Строительные материалы и изделия
Construction Materials and Products

journal homepage: https://bstu-journals.ru

ISSN
2618-7183

DOI: 10.58224/2618-7183-2024-7-2-4

Evaluation of the influence of an aggressive environment on the durability of the cement stone

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Abstract. The paper proposes methods for assessing the durability of building materials and structures based on Portland cement when exposed to aggressive environments that mimic the products of the vital activity of bacteria on building materials.

To determine the main parameters of the model of degradation of building materials under the action of aggressive environments, a mathematical model has been developed in the form of integral and differential relations connecting these parameters. A technique for identifying the mechanical characteristics included in these models based on the solution of inverse biodegradation problems has been developed. The analysis of changes in the structure of the cement stone was carried out using the results of computed tomography, and the regularities of the distribution of pores in the cement stone from the time of exposure were obtained.

Based on experimental and numerical studies, it has been established that the mechanism of destruction of cement stone obtained by the traditional method and activation in the vortex layer apparatus is different. The difference lies in the greater accumulation of cement stone interaction products in the activated sample, which is confirmed by a shift in porosity to less than 0.5 mm and a lower solubility value compared to the control composition. The compressive strength of the samples as a result of exposure for 28 days decreased by 37% and 20% for the control and activated compositions. The mass of the studied samples as a result of exposure decreased by 49% and 21%, respectively.

On the basis of this mechanism, a mathematical model of the process of material degradation in an aggressive one is developed, taking into account changes in porosity and acidity concentration, and dependence of material strength reduction are obtained.

Keywords: Portland cement, activation, aggressive environment, degradation, porosity, Fick's law

Please cite this article as: Ibragimov R.A., Shakirzyanov F.R., Kayumov R.A., Korolev E.V. Evaluation of the influence of an aggressive environment on the durability of the cement stone. Construction Materials and Products. 2024. 7 (2). 4. DOI: 10.58224/2618-7183-2024-7-2-4

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Received December 20, 2023; Received in revised form February 19, 2024; Accepted March 29, 2024

1. INTRODUCTION

Concrete and reinforced concrete structures during operation are exposed to various aggressive environments (alkaline and acid environments, exposure to waste products of bacteria and fungi, ultraviolet). As a result, the physical and mechanical characteristics of the material and its durability indicators deteriorate. The durability of concrete characterizes, among other things, the ability to withstand the harmful effects of the environment [1, 2]. It is noted that an acidic environment seriously reduces the durability of concrete [3], the impact of salts based on calcium, tartaric acid [4], acetic acid [5], formic acid [6] quickly leads to the destruction of concrete structures [7-8]. As a result of exposure to aggressive environments, the products of cement stone hydration are leached, which causes an increase in porosity [9]. It is the change in the pore structure of concrete that affects the kinetics of its degradation [10].

The authors [11-12] associate the mechanism of the action of aggressive environments on concrete with the dissolution of cement stone hydration products, which causes a decrease in the pH of concrete, dissolution of $Ca(OH)_2$ and decalcification of CSH gels. When exposed to aggressive media, the compressive strength of cement mortars decreases as a result of Ca_2^+ diffusion , as well as a result of a change in porosity [13-14]. An increase in temperature from 25 to 85 °C causes an increase in the leaching kinetics by a factor of 2-6 [15], and exposure to an electric field enhances degradation by a factor of 100 [16]. Therefore, it is necessary to predict the service life of building structures exposed to aggressive environments. One of the problems in developing methods for predicting the durability of building materials is the experimental determination of the kinetic dependence of changes in the properties of the material under separate and combined action of operational factors [17-18]. Various models are used to describe the behavior of materials when exposed to aggressive media. Thus, the physical nonlinearity of concrete is described by the Mazars damage model, and the Nernst-Planck-Poisson equation is used to describe fluid migration in the body of concrete [19-20]. The authors of [18, 21] developed a degradation model in which a decrease in the mechanical properties of a material is caused by sources of biodamage, reinforcement corrosion, and crack development. This model takes into account the decrease in the cross-sectional area of the concrete structure according to empirical relationships that take into account the time factor. Concrete damage caused by diffusion is estimated by the authors of [22] by introducing the law of mechanical failure, in which a probabilistic analysis is performed using the simulation of the Monte Carlo method. Using Fick's law to determine the time of onset of corrosion of reinforced concrete structures, and modeling the stress-strain state using the Monte Carlo method allows you to determine the probability of destruction of structures [23]. The diffusion of aggressive media into the structure of concrete is modeled by Fick's first law, according to which the dissolved substance diffuses in proportion to its concentration gradient [24]. In the case of using a material such as concrete, the diffusion coefficient of the material must be taken into account [25]. There are numerical studies of the degradation of reinforced concrete structures using the finite element method [26-27]. However, they did not take into account the degradation of concrete over time, taking into account changes in the porosity and structure of the cement stone. This is an actual scientific problem that needs further solution.

