Variety of structure and changes in the properties of building composites over time

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Abstract. The potential possibility of increasing the level of mechanical properties of composites on mineral binders remains during the long period. The initial structures hereditarily determine the change of the properties of the finished material in time. It can be assumed that an increase in the structural diversity of concretes should lead to such the organization of a structure that will guarantee the preservation or increase of the level of quality indicators at long terms of hardening (up to 720 days). The conducted researches showed that concrete of long hardening with the structure organized at selective adhesion of a cement matrix to the surface of dense or porous aggregates differed are more resistant to external influences. That is, those concretes, organization of structure which happened under the conditions of diversity of interphase interactions at the level of a macrostructure. This was ensured by the use of aggregates with various condition of a surface. The directed change of a ratios of adhesive-cohesive bonding forces at the surfaces of partition between a cement part and the surface of an aggregates provided structures, which caused increase the compressive strength of concrete by 18% and the module of elasticity by 24% at reducing the water by 29-36% and the open capillary pore volume by 44%. The structure of concrete organized at selective adhesion was the densest in comparison with of concrete of other structures. At the same time, the damage of material by technological cracks and inner surfaces of partition has grown more for concrete, the structure of which was provided by a variety of conditions of interactions between the cement matrix and aggregates. Favourable change of the parameters of these elements, along with the processes of hydration of relict binder grains, causes an improvement in time of indicators of the mechanical and deformative properties of concrete. Thus, structural diversity determines the potential changes of the concrete structure at long-hardening that must be taken into account at designing compositions and technology of the concrete, intended for difficult operating conditions.

1. Introduction

The concept of the "life cycle" of structures, buildings and structures is based on certain requirements. First, an assessment of the consumer value of construction projects should be carried out, depending on the provision of the required level of physical and technical properties of materials, taking into account the solution of the tasks of reducing their material consumption. Secondly, attention should be paid to the performance of the functions laid down in the material during the normalized operation time of structures and products. Maintaining the adjusted quality indicators of the cement and concrete component of structures and products during their long-term operation is especially important in case of probable changes in the intended purpose of construction projects. The maintenance of the design level of properties is provided by the structure that alone can guarantee the durability of the composites over time. For this, the structure must include a certain set of elements, the interaction of which is capable of determining an acceptable change in the physical and technical characteristics of the material of structures and products. The define role in the dynamics of the development of the entire structure, and, therefore, changes in properties affect cracks, internal interfaces and residual deformations as active elements of the structure, which instantly react to any intrastructural changes. The specific internal
design of the structure, spontaneously organized by the beginning of the operation period, has the potential to perform certain actions aimed at self-preservation of the structure (product) as a single integral object without significant loss of functional features. Regulation of the variety of sets of components at different large-scale levels of organization of the structure makes it possible to obtain building composites with increased quality indicators. It is possible to purposefully create conditions for the formation of a certain "spectrum" of structures in the structure of one construct by increasing the variety of qualitative and quantitative compositions of binders. The prerequisites for the implementation of adequate behaviour of composites under changing conditions are set by the structure, which should ensure the preservation of the values of crack resistance and the level of mechanical characteristics over time. The authors proceeded from the assumption that an increase in the variety of parameters of active elements of the structure should have a positive effect on the change in the properties of materials over time. Determination of indicators of fracture toughness and strength of cement compositions after 8 years of hardening in natural conditions and assessment of the influence of a variety of structures of different ages on the change in the properties of composites over time was designated as a research task.

2. Literature review
The conducted review of scientific and technical information showed that at the moment there is no unified theoretically substantiated way for assessing the increase in the strength properties of concrete. Calculation of changes in concrete strength over time can be carried out in several ways: - according to the logarithmic law; - using generalized experimental strength growth factors; - based on the determination of the effect of an increase in the activity of the cement component or a decrease in the water-cement ratio on the growth rate of concrete strength through its nominal grade strength. Traditionally, most of the research is devoted to addressing the issues of improving the mechanical characteristics of building composites at a relatively early age [1, 2, 3, 4, 5]. Taking into account that the most significant investigation on the research of the strength of cement compositions and concretes in the late periods of hardening were carried out several decades ago, there are certain restrictions on the use of their results at the present time. The previously established quantitative ratios of concrete strengths at different curing times aren't rational to use in calculations without experimental verification and adjustment in accordance with the characteristics of modern cements and concrete production technology. Thus, an important problem is the accumulation of experimental results on determining the strength of cement stone, as well as concretes on dense and porous aggregates, over time.

