatively assess kinetics of rock saturation with active solutions at all three stages. However, significantly different mechanisms of saturation at these stages do not make it possible to accurately describe the process with one equation. Additional information can be received from the investigation of the processes of filtration as fractal time sequences.

Investigation of the processes of permeability from the fractal positions are fulfilled on the basis of the *theory of percolation*. In classical statement of the problem, the research problem of the theory of percolation consists in the investigation of some conventional “fluid” trickling through some random inhomogeneous medium [8]. For the first time basic conditions and the term “percolation” itself, which can be translated as “trickle through”, were formulated in 1957 by S. R. Broadbent and J. M. Hammersley in a work dedicated to the problem of gas filters clogging. In their statement, a lattice of random geometry serves a model of percolation medium; the lattice consists of conductive channels (pipes), intersecting in the grid points. If the fluid passes into one of the points or channels, it freely percolates through the whole lattice with the passages being full-open. However, in real media (rocks, for instance) such ideal situation is admittedly impossible. Therefore, it was considered to solve the percolation problem under the condition of random closing of channels, unknown beforehand which exactly.

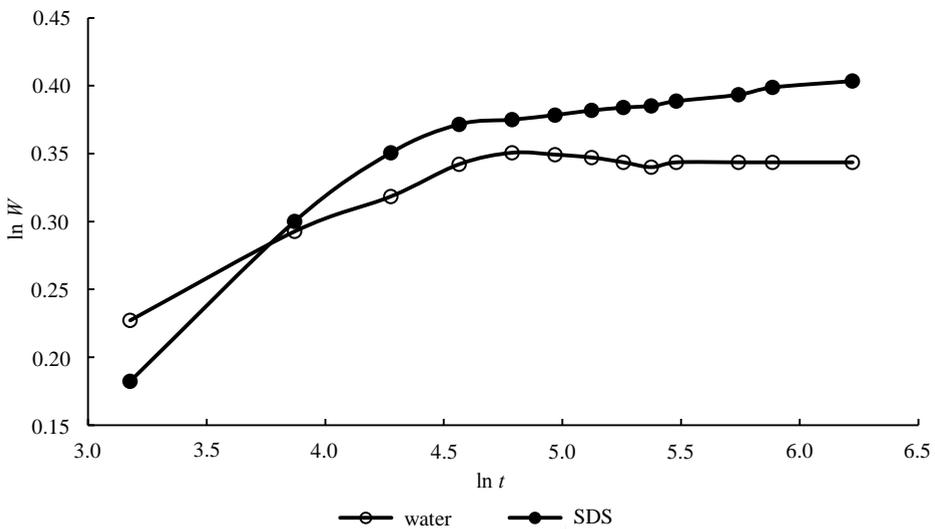


Fig. 2. Logarithmic representation of filtration process
Рис. 2. Логарифмическое представление процесса фильтрации

Suppose each bond of the lattice can be broken with the probability $q = 1 - p$. Then the probability p can be considered as a fraction of overall bonds in the lattice, or a fraction of open, i. e. intercommunicating, channels in rock. When $p = 1$ fluid percolates along the whole lattice and completely fills all open pores. Under sufficiently small p solution spreading in every direction finally meets a broken bond (closed pore channel), and the process of filtration stops. Local domain of solution thrust V_L when $p = p_L$ is called *percolating cluster*, or cluster. In general, percolating cluster is considered to be the system of interconnecting particles which possesses the property of scale self-similarity within the limits $R_0 - R$, where R is the maximum size of the system at which it still retains fractal properties; R_0 is a scale unit of linear measurement.

For surfactant solutions wetness index W is a key parameter, it characterizes the degree of solution penetration into the rock. In accordance with the theory of percolation its dimension can be expressed in terms of the probability of open conducting channels p : $W \sim (p - p_c)^\mu$, where μ – index characterizing the properties of percolation system; p_c – percolation threshold. The given correlation can serve as a basis for a filtration statistical model, where the probability p is variable. When fixing the interval of variability p the results of experimental works of the filtration process presented above should be taken into account. Their analysis shows that surfactant solutions filtration

features spontaneously developing process of solutions penetration, connected with the development of conductive channels. In this case the probability p is higher than percolation threshold p_c , which is accompanied by the formation of an infinite cluster. On the contrary, at clear water filtration p should be posed lower than percolation threshold. At that, the area of wetting forms clusters with finite dimensions, i. e. in the absence of the third stage of saturation the area of fluid distribution will be limited.