It is known that the resistance in aggressive environments of cement compositions obtained by activation of the binder in various kinds of vibratory mills increases [28-31]. However, in the above works, there are no models of concrete degradation as a result of exposure to such media. In this regard, this article proposes a numerical model for determining the durability of concrete and reinforced concrete structures in aggressive environments based on the finite difference method using Fick's second law.

2. METHODS AND MATERIALS

2.1. Materials and objects of study

When conducting research, we used portland cement CEM I 42.5 B (Novotroitsk, Russia), which meets the requirements of EN 197-1. The specified portland cement has the following mineralogical composition: C_3S – 64-65 %, C_2S – 11-13 %, C_3A – 5-6 %, C_4AF – 14-1 %. The average density of cement stone was determined on 2x2x2 cm cube samples dried to constant weight at a temperature of

105 °C. The compressive strength of the cement stone was determined on 2x2x2 cm cube samples (series of 6 samples).

Processing of Portland cement was carried out in a vortex layer apparatus (ABC) model 297, manufacturer LLC "Regionmettrans". The operating principle, apparatus design, and processing modes are described in [32]. In accordance with [33], the composition of the model medium (a mixture of carboxylic acids) was adopted, which imitates the products of vital activity of microorganisms in the pore fluid of concrete, presented in Table 1. To increase the degradation rate, the concentration of acids was increased to 10 % [34].

To conduct research on the impact of an aggressive environment on cement stone, cube samples 2x2x2 cm in size were placed in a model environment. The ratio of the solution volume, cm^3, to 1 cm^2 of the surface of the samples was assumed to be 25:1. In the process of testing for 28 days, the following parameters of the model environment were maintained at a constant level: temperature 20 ± 1 °C, the pH value of the medium was maintained constant and equal to 3. Samples were examined by X-ray computed tomography before exposure to an aggressive environment and after 14 and 28 days of exposure. On the basis of the obtained experimental data, the kinetic dependence of the change in the strength characteristics of the samples on the time of their exposure to the test medium were plotted. The structure of the cement stone was determined by X-ray computed tomography using the General electric V|tome|X S 240 (Germany). The studied samples were photographed using a microfocus tube at a voltage of 130 kV and a current of 130 mA. The shooting conditions for each sample were adjusted separately depending on the density characteristics of the minerals that make up the sample.

Table 1. Composition of the model environment.

Inorganic ions, mg/l			
Ca^{2+}	NO_3^{-}	S^{2-}	CO_3^{2-}
640	60	1000	700
Organic acids, mg/l			
Acetic		Lemon	Apple
1980		16020	400

2.2. Study Model

To assess the durability of concrete and reinforced concrete structures in an aggressive environment, it is necessary to determine the amount of a substance penetrating into the cement composite. To do this, use the ratio based on Fick's second law:

$$\frac{\partial C}{\partial t}=\frac{\partial}{\partial x}\left(\lambda\frac{\partial C}{\partial x}\right)+\frac{\partial}{\partial y}\left(\lambda\frac{\partial C}{\partial y}\right), \tag{1}$$

where C is the pH value of the pore fluid of the cement stone; λ is the diffusion coefficient; x, y – coordinates; t is time. The boundary conditions were taken as follows: $\mathrm{C}=\mathrm{C}_k$, at $x = 0$ and at $x = b$, or $y = 0$ and $y = h$, where C_k – pH of the aggressive environment.

