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REGULATION OF THE INFLUENCE OF THE STRUCTURE OF INORGANIC BINDERS ON THEIR PROPERTIES

Об'єктом дослідження є взаємодія цементного тіста з кремнеземними заповнювачами та наповнювачами, які містять іони заліза. В нормальних умовах тверднення в контактній зоні утворюються в основному гідросилікати кальцію, CSH(B), CS₂H і гідрогранати. Одним з найбільш проблемних місць є взаємодія тверднучого цементного тіста з алюмосилікатними та залізосилікатними заповнювачами. При цьому в контактній зоні з'являється підвищена кількість гідросилікатів різної міри основності, гідроалюмінати, гідрогранати з різним співвідношенням SiO₂, Fe₂O₃, H₂O.

В ході дослідження вивчався контактний шар, що виникає в результаті взаємодії тверднучого цементного тіста з поверхнею заповнювачів суміші, представлений склеювальною речовиною. Ця речовина забезпечує в тому або іншому ступені зчеплення заповнювача в загальний моноліт. Цемент сприяє відновленню і продовженню звичайних процесів гідратації. Це пов'язано з тим, що запропонований спосіб є активним способом зміцнення мікродисперсного матеріалу за допомогою введення додаткових речовин в неорганічні вяжучі матеріали в зону контактування або в цементне тісто.

Завдяки цьому забезпечується можливість підвищення адгезії за рахунок акумуляції води, закупорки пор і тріщин, збільшення змочуваності і розчинності мінералів цементу, виникнення нових комплексних кристалічних утворень і тому подібне. Мікронаповнювачі сприяють зменшенню деформацій усадки і набрякання, характерних при твердненні високодисперсної клінкерної частини цементу. Вони також можуть підвищувати стійкість цементного каменю проти дії агресивних чинників, знижуючи витрату цементу і вартість бетону.

Таким чином, різними прийомами можна регулювати структуру отримуваних речовин на основі неорганічних в'яжучих, впливаючи на їх властивості у бажаних напрямах.

Ключові слова: цементне тісто, утворення гідросилікатів, взаємодія цементного каменю з залізистими силікатами.

1. Introduction

With prolonged exposure to water, the effect of strength restoration is observed, which is associated with cement hydration, leveling of the moisture state of concrete, repayment or smoothing of moisture stresses. The interaction of cement stone with magnesian silicates, for example, talc, glauconite, etc. In the binding layer, mainly hydrosilicates of different degree of basicity, magnesium hydrosilicates, alumina ferrite and ferrite hydrogarnets appear.

A large role in the formation of the contact zone is played by the processes of physical interaction of aggregates with cement stone. The strength of adhesion of cement stone to the limestone surface is many times higher than with quartz, granite and other silicate materials and rocks. If the Portland cement hydration products bordering the surface of the carbonate aggregate contain an increased CO_2 content, a new property appears in the surface layers of carbonate rocks. Less solubility in hydrochloric acid, because a greater amount of silica is found than in the usual volume of the same rock.

Microcrystalline neoformations of magnesium and calcium carbonate are concentrated in the contact zone, coalesce with each other and with the surface of micronutrients and granular aggregates. As a result, strong microcrystalline neoformations appear in this zone. These same processes take place in the pores and capillaries of the aggregate, into which water saturated with products of hydrolysis and hydration of clinker minerals penetrates. The contact zone is a layer of well-crystallized carbonized mass, spreading from the grains of aggregates to the cement stone, with mutual germination of crystalline neoformations and mutually reinforcing, with the disappearance of the interface between them.

Therefore, it is relevant to study the effect of microcrystalline neoformations of hydrocarboaluminates and hydrocarboferrites on stability under conditions of exposure to corrosive media.

2. The object of research and its technological audit

The object of research is the properties of concrete containing fillers, which ensure its durability.

Under the influence of silica fillers and fillers, which contain iron ions, the formation of the structure of cement stone neoformations is observed, surrounding and homogenizing the granular aggregate of concrete.

The nature and completeness of these processes depend on many factors, but primarily on:

- variety of mineral binders;
- variety and dispersion of the aggregate;
- crystal-chemical properties and structure of minerals;
- composition of the liquid medium in the mixture;
 presence in the system of extraneous, for example, surfactants:

- temperature;
- compaction measures;
- other external and technological factors.