For well grounded use of the tools of percolation theory it should be verified that the process of filtration possesses fractal properties. A sign for these features is the exponential function (scaling) of saturation and time expressed by a linear graph in binary logarithmical coordinates [9]. The analysis from the positions of the research results listed before confirms the presence of fractal properties of filtration process. As an example, fig. 2 introduces logarithmic functions $\ln W = f(\ln t)$ for porphyrite saturated with water and SDS solution (fig. 1, a).

Fig. 2 clearly displays the inflection point of two linear sections of graphs. Slopes of lines can be matched with the coefficient of filtration D . Fractal dimension will be defined as $d_w = D + 1$. For the left side of graphs characterizing the first two stages of saturation (capillary filtration and two-dimensional migration) fractal dimension of percolation system under rock saturation with water is $d_{w1(0)} = 1.08$. When adding 0.16% SDS into the water the process activates and fractal dimension increases up to $d_{w1(SDS)} = 1.14$. Filtration of water with the second stage finishes. For surfactant solutions the third stage is observed, connected with spontaneously developing process of conductive channels (fissures) formation. Its dimension for the presented graph is $d_{w2(SDS)} = 1.021$.

Acquired fractal dimensions determine the rate of rock saturation with active solutions:

$$W(t) \sim t^{(d_w-1)}.$$

Permeability of rocks and massifs admittedly depends on the character of conductive channels, i. e. porosity [10, 11]. Many scientists, mainly oil industry experts, research the point in question. One of the basic indirect method of studying porosity is electrical-conductivity measurement of rocks saturated with electrically conducting fluid. Therein empirical equation (*Archie's law*) is used [12]:

$$\sigma = \alpha \sigma_0 \Phi^m,$$

where σ_0 – conductivity of fluid; α – proportionality factor; Φ – porosity as a fraction of pores volume V_p in rock volume V (in the given researches it is an experimentally observable quantity of the total porosity P); m – exponent, depending on the shape of the pore-space (oil experts call it *cementation exponent*).

Important characteristic here is m exponent. It reflects not only the geometry of pores, but in this case, the character of solutions interaction with the surface of the conductive channels. Researches of the given problem reveal that for the majority of real hard rocks m is within the limits from 1 to 2. Researches of a wide range of effusive rocks of the Urals, conducted by the author of this article, make it possible to specify the given exponent. It has been stated that between the maximum water adsorption W_{\max} and total porosity P there is exponential function, the graph of which is presented at fig. 3 in logarithmic coordinates.

Correlation analysis testifies to the statistically significant function with the approximation validity coefficient $R^2 = 0.97$. Pore-space shape exponent $m = 1.54$.

The obtained correlations make it possible to assess the kinetics of rock massif saturation with surfactant solutions. However the process of rock saturation with active

solutions is connected with the injection equipment installation and long-term face break, which discontinues mining technology. While it is evident that in the course of drilling boreholes and wells with flushing using surfactant solutions, active molecules penetrate into rocks and thus have its positive effect. Indeed, rock massif contacts with active solution during the entire period of drilling operations. The forecast calculations for typical conditions of tunnel driving at Severouralsk bauxite mine show, that only over the course of drilling rock massif manages to become saturated with surfactant solution up to 68% from the maximum possible value on average.