The solution of equation (1) is carried out in finite differences both in time and in the coordinate, and the explicit scheme was used in time:

$$\frac{C_{ij}^{k+1}-C_{ij}^{k-1}}{\Delta t}+=\frac{\left(\lambda_{i+1j}^{k}-\lambda_{i-1j}^{k}\right)\cdot\left(C_{i+1j}^{k}-C_{i-1j}^{k}\right)}{\Delta x^2}+\frac{+\left(\lambda_{ij+1}^{k}-\lambda_{ij-1}^{k}\right)\cdot\left(C_{ij+1}^{k}-C_{ij-1}^{k}\right)}{\Delta y^2}+ \\ +\lambda_{ij}^{k}\left(\frac{C_{i+1j}^{k}-2C_{ij}^{k}+C_{i-1j}^{k}}{\Delta x^2}+\frac{C_{ij+1}^{k}-2C_{ij}^{k}+C_{ij-1}^{k}}{\Delta y^2}\right). \tag{2}$$

To ensure the convergence of the solution of equation (2), a restriction (Courant condition) is imposed on the time step:

$$\lambda \Delta t\left(\frac{1}{\Delta x^2}+\frac{1}{\Delta y^2}\right) \leq \frac{1}{2}. \tag{3}$$

The permeability coefficient λ depends on the porosity p of the material:

$$\lambda = \lambda_0 \cdot p^{\alpha}, \tag{4}$$

where λ_0 is the initial permeability coefficient, α is the porosity parameter. The change in porosity will be taken as:

$$p = 1 + (p_0 - 1)e^{\beta t(C_b - C)}, \tag{5}$$

where p_0 is the initial porosity of concrete, β is the kinetic parameter, C_b is the initial reduced pH level of the pore medium of the cement stone. As the aggressive medium penetrated into the cement composite, based on the theory of limit equilibrium, the limit load $P^*(t)$on the test sample was determined according to the equation:

$$P^*(t) = \int_A R_b(C,t)\,dA, \tag{6}$$

where $R_b$is the compressive strength, A is the cross-sectional area of the sample.

The method for solving the problem was as follows. At the first step, the initial, and boundary conditions for the concentration of an aggressive medium in concrete are set. Then new concentrations of the aggressive medium are determined at different points of the concrete at a new time step. Further, the issue is solved iteratively in time, i.e., at each step, the concentration of the aggressive medium is determined through the previous values, taking into account the boundary conditions. After that, the ultimate load of the sample determined by equation (6). To solve the problem, a program developed in the Julia language.

3. RESULTS AND DISCUSSION

3.1 Results of natural tests

Using X-ray computed tomography, orthogonal sections were obtained, 3D visualization of the studied samples before exposure (K1, A1), after 14 days (K2, A2) and 28 (K3, A3) days of exposure in an aggressive environment (Fig. 1). The pore size distribution was established depending on the exposure time in an aggressive medium (Fig. 2).

The nature of the change in the distribution curves indicates the course of the process of dissolution of the components of the cement stone of the control composition. The proportion of pores with a size of 1.0 and 1,5 mm increases, and the proportion of pores with a size of 0,5 mm slightly decreases (Fig. 2, a). The course of dissolution processes and filling the pore space for cement stone from activated Portland cement with the products of the interaction of portlandite with citric acid (calcium citrate). In this case, the balance of pores with sizes of 1,0 and 1,5 mm decreases, while the proportion of pores with sizes of 0,5 mm increases (Fig. 2b).

A change in the volume and mass of cement stone is also observed: the control sample decreases on average from 7669 mm^3 to 6333 mm^3 and from 16,29 g to 10,13 g (after 14 days of exposure) and to 4714 mm^3 and 8,32 g (after 28 days of exposure), that is, by 17 % and 38 % (volume) and 37,8 % and 49 % (mass), respectively. The volume and weight of the cement stone of the sample obtained by activation decreases on average from 7797 mm^3 to 7606 mm^3 and from 19,185 g to 16,615 g (after 14 days of exposure) and to 6799 mm^3 and 15,07 g (after 28 days of exposure), that is, by 2,5 % and 13 % (volume) and 13 % and 21 % (mass), respectively.

