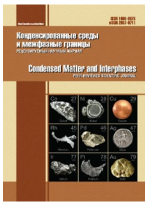

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Structural-phenomenological analysis of interrelation of microstructure indexes and properties of set cement systems

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Abstract

The study of the chemical and physical processes of solidification of polydisperse cement systems until now is based predominantly on empirical approaches. The phenomenological analysis of the interrelation of structure coefficients of set cement systems at the microlevel with their physical-mechanical properties was proposed as one of scientifically-practical approaches to control of physical and chemical processes of structure formation of controlled-quality concretes. The comparison of the quantity indicators of a microstructure of cement rock and its functional properties can be used for the estimation of structural modifications with a variation in the composition of cement systems. The aim of the study was to obtain quantitative data of the structural-phenomenological analysis of set cement systems for determination of interrelation of microstructure indexes with their physical-mechanical properties.

For the analysis of the structure of cement systems we used fractal geometry and the theory of passing (percolation)-based methods as well as modern modelling methods and scanning electronic and atomic-power microscopy. Fractal index D and micro-coarseness index S were used for a quantitative estimation of the microstructure of cement rock obtained without an additive and with an organomineral additive. These indexes were compared with the properties of cement rock determined during standard physical-mechanical trials.

The calculation of microstructure indicators and determination of the optimal content of the components of the organomineral additive allowed increasing the understanding of the fractal-cluster mechanism of self-organization of cement systems, taking into account the topology of particle distribution. The interrelation between the D and S indicators, compressive resistance and the density of the cement stone was shown. The higher fractal parameter and a relatively low level of micro-coarseness were indicators of the material with improved physical-mechanical properties. The monitoring of changes of D and S indicators can be used to control the structural formation processes of cement systems.

Keywords: Polydisperse cement systems, Fractal-cluster microstructures, Electron microscopy, Fractality, Micro-coarseness, Physical-mechanical properties

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1. Introduction

The phenomenological theory in the most general way is the formulation of regularities determining the relations between various observations of phenomena in accordance with the fundamental theory, but not following directly from this theory. In the natural sciences, the phenomenological analysis is an approach based on the correlation of various aspects and components of the phenomenon for the establishment of shapes and structures, modes and types of manifestation and functioning, in terms of relations with other phenomena and their mutual influence on each other. For construction materials science, further research aimed at improving methodological approaches for the analysis and assessment of the indexes of the structure of materials and the identification of their relationship with the physicochemical parameters that characterize the functional properties of the forming systems are important fundamental tasks. The development of ideas about the structure formation of the complex polydisperse heterogeneous systems, such as set cement stone, can be implemented comprehensively based on the system structural-phenomenological approach. The structural-phenomenological approach considers the interrelation between phenomena and their physical-mechanical properties, as well as a comprehensive microscopic analysis of quantitative indicators structures. This allows to establish the mechanisms of interaction between the components of cement systems and their influence on the physicochemical structural formation processes occurring at the microlevel.

The relevance of the implementation of the structural-phenomenological approach is associated with the widespread use of organomineral additives (OMA) for the production of cement concretes with improved physical and technical parameters based on multicomponent finely dispersed mixtures [1–18]. However, the control of the structural formation processes of cement systems have not yet been fully studied. The developed cement systems with OMA significantly differ from traditional concretes in terms of structural topology. As it is known, an inhomogeneous aggregated fractal-cluster structure is formed in the processes of hardening, as a result of chemical, physical intermolecular, interparticle and interfacial interaction of the components of cement systems in the solid phase. In the microvolume of set cement stone, the formed crystalline hydrate structure is a key element that determines the properties of the entire system [19–22]. At the same time, the formation of the microstructure is influenced by the properties of the surface of mineral components and the degree of dispersion, which manifests in the anisotropy of cement systems, their fractal-cluster heterogeneity [20–23].

The investigation of the structural formation processes of dispersed-disordered fractal-cluster systems at the microlevel can be successfully implemented based on the development of the provisions of fractal geometry, statistical physics, percolation theory with the involvement of modern numerical and computer models, physical and chemical research methods. One of the available methods for studying the microstructure of solids is electron and atomic force microscopy. In this case, the use of quantitative indicators of fractal dimension, micro-coarseness, which allow to assess the degree of homogeneity and ordering of complex fractal-cluster objects at the microlevel is efficient [24–29].