3. Statement of the purpose and objectives of research
The aim of the research is to increase the level of properties of cement stone and concrete over time by increasing the diversity of the potential of their initial structure by introducing polydisperse fillers and changing the ratio of adhesion-cohesive bond forces at the interfaces between the cement matrix and the surface of fillers in concrete of the same composition. Research objectives: to determine the indicators of fracture viscosity and strength of cement compositions after 8 years of hardening in natural conditions and to assess the effect of a variety of structures of different ages on the change in the properties of composites over time; analyse the conditions for the initial structure formation of concrete at the macrolevel and determine the change in the properties of heavy concrete and expanded clay concrete after long hardening.

4. Methods of research
Cement compositions, including quartz fillers of rational qualitative and quantitative compositions, and concretes on granite crushed stone and expanded clay gravel were accepted as objects of research. For the manufacture of samples, cement was used as a binder, obtained by joint grinding in a laboratory ball mill of clinker manufactured by Closed Joint-Stock Company "Odessacement" and two-water gypsum stone in the amount of 5% of the cement mass.

To control the organization of the structure of cement compositions in order to increase the diversity of its characteristic components (clusters, initial cracks and internal interfaces, residual deformation
fields), the amount of fillers \( N = 20 \pm 10\% \) of the volume of cement and their specific surface area \( S = 300 \pm 200 \text{ m}^2/\text{kg} \). Fillers were introduced in various mono- and polydisperse ratios.

The experiments were carried out on standard beams 40x40x160 mm in size. The following properties of the samples were controlled: technological (initial) damage through the damage coefficient \( (K_d, \text{cm/cm}) \) [2]; compressive strength and flexural tensile strength \((R_c, \text{MPa}, R_t, \text{MPa})\) according to the standard method; crack resistance with the recommended methods of crack initiation by means of stress intensity factors \( (K_{isc}^r, K_{ics}^r)\).

The assessment of the influence of the diversity of the structure of different ages on the change in the properties of the composites was determined as the ratio \( F = Q^k / Q^{k+5} \), where: \( \Delta F(Q) \) is the change in the property index of the cement stone over time; \( Q^8_{\text{years}} \) - an indicator of the properties of a cement stone at the age of 8 years; \( Q^{28\text{days}} \) is an indicator of the properties of a cement stone at the age of 05 years (200 days) from the moment of hardening.

The study of the properties of concrete was carried out on samples-cubes made from a concrete mixture on dense and porous aggregates with different surface conditions. Together with traditional compositions of heavy and light concrete, compositions were used that contained aggregates that were fully or partially processed by GKR-11. Grains of crushed granite and expanded clay gravel were subjected to hydrophobization by immersion in a water repellent solution at a concentration of 2% from weight, followed by drying at T100C. This made it possible to provide different conditions for the interaction of the cement matrix with the surface of the aggregates, even with the same concrete composition: \( R_{A, c} > R_{K, c} \), the adhesion of the matrix material to the aggregates is greater than its cohesive strength (concrete of composition I); \( R_{A, c} < R_{K, c} \), there is no adhesion of the matrix component to the surface of the aggregates (concrete of composition II); \( R_A = R_K \), volumes with perfect and imperfect adhesion of the matrix material to the surface of the aggregates (concrete of composition III) coexist in the material. \( R_A – \) is the adhesion of the matrix to the surface of the aggregates, \( R_K – \) is the value of the cohesive strength of the matrix material.

During the constant mobility of the concrete mixture, which required adjusting the compositions according to the values of the water-cement ratio, concretes with the corresponding property indicators were obtained. The change in the following properties of concrete and expanded clay concrete over time was monitored: average density \( (\rho_{av}, \text{kg} / \text{m}^3) \), volume of open capillary pores \( (P_{oc}, \%) \), water absorption \( (W, \%) \), velocity of ultrasonic waves \( (U, \text{m/c}) \), technological damage \( (K_d, \text{cm} / \text{cm}^2) \), modulus of elasticity \( (E, \text{MPa} 10^3) \) and compressive strength \( (f_{ck\text{-}cube}, \text{MPa}) \). Property indicators were determined using standard methods.