Studies show that the structure of the contact layer is always homogeneous and finer than in the cement stone, and even more so in the aggregate. The reactivity of monoor polymineral inorganic binders with respect to aggregates is selective. In the areas bordering the filler in the contact zone in 20–40 μm , hydrate neoformations, crystallinity, density, strength and microhardness of which differ from the internal microstructure of the hardening binder are concentrated.

In the contact zone are different by the crystallochemical nature, the degree of weathering, and also by the orientation, aggregate minerals that interact with the binder and neoformations in this zone. In the interaction of Portland cement stone with the surface of carbonate aggregates the contact zone during the hardening under normal conditions is characterized by the presence of hydrocarboaluminates and hydrocarboferrites of binder and aggregate.

That is why the use of aluminate-alumoferrite cements is preferable when using calcareous aggregates. The most typical contact zone in a new compound is $3CaO\cdotAl_2O_3(CaO_3, MgCO_3)\cdot11H_2O$. This new formation is associated with the dissolution and recrystallization of crystals formed earlier.

One of the most problematic places is the interaction of Portland cement stone with the surface of carbonate aggregates. The contact zone for solidification under normal conditions is characterized by the presence of hydrocarboaluminates and hydrocarboferrites binder and aggregate, which are affected by numerous factors during hardening.

3. The aim and objectives of research

The aim of research is substantiation of the development of the structure in the binder test, where processes occur along the contact boundaries with the surface of the filling part and the factors affecting them.

To achieve this aim, the following tasks are identified: 1. To investigate the effect of filler on the frost resistance of concrete.

2. To investigate the influence of filler on the corrosion resistance of concrete.

4. Research of existing solutions of the problem

It is known that both medium quality concrete [1], like high-quality concrete [2] and special [3], consist of two systems: cement stone and aggregates. Therefore, the nature and completeness of the processes occurring in the volume of concrete at the contact boundary between cement stone and aggregate depend on many factors, and directly affect the durability of both concrete on cement [4] and slag-alkali [5].

The physicochemical interaction between the cement test and filler occurs at all stages of concrete preparation and is manifested in wetting, adsorption and chemisorption, the formation of adhesive bonds of various nature (van der Waals, hydrogen, electric, crystal chemistry and chemical) [6]. These factors affect the intensity of the processes of establishing contacts and the strength of the bonding of cement stone hydrates with aggregate while providing the main contribution to the formation of strength of cement materials [7].

Along with aggregate, cement stone and pores, the contact zone between cement stone and aggregate is treated as a separate structural element of cement materials, which is responsible for properties and operational capabilities. The formation of the contact zone occurs according to the general scheme of the structure formation of cement materials and determines the features of the structure that arises. For the contact zone is considered a microbe near the interface of the «cement stone – aggregate», which includes the surface layer of the aggregate, the layer of adjacent cement stone and the interface between them [7].

The nature of the contact zone is determined by the features of the hydration hardening of binders and the structure of the artificial stone around the surface of the aggregate with various crystal-chemical and structural features. However, the hydration hardening of binders depends on their composition, properties and complex external factors, including the water-hard ratio, the availability of various additives and the various technologies that are used.

The state of the contact zone (the bond strength of cement stone with aggregate grains), the composition and structure of neoformations on contact determine the main technical characteristics of concrete – strength, frost resistance, corrosion resistance [8]. Morphology, porosity, density and chemical composition are interrelated factors that influence the structure and strength of the material.

The physical and mechanical properties of the contact zone are due to the adhesion of the cement stone to the aggregate grain. The structure and interrelation of the elements of the microstructure of the cement stone, the size and nature of the pores, the relationship between the phase components depend on the composition of the hydration products, which are determined by the chemical-mineralogical features of the initial binders and the amount of water necessary for hydration, provided that the technology of preparation of the concrete mix, time and curing regime.

The properties of the contact zone, including the interface between the aggregate and the cement stone, as well as the contact layers of the cement stone and aggregate, are further conditioned by factors such as [9]:

 chemical and mineralogical composition and physical and mechanical properties of the aggregate;

- methods of preparation;
- laving and consolidating concrete mix;
- conditions of hardening.

Based on the evaluation of the structural features and the phase composition of the contact zone before and after the operation of concrete, it is possible to determine its ability to resist the effects of various factors, to evaluate the mechanisms that determine the stability of the materials as a whole. The participation of the filler in the formation of the contact zone is manifested in the effect of crystal-chemical features and the specific surface of minerals in its composition on the processes of structure formation during the binder hydration.