Thus, in our opinion, the development of a structural-phenomenological approach to the analysis of forming systems at the microlevel is one of the most important aspects of controlling the processes of structure formation and directed regulation of the properties of cement systems. New scientific knowledge in this direction allow to expand the understanding of the mechanism of interaction of components with various physical and chemical properties and their influence on the microstructure of cement concrete. This will become the basis for further improvement of the compositions and technology of concretes with improved functional characteristics, as well as the basis for the development of composites with unique properties.

The aim of the study was to obtain quantitative data based on the structural and phenomenological analysis of set cement systems for the identification of the interrelations of microstructure indicators with their physical-mechanical properties.

2. Experimental

The object of research was model and set cement systems - cement stones with and without OMA additives. The characteristics of the microstructure and physical-mechanical properties of the set cement system were investigated. For comparative analysis, two series of samples with the size of 2.0×2.0×2.0 cm were prepared: 1) control composition - cement stone without additives; 2) cement stone of a composite system with a complex OMA additive – "Portland cement + OMA". For these purposes, Portland cement class CEM I 42.5N with a specific surface area of 3000 cm^2/g, particle density 3.1 g/cm^3 was used. The mineralogical composition of Portland cement (wt. %) was as follows: $3CaO{\cdot}SiO_2$ – 68.98; $3CaO{\cdot}SiO_2$ – 10.87; $3CaO{\cdot}Al_2O_3$ – 8.77; $4CaO{\cdot}Al_2O_3{\cdot}Fe_2O_3$ – 11.38. Raw materials that showed high efficiency according to the results of preliminary studies [21–23] were used as components of the OMA. The chemical component of OMA was a plasticizing additive based on Melflux 2651F polycarboxylates, the dosage was 0.8% by weight of cement. The mineral component of the OMA was finely dispersed quartz sand with a fineness of 7000 cm^2/g, SiO_2 content 97%, particle density 2.6 g/cm^3, dosage 5, 10, 15, 20% by weight of cement.

The introduction of a pre-prepared complex OMA additive into the composition of the cement system was carried out in a dry form. Water content in the studied systems was selected based on constant rheological characteristic, estimated based on the cone flow diameter, which was 13–15 cm [21–23].

Technical tests of samples for compressive resistance (MPa) and average density (g/cm^3) was carried out according to GOST 30744-2001 "Cements. Test methods using polyfractional sand." After testing, samples from the destroyed cement stone were prepared by mechanical grinding for the analysis of their microstructural characteristics. The size of the studied samples was 5×5×2 mm.

An analytical assessment of the indexes of the microstructure of the cement system, including OMA with different dosages of finely dispersed quartz sand, was carried out by numerical and computer simulation methods based on the concepts of the percolation theory [22, 29]. For the studied systems, the concepts of flow along tangent and overlapping spheres, which were models of particles of the solid phase of the microfiller - finely dispersed quartz sand and cement were used. During the calculation, the quantitative ratio (by volume) was evaluated, which characterizes the proportion of microfiller, determining the processes of formation of the structure and properties of the cement system according to the formula:

$$V = \frac{N\pi d^3}{6}, \quad (1)$$

where V is volumetric content of spheres (models of particles of the solid phase of the microfiller); N is the number of spheres (models of particles of the solid phase of the microfiller); d is the diameter of the sphere (models of particles of the solid phase).

Also, during the analysis of the structural characteristics of the cement system, the ratio of microfiller particles to cement particles was calculated using the formula:

$$C = \frac{n_{mk}}{n_c}, \quad (2)$$

where n_{mk}, n_c are concentrations (countable) of particles of microfiller and cement, which were calculated using the formulas:

$$n_{mk} = \frac{6m_{mk}}{\pi d_{mk}^3 \rho_{mk}}, \quad (3)$$

$$n_c = \frac{6m_c}{\pi d_c^3 \rho_c}, \quad (4)$$

where m_{mk}, m_c is the proportion of microfiller and cement particles (by weight); d_{mk}, d_c is average microfiller and cement particle diameter; ρ_{mk}, ρ_c is the density of microfiller and cement particles.