The damage was quantitatively assessed by the following damage coefficients:
- as the ratio \( K_{d1} = \Sigma L / S, \text{ cm} / \text{cm}^2 \), where: \( \Sigma L_i \) - is the total length of technological cracks and internal interfaces on the selected surface of the sample, cm; \( S - \) fixed surface area of the sample, cm²;
- as the ratio \( K_{d1i} = \Sigma L_i / L_{d1}, \text{ cm} / \text{cm} \), where: \( \Sigma L_{di} \) - the actual length of the fracture crack, cm; \( L_{d1} \) - is the shortest distance between the points of exit of the actual fracture crack on the end surfaces of the sample, cm.

The impact of the age of concrete samples on the change in the level of their properties \( (Q) \) was assessed according to the corresponding coefficients: \( \Delta \rho_{av}, \Delta P_{oc}, \Delta W, \Delta U, \Delta K, \Delta E, \Delta F \), which were determined as: \( \Delta Q = f_{ck\text{-}cube}^{720} / f_{ck\text{-}cube}^{28} \), where: \( \Delta Q \) – change an indicator of a specific property of concrete; \( Q^{720} \) - indicator of concrete properties at the age of 720 days; \( Q^{28} \) – is an indicator of concrete properties after 28 days of normal hardening.

5. The results of research.

Preservation of a preassigned level of properties it through time can be ensured by intensive changes in the structure, the adequacy of the manifestation of which depends on the possibility of realizing the interrelationships of all its individual components. This, in turn, is determined by the internal potential of the structure, which largely depends on the diversity of the set of its constituent parts.

The polyminerality and polydispersity of cement compositions a priori leads to the genesis of cluster structures with a unique combination of particles in them in terms of mineralogical composition, size
and quantity. The processes of multi-scale clustering and the emergence of “structures in the structure” interacting with each other to form even more hierarchically complex structures suggest a potential variety of parameters of initial cracks, interfaces, residual stresses and deformations. These integral elements of the structure of any composite material determine its potential for timely adaptation to internal and external influences. Taking into account the possible change in the operating conditions of building objects, it is necessary to provide for situations requiring the appropriate resistance of the material of products and structures. The prerequisites for the implementation of adequate behaviour of composites under changing conditions are set by the structure, which should ensure the preservation of the values of crack resistance and the level of mechanical characteristics over time. In this case, they proceeded from the assumption that an increase in the variety of parameters of active elements of the structure should have a positive effect on the change in the properties of materials over time.

The research results are presented in Table 1 and Table 2.

Table 1. Assessment of changes in the properties of cement compositions with monodispersed fillers over time

| Indicator | The compositions of fillers |  |
|-----------|-----------------------------|-----|
| ΔF=Q^{28day}/S | S_1=100 m²/kg | S_2=300 m²/kg | S_3=500 m²/kg |
|            | 10% | 20% | 30% | 10% | 20% | 30% | 10% | 20% | 30% |
| ΔF(P) | 0.97 | 1.00 | 1.03 | 1.00 | 0.99 | 1.04 | 0.97 | 1.03 | 1.07 |
| ΔF(Rc) | 1.36 | 1.06 | 1.41 | 2.57 | 1.43 | 1.35 | 1.98 | 1.83 | 1.40 |
| ΔF(R_S) | 1.01 | 1.37 | 1.25 | 1.09 | 1.16 | 1.17 | 1.11 | 1.00 | 0.98 |
| ΔF(K1S^2) | 0.52 | 0.80 | 0.21 | 0.36 | 0.31 | 0.78 | 0.38 | 0.38 | 0.44 |
| ΔF(K1S^R) | 0.76 | 0.63 | 0.45 | 0.41 | 0.35 | 0.50 | 0.80 | 0.51 | 0.61 |