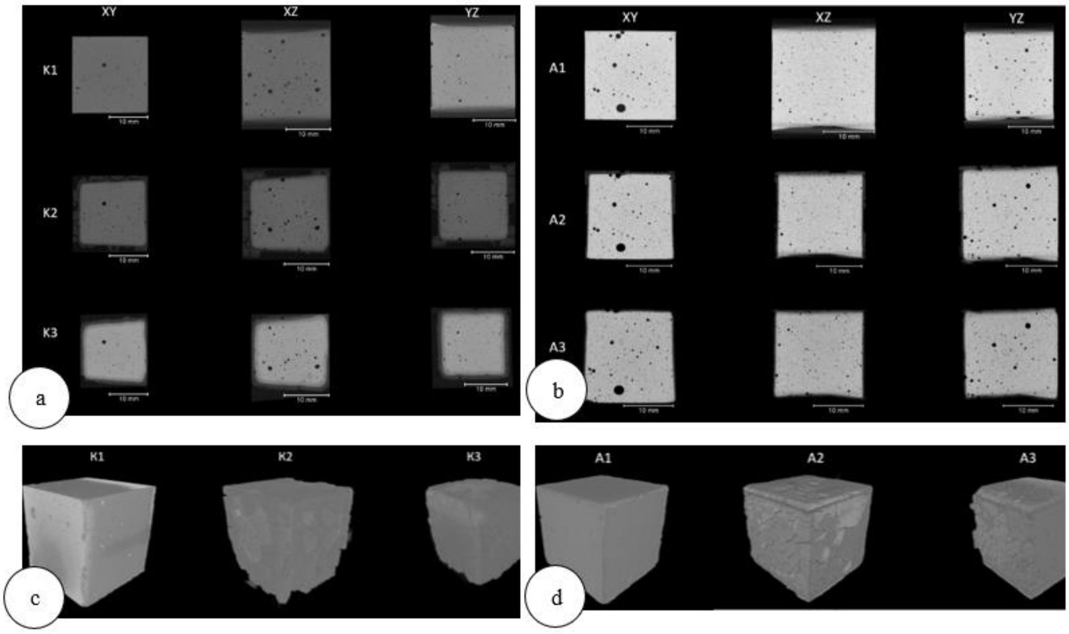


Fig. 1. Orthogonal section and 3D visualization of samples: a) and c) – control; b) and d) – obtained by activation in ABC.

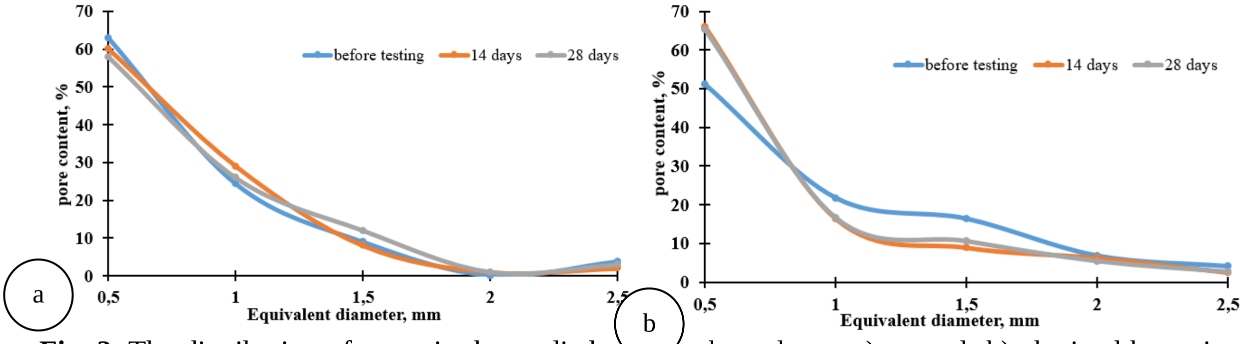


Fig. 2. The distribution of pores in the studied samples by volume: a) control; b) obtained by activation in ABC.

Obviously, the aggressive environment has an effect on the external geometric characteristics of the sample and on the internal volume of the pore space. The mass absorption of samples exposed to an aggressive environment reflects the processes occurring in the internal pore space of the material (Fig. 3). In the initial period (up to 4 days), a regular increase in mass absorption is observed, associated with the penetration of an aggressive medium into the volume of the sample through pores and capillaries. Then there is a decrease in mass absorption due to chemical interaction and the removal of the dissolved substance from the sample volume. From the material balance equation, it can be written that the change in mass absorption in this case will be equal to:

$$W_m = \frac{1}{m_0}\left(\left(V_{pp} + V_{pr}\right)\rho_s - V_{pr}\rho_t\right) \tag{7}$$

where $V_{pp}$is the volume of the pore space filled with an aggressive medium; V_{pr} is the volume of the dissolved substance; $m_0$is the initial mass of the sample; ρ_s is the density of the aggressive medium; ρ_t – the density of the components of the cement stone, interacting and dissolving in an aggressive environment.