Visualization and geometric modelling of particles in the studied two-component cement system was carried out in the Unity 3D computer software environment. The program created spheres, representing models of solid phase particles, the calculated values of the characteristics of the components were entered and the indexes of the structure were calculated and its model was visualized [22].

Analysis of the formed structure of the set cement system at the microlevel was carried out using two methods of physicochemical studies: scanning electron microscopy and atomic force microscopy. Micrographs of the surface of the structure with a resolution of 5 nm at a magnification of 2000 times were obtained by scanning microscopy method using a "Jeol jsm-6380LV" scanning electron microscope. The resulting electronic images were used for fractal analysis of the microstructure. Quantification of the fractal dimension index *D* was carried out by the islands cross-section method using Fractall.Stat 3.1 software package [24]. For the implementation of this method, a digital image of the surface of the microstructure, obtained by an electron microscope in gray scale, was converted into black and white, while adjusting the brightness and contrast in the Paint.NET program. The transformed image was loaded into the Fractall.Stat 3.1 computer program, in which the structure image was sequentially divided into fragments (islands) and the area of dense zones *A* and perimeter *P* for each selected fragment were calculated. The fractal dimension index was calculated as the slope of the dependence of the perimeter of dense zones *P* from their area *A*, plotted in double logarithmic coordinates. To ensure the correctness and reliability of the obtained data, the number of fragments of the structure image was more than 10. An example of a graphical representation of the results of calculating the fractal dimension is shown in Fig. 1.

Three-dimensional topology images were obtained by atomic force microscopy using a NanoEducator unit and the surface micro-coarseness index *S* (nm) was determined based on the maximum peak of the three-dimensional image (along the z axis). The size of the scanning area for obtaining the image was 9×9 μm.

3. Results and discussion

Physical-mechanical tests demonstrated that the set cement system with the OMA complex additive predictably had significantly higher physical-mechanical properties (Table 1) than the system without additives. The obtained results were a consequence of the active influence of the organic and mineral components of the OMA complex additive on the formation of the microstructure of the cement stone. The mechanism of action of the organic component "Melflux 2651F" based on polycarboxylates was due to the plastification and modification of the cement system, resulted from the effect of "steric" repulsion of watered particles of the solid phase during the adsorption of the surfactant [21, 23]. The action of the mineral OMA component - finely dispersed quartz sand was based on the modification of the structure of the cement system due to the presence of polar silanol groups capable of chemically participating in the process of hydration hardening on the amorphised surface of SiO_2 particles [23].

The physical factor of interparticle interaction also had an influence exhibited as the reshaping of the structure of the cement stone at the microlevel when OMA with a different ground quartz sand content was introduced [22]. According to the model representations of the percolation theory, the topological distribution of microfiller particles corresponding to the formation of a percolation "infinite" fractal cluster will determine the properties of the entire cement system (Fig. 2a) [22, 29]. It has been experimentally established that cement systems with a volume content of ground quartz sand *V* ≈ 0.1–0.15 had the highest strength and density indicators (see Table 1). Convergence of the obtained experimental values and the

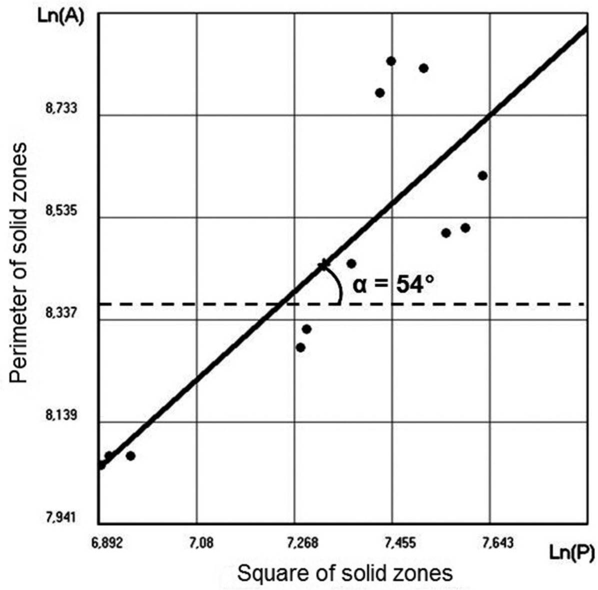


Fig. 1. Example of graphical representation of calculation results for determination of the fractal dimension using the Fractall.Stat 3.1 program [24]