Among the main directions of the solution of the problem of increasing the durability of concrete, for example, the use of binding substances of high resistance to aggressive media can be singled out [3, 10]. With regard to studies of the effect of the properties of the contact zone between the aggregate and the cement stone on the indices of the durability of concrete, they are limited:

- investigations of the influence of the mineralogical composition of cement [11], the properties of bond strength with an amorphous silica filler [12] and carbonate rocks [13];

- interaction of aggregates for concrete with water [14]. In particular, the paper [14] is devoted to the determination of the interaction of aggregates with water and the relationship between moisture and filler material is considered.

The moisture contained in the concrete is classified according to the size of the energy of its connection with its constituents. In general, three large groups of moisture bond are distinguished from the material: chemical, physicochemical, physicomechanical. Chemical bond of water in concrete as a result of chemical reactions of cement hydration is carried out in precise quantitative stoichiometric ratios. Physicochemical (adsorption) bound water is present on the hydrate shells of crystals. The reason for this connection is the presence of force fields of the atomic-molecular nature. The monomolecular layer of water is most strongly bound. The following layers are contained with weaken force.

Physicochemical (adsorption) bound water is present on the hydrate shells of crystals. The reason for this connection is the presence of force fields of the atomic-molecular nature. The monomolecular layer of water is most strongly bound. The following layers are contained with weaken force. Such polymolecular layer of adsorption water is about a few hundredths of the diameter of water molecules. Adsorption-bound water has certain properties that distinguish it from ordinary water. So, due to the increased density, this water has the properties of an elastic body and performs a wedging action. The adsorption bond of water in concrete refers to the number of medium-intensity bonds that can be destroyed by desorption and evaporation, but this requires time to be determined [3].

Minerals of cement stone (hydrosilicates, hydroaluminates and hydroferrites) can persist in the solid phase only when the concentration of $Ca(OH)_2$ in water that is in concrete remains above a certain equilibrium concentration for a given mineral.

In the initial stage of the action of fresh water (with low time rigidity) on concrete, $Ca(OH)_2$ concentration remains equal to the concentration of saturated solution (at a temperature of 20 °C – 1.18 g/l, calculated as CaO) as a result of the dissolution of lime, which is in the solid phase. The mass of free lime in Portland cement stones is 3 months old at 15 % of the cement mass. After dissolution and removal of all free lime, its concentration in the limiting volume of concrete resumes as a result of the transition to the liquid phase of calcium, chemically bound in the minerals of the cement stone [5, 6].

According to the authors of [6, 13], it should be noted that when Portland cement is hardened, the liquid phase is saturated with Ca^{+2} ions, increasing the pH of the medium. This contributes to the formation of aluminuimoxygenic and silicone-oxygenic and also ferrum-oxygen aquacomplexes [3], which are the main structure-forming motifs of low-basic hydrosilicates, which have a higher resistance to aggressive media. Moreover, the lower the CaO concentration in the cement solution hardens, the faster the hydrolysis and hydration of C_3S and the less basicity of the formed hydrosilicates.

One of the most well-known minerals formed during the hardening of Portland cement compositions containing a significant amount of chemically bound water is calcium hydrosulphoaluminates, of which the most known are two hydrosulphoaluminates, 3CaO·Al₂O₃·3CaSO₄·31H₂O and 3CaO·Al₂O₃·CaSO₄·12H₂O, as well as 3CaO·Fe₂O₃× \times CaSO₄·12H₂O. These minerals are stable in aqueous solutions of Na₂SO₄, NaCl, CaCl₂, that is, they determine the corrosion resistance of the system in these aggressive media. And also bind a significant amount of water, increasing the density of cement stone and the value of its initial modulus of elasticity. Consequently, ensuring the formation of a dispersed system in the hydration products solidifies, a significant amount of minerals such as hydrosulphoaluminate and calcium hydrosulphoferrite will increase the density of the cement stone and, as a result, strength and corrosion resistance. And also a decrease in its deformability. However, the amount of these minerals in the known disperse systems based on Portland cement is limited, and their interaction with the concrete filler has not been sufficiently studied, which necessitated the carrying out of this work.