Table 2. Assessment of changes in the properties of cement compositions with polydisperse fillers over time

| Indicator | The compositions of fillers |  |
|-----------|-----------------------------|-----|
| ΔF=Q^{28day}/S | 50%S_1 | 50%S_2 | 50%S_1 | 50%S_2 | 30%S_1 | 30%S_2 + | 30%S_1 | 30%S_2 |
|            | 10% | 20% | 30% | 10% | 20% | 30% | 10% | 20% | 30% |
| ΔF(P) | 1.04 | 1.06 | 1.01 | 1.03 | 1.05 | 1.04 |
| ΔF(Rc) | 2.20 | 1.81 | 1.76 | 1.59 | 2.04 | 1.86 |
| ΔF(R_S) | 1.79 | 1.59 | 1.79 | 1.24 | 1.14 | 1.30 |
| ΔF(K1S^2) | 0.48 | 0.49 | 0.94 | 0.51 | 0.89 | 0.49 |
| ΔF(K1S^R) | 0.73 | 0.82 | 0.80 | 0.52 | 0.98 | 0.72 |

Cracks and internal interfaces are elements of the structure that arise at the initial stages of its organization and hardening. This presupposes the participation of cracks and interfaces in all further processes of the structural development of the material for many years. The presence of cracks and interfaces causes an uneven distribution of various deformations and stresses that permanently arise in the cement stone, due to the fact that the processes of hydration and the processes of internal mass transfer do not stop in it. A constant increase in density fluctuations and a permanently redistribution of local residual deformation fields was provided. This maintains the structure of the material in a non-equilibrium state. Permanent change in the structure of the cement stone will determine its damage by technological cracks and internal interfaces, fracture toughness, compressive and bending strength.

This maintains the structure of the material in a non-equilibrium state. Permanent change in the structure of the cement stone will determine its damage by technological cracks and internal interfaces,
fracture toughness, compressive and bending strength. This manifests itself as structural changes in the material due to the fact that all the constituent structures are interconnected and mutually affect the state of each other. These are determined by the reorganization of the integral structure of the cement stone. The use of polydisperse fillers leads to a potential increase in the diversity of the material structure, regardless of the accepted ratios of sizes and number of particles. This, as shown by researchers to determine the damage of cement samples, leads to an increase in $K_1$ values during prolonged hardening under normal conditions. Damage to samples with monodisperse fillers increased only at certain compositions, or practically did not change. The interaction of filler particles with the multivariance of the ratios of their geometric parameters led to the organization of primary cluster structures, which provided a more varied set of initial cracks and internal interfaces, which influenced the changes in the structure of the cement stone over time.

Technological damage determines the cracking resistance of materials. Analysis of the results obtained for the determination of $K_{1c}$ and $K_{1c}'$ showed a general tendency towards a decrease in the failure viscosity of cement stone during long-term hardening; by 24-41% for samples with polydisperse fillers and by 44-53% for samples with monodisperse fillers. At the same time, during using filler compositions with a specific surface area of $S_1+S_2$ or $S_1+S_3$ in a 1:1 ratio, it is possible to increase the cracking resistance of a cement stone to 11% and 20%, respectively. After the introduction of fillers with the composition $33\%S_1+33\%S_2+33\%S_3$, the failure viscosity over time of the hardened cement compositions practically didn't change.

Strength indicators increase with time for all the accepted compositions of polydisperse fillers, up to 31% ($R_1$) and 47% ($R_2$). The structures formed upon the introduction of monodisperse fillers ensured an increase in the strength of the samples in bending and compression, on average, up to 13% and 30%, respectively. In systems with a wide variety of structures, the effect of the accepted age of the samples on the change in compressive strength was up to 2 times higher than those of systems that included filler particles close in specific surface area. Thus, depending on the composition of the fillers, the parameters of the substructures are set at the level of interaction of the binder particles. Thus, the conditions of hydration are determined, which affects the provision of sets of cracks and internal interfaces, on which the potential ability of the cement stone to reorganize the structure over long time intervals depends. Favourable structural changes lead to an increase in the strength of the cement samples.