Table 1. Influence of OMA with different content of ground quartz sand on the physical-mechanical properties of the set cement system

Cement system	Contents of system components			Physical-mechanical properties	
	mass content of ground sand, %	volume content of ground sand, V	the ratio of components in the system, C	compressive resistance, MPa	density, g/cm^3
with complex OMA additive	5	0.054	0.47	114	2.26
	10	0.104	0.99	135	2.28
	15	0.149	1.57	121	2.26
	20	0.189	2.23	103	2.25
without additives	–	–	–	57	2.07

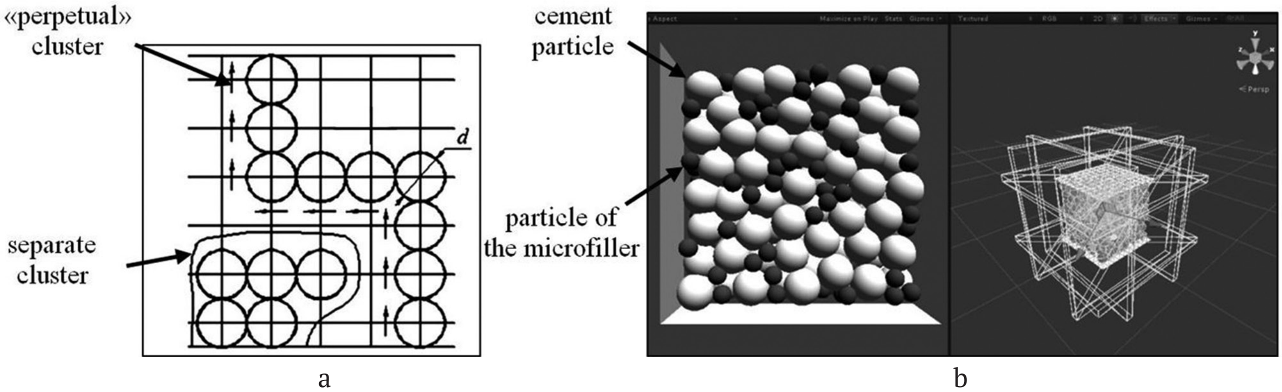
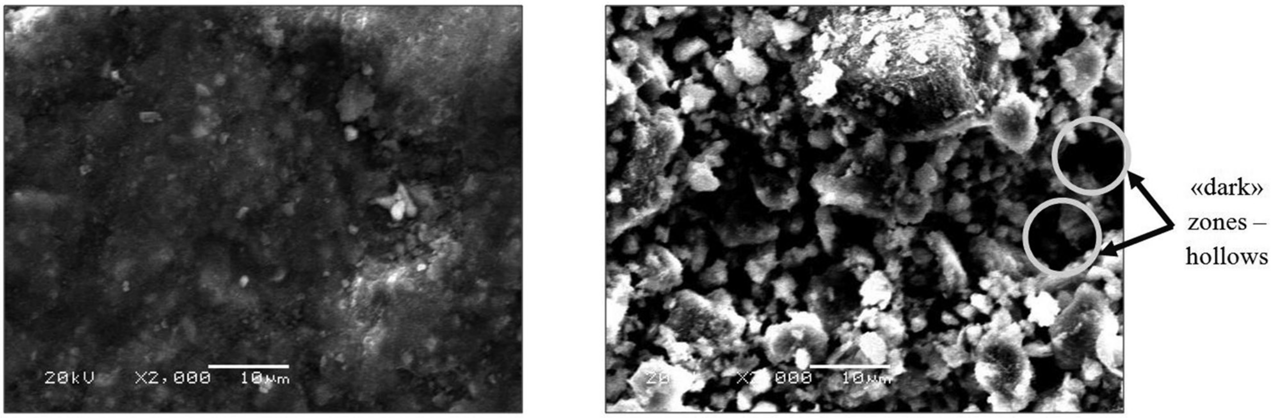