5. Methods of research

The following scientific methods are used in the study: – method for determining frost resistance of concrete in accordance with the requirements of state standards of Ukraine;

- method for determining the resistance of concrete to aggressive media.

6. Research results

When hardening concrete samples in air-dry conditions, an increase in strength is observed, the optimum of which is reached on the 90th day of hardening, but for 180 days there is a decrease in prismatic strength due to the processes of recrystallization, the flowing hydrate neoformations. This leads to a change in the elastic-plastic state of the artificial stone.

The samples of normal hardening concrete are characterized by intensive growth of the elastic modulus in the first 28 days, after 90 days – the elastic modulus is stabilized and then changes insignificantly. Steaming at a lower temperature isotherm (338 ± 5) K and subsequent preservation under normal conditions makes it possible to obtain an artificial stone, which modulus of elasticity at the age of 28 days is comparable to the value of the modulus of elasticity of concrete when hardened in natural conditions. Moreover, comparing the modulus of elasticity of Portland cement and slag-alkali concretes of the same class, let's note the lower deformability of the latter.

In the process of further formation of the concrete structure due to a uniform distribution of internal stresses, a corresponding growth of the prismatic strength of concrete at the age of 270 days is observed. In contrast to the foregoing, cube strength is characterized by a constant increase in the time interval under study.

Kinetic curves of changes in the modulus of elasticity and prism strength of concrete in air-dry conditions similar. Moreover, the decrease in these indicators at the age of 180 days is typical for Portland cement concrete and slag-alkali concrete, solid under similar conditions.

This is apparently due to the reasons that determine the elastic-plastic state of concrete, in particular the concentration of stresses in individual zones of concrete and the appearance of shrinkage cracks that reduce the elastic properties of the material due to uneven drying.

Frost resistance of concrete is its property, which ensures its durability. In this group of experiments, the frost resistance of concrete as a composite material obtained as a result of the hardening of the system «hydraulic binderiron-containing filler-filler» was investigated, depending on its composition and the content of the iron-containing aggregate in it.

Under the experimental conditions, the presence of an iron-containing aggregate in the system leads to an increase in frost resistance of concrete by 20...30 % (Table 1).

Table 1

Table 2

Frost resistance of concrete obtained on the basis of the system «Portland cement – iron-containing filler – filler»

	Frost				
Portland cement	Water	Sand	Crushed Iron-containing stone filler		resistance, cycles
439	175	464	1280	-	110
351	175	464	1280	88	135
350	210	464	1280	-	75
280	210	464	1280	70	95

In the next group of experiments, the stability of concrete under conditions of exposure to corrosive media was investigated depending on its composition and the type of corrosive medium.

During the research, it was determined: acid resistance, sulphate resistance, resistance to leaching, depending on the amount of iron-containing filler in concrete 1:1.8:4.3 on Portland cement.

The determination of acid resistance was carried out by the bending test of $10 \times 10 \times 40$ cm beam samples stored in an aqueous acid solution for 6 months.

Under the conditions of carrying out the experiment, introduction of iron-containing filler into the system increases the stability of concrete by 15...20 % when it is hardened in a medium of 0.01 n HCl solution (Table 2).

Acid resistance of concrete obtained on the basis of the system	
«Portland cement – iron-containing filler – filler» in 0.01 n HCl solution	

Water-cement	Concrete strength, MPa for iron-containing filler, $\%$							
ratio	0	20	30	40				
0.40	10.0	8.4	7.5	6.2				
0.45	9.5	8.0	7.1	5.7				
0.50	8.6	7.9	7.2	5.9				

The sulfate resistance of concrete is determined by the action of solutions of different composition:

- 1 drinking water (control);
- 2 5 % solution of Na₂SO₄;
- 3 1 % solution of MgSO₄;

4-5 % solution of equal mixture of Na₂SO₄ and MgSO₄.

After 6 months of storage in these solutions, the concrete sample beams, which had a size of $10 \times 10 \times 40$ cm, made of concrete 1:1.8:4.3 on Portland cement have been tested for bending. Under the experimental conditions, the introduction of iron-containing filler into the system increases the sulfate resistance of the resulting concrete (Table 3).