It follows from this equation that for $(V_{pp}+V_{pr})\rho_s < V_{pr}\rho_t$ and $\rho_t > \rho_s$ the mass absorption decreases, as shown in Fig. 3. In addition to dissolution, as shown by the data in fig. 2, the process of filling the pores with the products of chemical interaction takes place. For cement stone of the control composition, this process is less intense compared to cement stone on activated Portland cement. This difference is also clearly seen in Fig. 3: the change in mass absorption for the control composition proceeds more intensively. The processing of the obtained results by X-ray computed tomography in Amira-Avizo software is shown in fig. 4. The change in the quantitative content of pores in the samples during degradation is presented in Table 2.

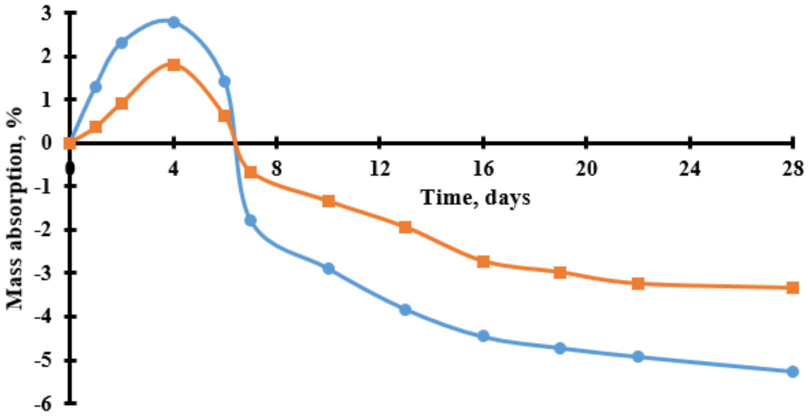
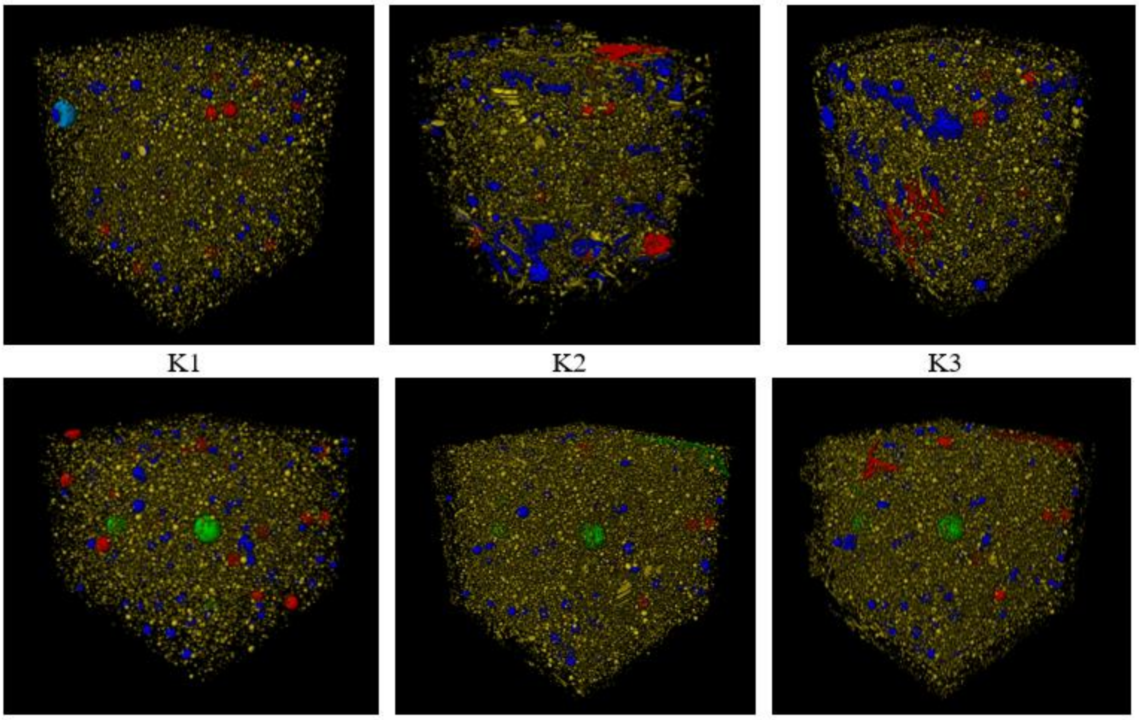


Fig. 3. Kinetics of mass absorption in a model environment: ○ – control sample; □ – sample obtained from activated Portland cement.