The potential structure of the concrete begins to organize with the final distribution of the initial components after the completion of the formation of the product. The limited manifestation of sedimentation phenomena in the compacted concrete mix makes possible a fixed distribution and mutual orientation of aggregates with the formation of certain interfaces between them and the matrix. Thus, the prerequisites are laid for the formation of characteristic elements of the material structure at the macrolevel in the form of peculiarly ordered cells. A more rational approach is to model concrete as a set of cells with different parameters. This model reflects the variety of concrete at the matrix material - aggregates level. All processes of interaction of the matrix material and aggregates depend on the shape of the cells and the ratio of the values of cohesion of the soluble part and its adhesion to the surface of grains of crushed stone or gravel. Products based on concrete of industrial compositions include various sets of cells in terms of geometric and physical parameters. In this case, the shape and size of the cells are spontaneous, however, a directed change in the state of the surface of the aggregates contributes to an increase in the variety of cells in concrete of the same composition.

Due to the different orientations of the fillers and different ratios of the bonding forces on the interfacial surfaces in each cell, there is an individual manifestation of the properties of the matrix material as a discrete medium [4]. Depending on the shape of the cells and the state of the surface of the aggregates, a unique block structure of the matrix component is formed. This, in turn, affects the length of the internal surfaces of the matrix interface in individual cells, which affects the manifestation of volumetric deformations and the setting time of the material as a result of changes in the conditions of the hydration reactions.

The researches carried out on physical models of structural cells made it possible to determine that the length of the external boundaries and the area of the matrix material can change up to 39% and 35%.
respectively, depending on the geometric characteristics of the cells. Different conditions of adhesion of the matrix to the surface of the fillers in the cells cause a change in the size of the matrix component by 27-36% and a change in the density of the material up to 40%. The initial volumetric deformations of the matrix can differ in magnitude by 10-26%, in the kinetics by 7-44%. During transition from a cell of one form to cells of other forms, the setting time of the material changes by 20-150 minutes. The geometric parameters of the cells and the ratio of \( R_A \) and \( R_K \) values at the interfaces between the mortar component and aggregates are a powerful factor in controlling the structure formation of concrete at the micro level in the early periods. Thus, an increase in structural variety due to the directional obtaining of a complex set of initial structures at the level of binder particles by changing the parameters of the macrostructure provides the structural and functional potential in all material. This should affect the mechanical and deformative characteristics of concrete over time.

Table 3 shows the analysis of the coefficients obtained from the experimental results, the change in the level of properties of concrete on dense and porous aggregates in time was determined by the initial structures provided with different ratios of the binding forces at the interface of the cement matrix with the surface of the aggregates (\( R_A/R_K \)).

**Table 3. Analysis of changes in the properties of concretes with different surface conditions of aggregates during long-term hardening**

| R_A>R_K | Coefficients for assessing changes in concrete properties over time |
|---------|---------------------------------------------------------------|
|         | \( \Delta \rho_0 \) | \( \Delta P_0 \) | \( \Delta W \) | \( \Delta U \) | \( \Delta K \) | \( \Delta E \) | \( \Delta F \) |
| R_A>R_K | 1,01 | 0,66 | 0,69 | 1,12 | 1,05 | 1,23 | 1,03 |
| R_A<R_K | 0,96 | 0,70 | 0,74 | 1,12 | 1,04 | 1,17 | 1,13 |
| R_A=R_K | 0,95 | 0,55 | 0,64 | 1,17 | 1,07 | 1,32 | 1,20 |

| R_A>R_K | Concrete on crushed granite |
|---------|----------------------------|
| R_A>R_K | 0,96 | 1,0 | 0,87 | 1,0 | 1,01 | 0,97 | 1,10 |
| R_A<R_K | 0,96 | 0,82 | 0,78 | 1,03 | 1,03 | 1,0 | 1,15 |
| R_A=R_K | 0,98 | 0,89 | 0,71 | 1,0 | 1,10 | 1,01 | 1,22 |

| R_A>R_K | Concrete on expanded clay gravel |
|---------|----------------------------------|
| R_A>R_K | 0,96 | 1,0 | 0,87 | 1,0 | 1,01 | 0,97 | 1,10 |
| R_A<R_K | 0,96 | 0,82 | 0,78 | 1,03 | 1,03 | 1,0 | 1,15 |
| R_A=R_K | 0,98 | 0,89 | 0,71 | 1,0 | 1,10 | 1,01 | 1,22 |