Table 3

Sulfate resistance of concrete obtained on the basis of the system «Portland cement – iron-containing filler – filler»

Water-	Concrete strength, MPa for iron-containing filler, % for media											
cement	0			20		30			40			
ratio	2	3	4	2	3	4	2	3	4	2	3	4
0.40	0.99	1.2	1.1	0.96	1.08	0.9	0.94	1.05	0.9	0.99	1.0	0.9
0.45	0.92	1.0	0.9	0.8	1.0	0.9	0.91	1.02	0.98	0.92	1.0	0.9
0.50	0.90	0.9	0.8	0.88	1.0	0.9	0.90	0.97	0.95	0.97	1.0	0.9

Concrete based on the system «Portland cement – iron-containing filler – filler» prepared from the composition 1:1.8:4.3 on the basis of Portland cement and after storage for 6 months in flowing distilled water shows such stability results by the bending test of beam samples measuring $10 \times 10 \times 40$ cm (Table 4). It is determined that this concrete has a sufficiently high resistance to corrosion of the 1st type.

Table 4

Stability of concrete obtained on the basis of the system «Portland cement – iron-containing filler – filler» to leaching

Composition of th	Concrete strength, MPa, months			
Portland cement	Iron-containing filler	0.033	3	6
100	0	8.0	7.8	7.1
90	10	9.0	9.0	7.3
80	20	10.6	9.7	8.8
60	40	8.9	7.8	6.6

Thus, the conducted studies show that the concrete obtained on the basis of the «Portland cement – ironcontaining filler – filler» system has an increased corrosion resistance in comparison with concrete obtained for a non-additive Portland cement. The increased corrosion resistance of the resulting concrete can be explained by the formation of iron-based minerals with a high content of bound water (Fig. 1), which leads to a decrease in its permeability to external media.

The amount and the rate of formation of calcium hydrogencarbonate increases with the degree of grinding of the carbonate aggregate or filler.

In the interaction of cement paste with silica fillers sandstone, quartz, tuff, trefoil, etc. under normal conditions of hardening in the contact zone, mainly hydrosilicates of calcium, CSH(B), CS₂H and hydrogarnets are formed. The interaction of the hardening cement test with aluminosilicate aggregates of orthoclase, albite and other types. There is an increased amount of hydrosilicates of different degree of basicity, hydroaluminates, hydrogarnets with a different ratio of SiO₂, Fe₂O₃, H₂O.

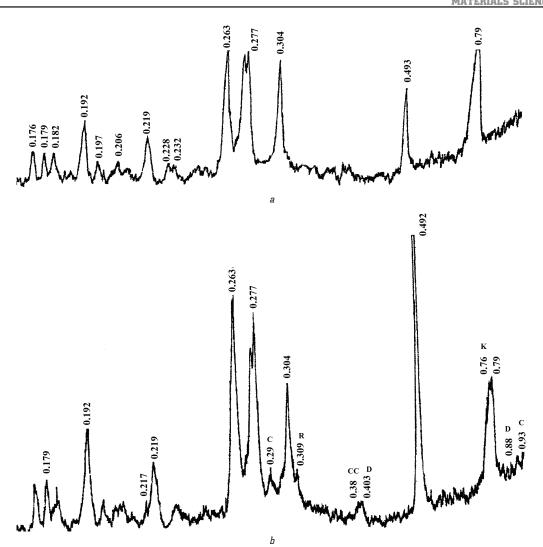


Fig. 1. X-ray patterns of cement stone obtained as a result of hardening: *a* – Kryvyi Rig Portland cement without additives; *b* – disperse system «Portland cement – iron-containing filler – filler», containing 80 % of Portland cement and 20 % of filler. R – riebeckite, C – chabazite, CC – cancrinite, D – desmin, K – 3CaO·Fe₂O₃(Al₂O₃)·CaCO₃·12H₂O

In the conditions of steaming and autoclaving, the basicity of calcium hydrosilicates decreases, and the hydrogarnets are enriched with silica and iron oxide with a change in the phase relationship. The interaction of cement stone with magnesian silicates, for example, talc, glauconite, etc., mainly hydrosilicates of different degree of basicity, magnesium hydrosilicates, aluminum-hydrate garnets and ferrites appear in the binding layer. When cement paste is interacted with aggregate from igneous rocks - granite, svenite, diabase, gabbro, diorite, basalt, solidification of calcium hydrosilicates of various degrees of basicity occurs in the contact zone under normal conditions. The strength of adhesion in this zone is also influenced by structural and texture features of rocks used in the production of aggregates. In order to increase the adhesion to the system, mineralizers are introduced in the form of calcium chloride, alumina sulphate, liquid glass or filler. The contact layer resulting from the interaction of the hardening cement paste with the surface provides, to varying degrees, the adhesion of the aggregate to the overall monolith. Like other adhesives, its adhesive properties and ability to create a monolith depend on the phase composition and structure of the cement stone. Strength of adhesion, as a rule, increases with the increase in the contact zone of calcium hydrosilicates such as CSH(B), followed by a transition to a stable form of tobermorite $C_5S_6H_5$, ferrite and aluminoferrite hydrogarnets, magnesium hydrosilicates. The adhesion on the edge of the contacting components also increases with an increase in the dispersion degree of the contact zone, the growth of the crystal lattices of the cement stone, and the rock-forming minerals.