Fig. 2. Flat schematic model describing the flow of an "infinite" cluster (a) and geometric visualization of the model of the structure of the cement system with microfiller in the Unity 3D program (b) [22]

optimal indicators previously calculated for filled composites $V \approx 0.076–0.16$ confirmed the cluster mechanism of aggregation of particles of the solid phase and the possibility of the topological analysis of the structure using the percolation theory methods [22]. It was shown that the ratio of components in the system was $C \approx 1$ for a structure with a ground quartz sand content of 10% at $V \approx 0.1$ (see Table 1). A fragment of the visualization and geometric modelling of particles with the given structural characteristics of the cement system, carried out in the Unity 3D computer environment, is shown in Fig. 2b. Probably, under real conditions, the topological distribution of the aggregated microfiller provides its location in voids and between cement particles, which contributes to the compaction of the cement system. The obtained calculated values and the presented models characterize the optimal content of the OMA components for the formation of the microstructure and the improvement of the physical-mechanical properties of the filled cement system.

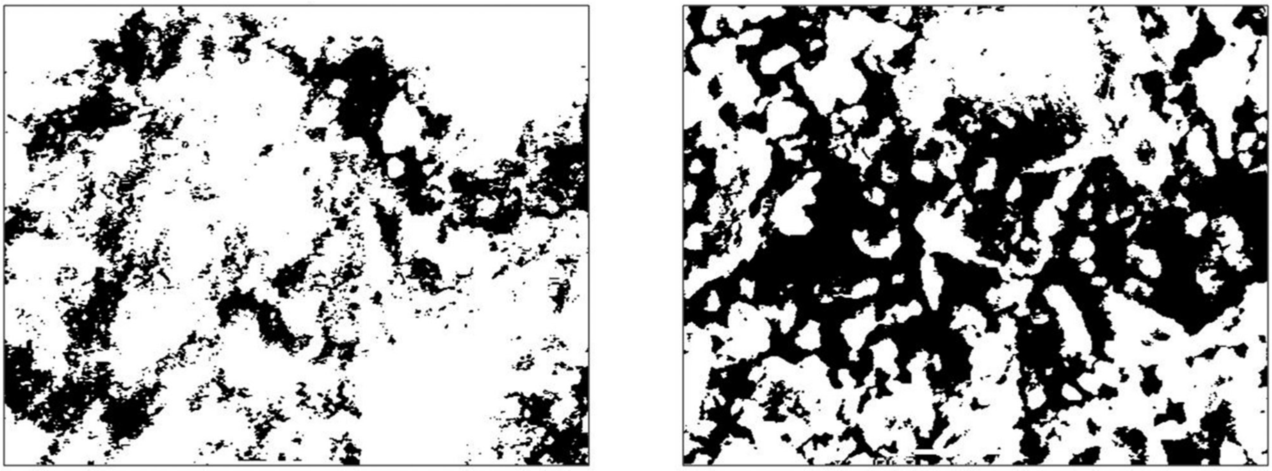
The analysis of images obtained using scanning electron microscope allowed to visually establish a qualitative change in the microstructure of the cement system formed during hydration hardening (Fig. 3). For the cement system, including the OMA complex additive, a distinctive feature was the formation of a more homogeneous and spatially ordered microstructure, including dense crystalline new growths (Fig. 3a). In turn, the microstructure of the set cement system without additives was characterized by a less uniform distribution of the solid phase with the presence of structural void elements - "dark" zones (Fig. 3b).