K1 K2 K3

A1 A2 A3

Fig. 4. 3D visualization of pores in samples.

Table 2. Changes in pore volume in samples during exposure.

Sample type	Total pore volume, mm^3		
	Before testing	After 14 days. tests	After 28 days. tests
Control	84,7	82,7	72,4
Received by activation	70,7	39,2	35,2

According to Table 2, it can be seen that with an increase in the exposure of samples to an aggressive environment, the total pore volume decreases in the studied compositions. This may indicate that the products of the interaction of the aggressive environment (calcium acetate, calcium citrate) precipitate and are deposited in the pores and capillaries of the cement stone. This circumstance is confirmed by the data in Fig. 2.

3.2 Numerical studies of changes in the compressive strength of cement stone aged in an aggressive environment

The compressive strength of cement stone aged in an aggressive environment was determined at various exposure times (Table 3).

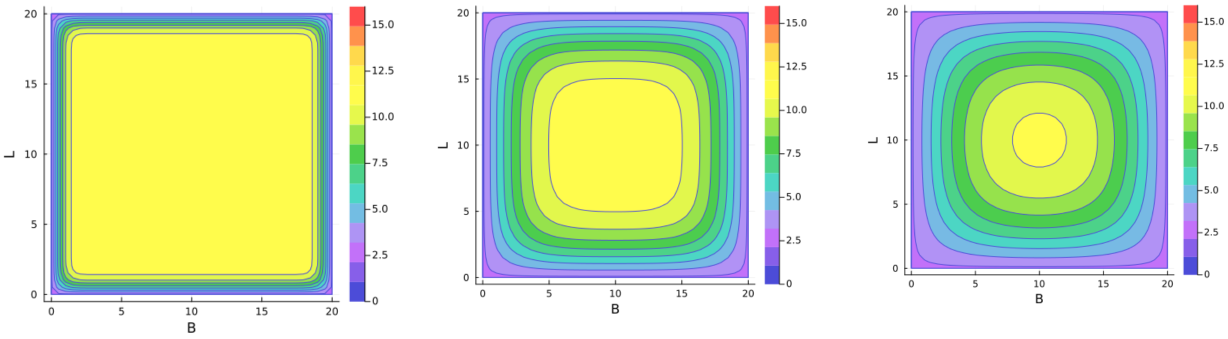
Table 3. Compressive strength of cement stone.

Compound	Compressive strength, MPa		
	before exposure	after 14 days of exposure	after 28 days of exposure
control	42,5 100 %	29,8 70 %	26,8 63 %
obtained by activation	54,8 100 %	47,1 86 %	43,7 80 %

The obtained experimental results are analyzed by a numerical method. Based on the analysis of kinetic dependencies, it was found that the decrease in strength characteristics depending on the pH value of an aggressive medium can be represented as the following function:

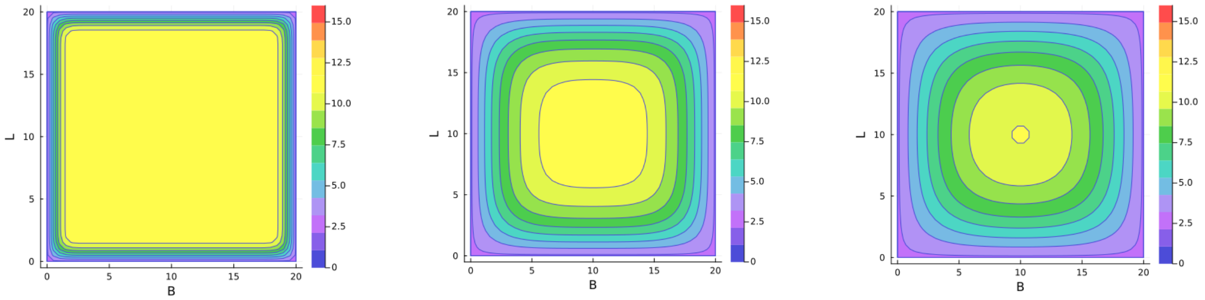
$$R_b = R_{b0} + R_{b1}t + R_{b2}t^2, \tag{8}$$

where R_{b0}, R_{b1}, R_{b2} – mechanical (strength) characteristics, R_b – compressive strength. On fig. 5 and 6 it is shown the results of numerical calculations.