In the region of low temperatures (below 0 °C), energy and kinetic deviations are observed during the hardening of the cement paste. The processes of interaction of clinker minerals with water are slowing down, but not at sufficiently low negative temperatures, depends on a number of reasons, especially from the stage of hydration of the cement to freezing, for example to -10 °C and lower. This is facilitated by exothermic reactions that occur when diffusion penetration of moisture in the fresh surface of cement particles through the helium layers, as well as the supercooled state of the liquid phase in the presence of mineral ions, contributes to the law of phase equilibrium shift. Crystalline hydrates, which are gradually formed in a supercooled liquid – ettringite and tobermorite – are stable at lower temperatures than at normal temperatures and have a relatively higher dispersity than with conventional hydration. This has a positive effect on the strength of cement stone. Freezing stops the processes of cement hydration.

However, cement contributes to the restoration and extension of conventional hydration processes. An active way to strengthen the micro-dispersed material is the introduction of additional substances into inorganic binders, into the contact zone or into the cement paste. These substances increase adhesion by accumulating water, blocking pores and cracks, increasing the wettability and solubility of cement minerals, the emergence of new complex crystalline formations, and the like. In addition to surfactants having the above, in many cases, powder additives of micronutrients, for example Portland cement, are very useful. In optimal amounts, sometimes up to 15-30 % and higher by weight of cement, can favorably reflect on the density without significantly reducing the strength. Both due to the mechanical filling of the micropores, and under the influence of the emergence of an additional number of new complex cementing neoformations, for example, $3CaO\cdot Al_2O_3\cdot 12H_2O,\ 3CaO\cdot Fe_2O_3(Al_2O_3)\cdot CaCO_3\cdot 12H_2O$ or 3CaO·Al₂O₃·MgCO₃·12H₂O and other.

7. SWOT analysis of research results

Strengths. Microfillers contribute to reducing deformation shrinkage and swelling, characteristic of hardening the highly dispersed clinker part of cement, can increase the resistance of the cement stone to the action of aggressive factors that reduce cement consumption and the cost of concrete.

Weaknesses. In connection with the fact that the mechanism of the positive effect of repeated vibration is not yet clear, after 1.5-2 g from the moment of filling the cement with water occurs:

- mechanical destruction of shells of neoformations;
- new surfaces of cement grains are exposed;
- new masses are attracted in the process of hydration;
- increases the amount of coherent water neoformations;
- cement stone is strengthened and adhesion on the boundaries of its contact with the aggregate.

Opportunities. It is necessary to investigate the mechanism of the positive effect of repeated vibration during the formation of concrete products.

Threats. Additional costs for materials and energy are possible. For example, the adhesion strength, as a rule, increases with the increase in the contact zone of calcium hydrosilicates of the CSH(B) type, followed by a transition to a stable form of the tobermorite $C_5S_6H_5$ type, ferrite and aluminoferrite hydrogarnets, magnesium hydrosilicates. If in the products of Portland cement hydration, which border on the surface of the carbonate aggregate, there is an increased content of carbon dioxide CO₂, then a new property appears in the surface layers of carbonate rocks. Namely, there is less solubility in hydrochloric acid, since a greater amount of silica is found than in the usual volume of the same rock.

8. Conclusions

1. The effect of iron-containing filler on the frost resistance of concrete is investigated. It is established that the introduction of iron-containing filler in concrete leads to an increase in its frost resistance by 15...40 %.

2. The effect of iron-containing filler on the corrosion resistance of concrete is studied. It is established that the introduction of iron-containing filler in concrete leads to an increase in its corrosion resistance by 25...40 %.

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