The images of the structure obtained using a scanning electron microscope were processed for quantitative evaluation by fractal analysis methods (Fig. 4). It was established that for a more homogeneous and ordered microstructure formed in a system with a complex OMA additive, the fractal index was higher $D = 1.85$ (Fig. 4a) in comparison with the index obtained for the structure without additives $D = 1.43$ (Fig. 4b). An increase in the fractal index quantitatively



a b

Fig. 3. Images of the microstructure of the set cement system obtained using a scanning electron microscope (×2000): a) cement system with the OMA additive; b) cement system without additives



a б

Fig. 4. Processed images of the microstructure of the set cement system: a) cement system with OMA additive (D = 1.85); b) cement system without additives (D = 1.43)

characterized the geometric restructuring of the formed microstructure and correlated with the strength indexes and density of the cement stone (Table 2). In particular, the higher the fractal index, and its value closer to D = 2, the more evenly dense elements (areas) are distributed in the microstructure space of the system and, respectively, the formed system of cement stone is denser and more durable.

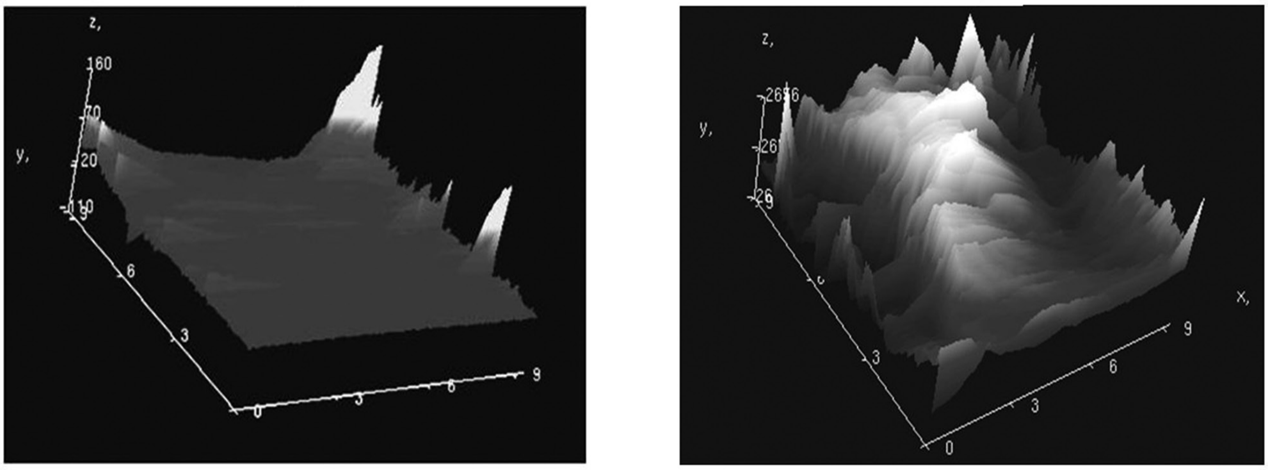
The interrelation of the micro-coarseness index S with physical-mechanical properties of systems was revealed by the analysis of the microstructure using an atomic force microscope,(Fig. 5, see Table. 2). It was established, that denser and more durable cement system with the OMA additive was characterized by a more uniform and ordered surface relief (Fig. 5a). Complex relief surfaces with a large number of protrusions and a higher value of micro-coarseness was characteristic for less homogeneous structure of the cement system (Fig. 5b).

4. Conclusions

The structural-phenomenological approach to the analysis and evaluation of the formed cement systems allowed to expand the understanding of the fractal-cluster mechanism of their structure formation. The relationship of microstructure indicators with the properties of cement systems was established by the comparison of quantitative data of modelling of the topology of particle distribution, and fractal geometry indicators of electronic images with microrelief indexes and physical-mechanical properties. The developed

Table 2. Indicators of the physical-mechanical properties and the microstructure of the set cement system

Cement system	physical-mechanical properties		Indicators of microstructure	
	compressive resistance, MPa	density, g/cm³	fractality, *D*	microcoarseness, *S*, nm
with complex OMA additive	135	2.28	1.85	160
without additives	57	2.07	1.43	2656



a b

Fig. 5. Images of the microstructure of the surface of the set cement system, obtained using an atomic force microscope (dimensions x, y – μm, z – nm): a) cement system with OMA additive (S = 160 nm); b) cement system without additives (S = 2656 nm)

methodology can be used for further study of the factors controlling the processes of structure formation and regulation of the properties of cement systems.

Author contributions

All authors made an equivalent contribution to the preparation of the publication.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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