Under the conditions of the experiment, concrete of composition I and expanded clay concrete of composition III were distinguished by a minimum decrease in average density indicators over time. However, all types of concrete with a structure organized at \( R_A = R_K \) had the highest values at 28 and 720 days of hardening. The volume of open capillary pores in concretes of the accepted compositions decreased over time. The maximum Po indicator decreased with the composition of III concrete, up to 44%, and with the composition II of expanded clay concrete, up to 18%. After long-term hardening during 720 days, the lowest values of water absorption were observed for concrete and expanded clay concrete, the structure of which was initiated under conditions of selective adhesion of the matrix to the surface of the aggregates. Thus, concretes with \( R_A = R_K \) are denser in the accepted time interval in comparison with concretes of other compositions. At the same time, the damage caused by technological cracks and internal interfaces increases to a greater extent for concretes, the structure of which was provided by a variety of ratios of values \( R_A \) and \( R_K \). Damage to heavy concrete increased during structure formation under conditions \( R_A = R_K \) by 21% and 29%, compared to structures organized under \( R_A<R_K \) and \( R_A>R_K \) conditions, respectively. For expanded clay concrete samples, the selectivity of matrix adhesion to the surface of aggregates leads to the formation of structures that provide a damage value that is less than the values of \( K_p \) of expanded clay concrete of composition I - by 39%, composition II - by 19%. The development of the main crack repeats the trajectory of operational cracks and internal interfaces, which accumulate in the material under the
action of various types of loads and arise as the growth of technological cracks present in the material for operation [5]. This assumes that the geometric parameters of cracks and fractures are determined by the number and relief of the banks of hereditary cracks and internal interfaces, oriented along the front of their movement. The value of the damage coefficient Kp1, through which it is possible to estimate the length of the fracture crack, with the structure initiated under the conditions of Rα and Rκ, were 7-18% less than the indicators Kp1 of the samples with the structure formed with perfect or absent adhesion of the matrix component to the surface of the aggregates.

Structural changes associated with the growth and transition of cracks to the rank of internal interfaces, along with the processes of hydration of relic binder grains, cause an improvement in the level of mechanical and deforming properties of concrete and expanded clay concrete over time. During selective adhesion of the matrix to the aggregates, structures are formed that provide the maximum increase in the elastic modulus of concrete under experimental conditions, up to 24%. The E values for expanded clay concrete are maintained at the original level during the long-term approval. After providing a variety of initial conditions for the interaction of the matrix material with aggregates, the compressive strength of concrete increases with time by 16%, the values of the compressive strength of expanded clay concrete increase by 18%. The greatest increase in strength under experimental conditions is observed in concrete and expanded clay concrete of composition III.

6. Conclusions
It can be concluded that increasing the diversity of the set of characteristic structural components by introducing rational ratios of mono- and polydisperse fillers and changing the ratio of adhesive-cohesive bond forces between the matrix component and aggregates allows maintaining the design level of mechanical properties and crack resistance of cement compositions and concretes over time. The change in the damage of the cement stone during long-term hardening in advantageous conditions indicates continuous structural transformations, the main role in which is played by technological cracks. An increase in various combinations of parameters of active elements of the structure determines the timeliness of their reactions to the changing dynamics of hydration processes of relic binder grains. This contributes to the maintenance of the specified quality indicators of the cement component during the long-term operation of building products and structures.

The use of a mixture of aggregates with different surface conditions led to the formation of structures in which the compressive strength of concrete increased to 18%, their modulus of elasticity increased to 24%, water absorption rates decreased to 29-36% with a decrease in open porosity to 44%, the length of cracks destruction decreased by 7-18% with an increase in technological damage by 19-39%. The change in damage and parameters of main cracks in time is determined by the organization of the structure of building composites at the early stages of structure formation. Thus, the initial structure under the influence of the accepted structure forming factors hereditarily determines the structural changes and the manifestation of the physical and technical properties of concrete and expanded clay concrete during prolonged hardening. It is recommended to take into account the results of the investigation carried out during prescribing the compositions of the cement binder and concrete mixtures in the manufacture of small-piece products and monolithic housing construction.

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