K1 K2 K3

Fig. 5. The process of diffusion of an aggressive medium into a control sample.



K1 K2 K3

Fig. 6. The process of diffusion of an aggressive medium into a control sample, taking into account changes in porosity and permeability coefficient.

To determine the permeability parameters and change the porosity values, the following data were taken into account:

1) the porosity of the cement stone *p* (sample before exposure) was determined according to X-ray tomography, where the pore areas were separated and statistical processing of the porosity results was carried out.

2) the permeability coefficient λ_0 is obtained by solving the inverse problem of water penetration into a cement-sand mortar sample.

Based on these data, the parameters α, β, R_{b0}, R_{b1}, R_{b2} indicated in formulas (5) and (8) were calculated: $p_0 = 0{,}25$; $\alpha = 1{,}2$; $\beta = 0{,}01$; $\lambda_0 = 0{,}3$. In formula (8) the values of the coefficients for the control sample: $R_{b1} = -1{,}25$; $R_{b2} = 0{,}0247$; for the sample obtained by activation - $R_{b1} = -0{,}703$; $R_{b2} = 0{,}0109$.

On Fig. 7 shows the dependence of the pH value on the duration of the penetration of an aggressive medium into the material volume at constant and variable permeability coefficients.

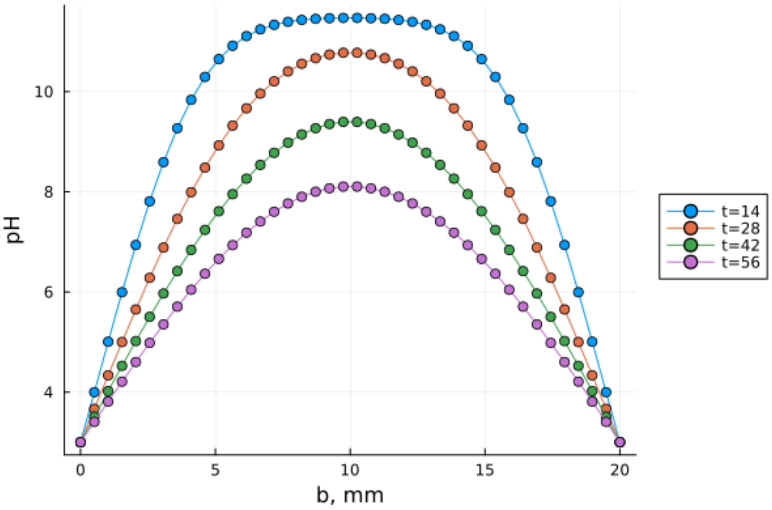


Fig. 7. The dependence of the pH index on the time of penetration of an aggressive medium into the sample (B is the width of the sample, t is the time in days).

4. CONCLUSIONS

An aggressive environment, which is a model environment for the products of vital activity of microorganisms, affects the porosity and pore size distribution in the studied cement composites. So, in the control sample, as a result of exposure, the number of pores with a diameter of 1,0 and 1,5 mm increases, and in the sample obtained by activation, the number of pores with a diameter of 0,5 mm increases. The volume of samples decreases by 38% and 13% for the control and activated formulations after 28 days of exposure. In addition, a decrease in the mass absorption of samples is recorded (after 4 days of exposure) due to the dissolution of the hydration products of Portland cement in an aggressive environment.

The compressive strength of the samples as a result of exposure for 28 days decreased by 37 % and 20 % for the control and activated compositions. The mass of the studied samples as a result of exposure decreased by 49 % and 21 %, respectively.

In order to assess the destruction of the cement stone by identification methods, the parameters of the process of penetration of an aggressive medium into the volume of the material were determined: the permeability coefficient, the porosity parameter and the kinetic parameter.

The dependence of the impact of an aggressive environment on the parameters of the durability of cement composites at different exposure times are obtained, on the basis of which a mathematical model of the process of material degradation in an aggressive environment is developed, taking into account changes in porosity, acidity concentration, and dependence of the decrease in the strength of the material are obtained.

A further direction of research is the determination of the permeability coefficients of heavy concrete of various classes. This will allow modeling the process of concrete degradation in time under given conditions and predicting the service life of structural elements in an aggressive environment.

ACKNOWLEDGES

The study was supported by the Kazan Federal University under the state assignment in the field of scientific activity, project No. FZSM-2023-0009

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