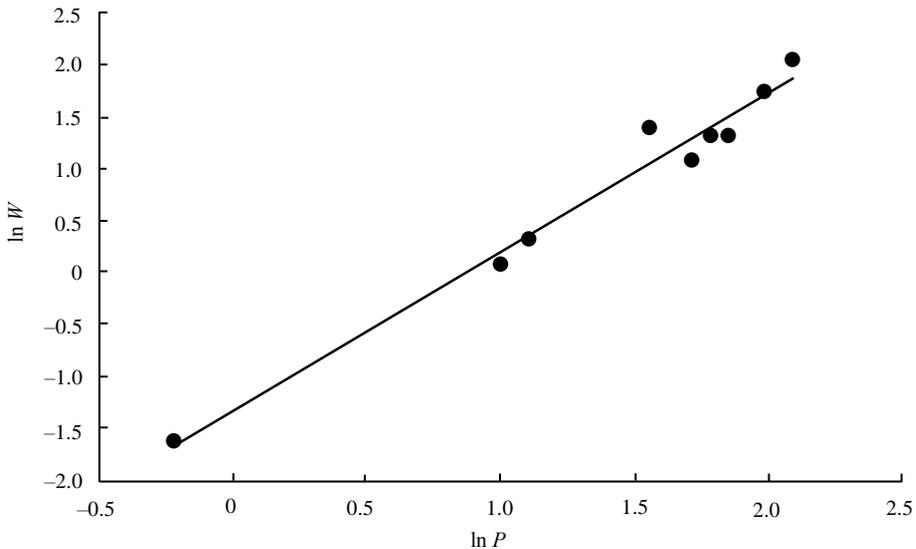


Fig. 3. Correlation of rocks permeability W and their porosity P
Рис. 3. Зависимость проницаемости пород W от их пористости P

Another important effect should be noted. Apart from adsorptive weakening in conditions of cyclic loading typical for the process of rock breaking at drilling, screening effect of surfactant solution is of great importance. During the unloading part of the cycle (for example, at reverse motion of the tool) adsorption layers penetrating into the micro fissures prevent such fissures from closing and healing of such fissures. This effect is especially significant in the mode of fatigue rock breaking.

It is important to note that the acquired assessments are minimal, reliably indicating the lower threshold of rock massif saturation with active solution. In real conditions a greater effect can be expected. It is determined by the following factors. Under the action of rock pressure in the course of drilling operations the process of fissures spontaneous development in rock massif and the third stage of saturation connected with it will continue. Besides, favorable for massif processing with surfactant solutions is the interaction of boreholes and wells as a single set: their influence zones overlapping, merging of fissures at fracture process zones, etc. Experimental industrial surfactant solutions injections in the faces of Severouralsk bauxite mines [3] confirm the given assessments.

Conclusions. Thus, the given investigations make it possible to assess the character of filtration of various active solutions in hard rocks and forecast the degree of the massif saturation in the process of drilling. Study of the kinetics of active solutions from the fractal positions gives valuable information. The acquired correlations are the base for the development of statistical (simulation) models of filtration on the basis of the theory of percolation with Monte-Carlo method.

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ИССЛЕДОВАНИЕ ФИЛЬТРАЦИИ РАСТВОРОВ ПОВЕРХНОСТНО-АКТИВНЫХ ВЕЩЕСТВ В СКАЛЬНЫХ ПОРОДАХ

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Цель. Перспективный путь повышения эффективности процессов горного производства – направленное изменение свойств пород поверхностно-активными веществами (ПАВ). При проектировании технологии использования ПАВ важными задачами являются исследование и прогноз фильтрации активных растворов в обрабатываемых горных породах. Цель исследований – установление закономерностей фильтрации растворов ПАВ в горных породах.

Методология исследований включает экспериментальное изучение и теоретический анализ процессов фильтрации активных растворов в горных породах.

Результаты исследования. В работе на базе экспериментальных исследований установлено, что процесс фильтрации активных растворов происходит в три стадии, отличающиеся механизмом движения активных жидкостей в горных породах. Дается аналитическая оценка параметров данных стадий насыщения. Показано, что процесс фильтрации обладает фрактальными свойствами и может быть исследован с позиций теории перколяции. Установлено, что в логарифмических координатах зависимость количества проникающего в породы раствора от времени описывается двумя линейными уравнениями, соответствующими стадиям фильтрационного насыщения и саморазвивающегося процесса двумерной миграции активных растворов.

Область применения. Полученные результаты используются при проектировании способов и средств управления свойствами и состоянием горных пород и массивов.

Выводы. Установленные закономерности позволяют осуществлять прогноз насыщения породных массивов активными растворами в различных процессах горного производства. Полученные количественные оценки (уравнения) могут служить базой статистического моделирования (метод Монте-Карло) проницаемости горных пород как перколяционной системы.

Ключевые слова: горные породы; поверхностно-активные вещества; механизмы фильтрации; теория перколяции.

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