Concrete structure with complex additives

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Abstract. The article presents the results of a study of the structure of concrete with complex chemical additives, which are used for water-resistance of concrete. Studies have shown that an increase in the physical and mechanical properties of concrete, including water resistance and frost resistance, is achieved by controlling the process of structure formation both at the design stages of the composition and preparation of the concrete mixture and at all stages of product manufacturing. The objective of this research is to study the structural indicators (W₀, λ, α) of concrete with increased water resistance, depending on the cement consumption with an increase in cement consumption from 350 kg/m³ to 450 kg/m³ in concretes with a complex additive C-3 + KE 119-215 and without chemical additives, a decrease in the kinetics of water absorption is observed. The results of the study showed that concrete with complex chemical additives has uniformly distributed small closed pores, while the average pore size and water absorption decrease. The introduction of the KTP additive into the composition of the cement stone promotes air entrainment, as evidenced by the pores in the photo. No neoplasms are observed in the pores, crystals of Ca(OH) on calcium hydrosulfoaluminate (ettringite) are visible. The study of the cement stone with the addition of C-3 showed that the formation of calcium carbonate CaCO₃ is observed, and in the samples with additives, KTP - the formation of calcium carbonate CaCO₃ of the hydrosulfoferrite C₃FGH32 (analogous to ettringite).

1. Introduction
In our country, great attention is paid to hydromелиorative construction, since large area needs irrigation. The main building material for irrigation and drainage construction is concrete.

The following technical requirements are imposed on concretes for irrigation and drainage construction: grade for waterproofing W 6, grade for frost resistance F 300, compressive strength B 25, and durability must be at least 20 years.

However, in practice, due to various production reasons, concrete does not always meet the specified requirements, which negatively affects the durability of products used in irrigation and drainage construction. For example, studies of destroyed chutes have shown [1] that they are mainly destroyed in the bottom part and at a height of 1/3 of the cross-section, i.e. in the area of the operational water level.

The reason for the early destruction in the bottom of the tray is the loosening of the concrete from the action of alternate freezing and thawing of water residues. This is aggravated by the occurrence of tensile stresses from the tray's own weight.

It is known that an increase in the physical and mechanical properties of concrete, including water tightness and frost resistance, is achieved by controlling the process of structure formation both
at the stages of designing the composition and preparing the concrete mixture, and at all stages of product manufacturing [2-4].

In production, the specified physical and mechanical properties of concrete are most often achieved due to a significant increase in comparison with the rates of cement consumption. However, this direction is not optimal in terms of technical and economic indicators. As studies show, the water resistance and frost resistance of concrete can be increased through the use of complex chemical additives [5-11].

One of the determining factors for the water resistance of concrete is its porosity, with an increase in which the water resistance decreases. In the work of FM Ivanov [3] it is noted that the characteristics of porosity are important for assessing water resistance, gas permeability and frost resistance, as well as, to a large extent, the chemical resistance of concrete.

On the issue of the relationship between porosity and water resistance, there are a number of works [1, 4, 10, 12]. So MK Sharovar in his work [1] provides data characterizing the relationship between water resistance and the average size of open capillary pores. And in work [13-21] there is even a table that makes it possible to predict its water resistance by water absorption of concrete and other parameters of concrete.

As noted above, the total porosity may not fully characterize the water impermeability of the concrete. Pores in concrete in shape, size and location in relation to the direction of movement to the filtering water flows can be different. Some concretes, despite their high porosity, have increased water resistance. And it happens the other way around. But despite this, there is a certain relationship between porosity and water resistance, and this issue in concrete technology remains insufficiently studied.

One of their characteristics of concrete water resistance can be its water absorption $(W_o)$, since it is determined by the volume of open capillary pores $(P_o)$. An important structural parameter that determines the water resistance of concrete is the indicator of the average size of open capillary pores $(\lambda)$ and the indicator of uniformity of the sizes of open capillary pores $(\alpha)$.

The objective of this research is to study the structural indicators $(\lambda, \alpha, W_m)$ of concrete with increased water resistance, depending on the cement consumption. As you know, one of the ways (but not rational) to increase the water resistance of concrete is to increase the consumption of cement. The reasons for the increase in the water resistance of concrete in this case are explained by the formation of homogeneous pores, a decrease in the size of through pores, etc.

2. Method
In this work, to assess the structural characteristics of concrete, parameters such as the average size of open capillary pores $(\lambda)$, the uniformity of the size of open capillary pores $(\alpha)$ and water absorption $(W_m)$ were determined.

To study the structure of concrete, depending on the consumption of cement, samples were made - cubes with dimensions of 70x70x70 mm. The study was carried out 10 days after heat treatment according to the $2 + 3 + 6 + 3$ h regime at a temperature of 80 °C. The samples were made with a cement consumption of 350, 400 m 450 kg / m$^3$ with a complex additive $S-3 + KE 119-215$ and without additives. The mobility of the concrete mixture with the complex additive $C-3 + KE 119-215$ and without additives was $OK = 2 - 4$ cm. In all compositions, the sand content in the aggregate mixture was constant $r = P / P + U = 0.38 = \text{const}$.

Determination of structural indicators $(\lambda, \alpha, W_m)$ was carried out in accordance with GOST 12730.4-78 “Concrete, methods for determining porosity indicators” (Method M.I.Brusser).

The work also investigated the structure of the cement stone with a complex additive $S-3 + KE 119-215$ and without additives with a scanning electron microscope.

To study the structure of the cement stone, samples of cement paste were made with a water-cement ratio (W/C) of 0.30 and a melt cone equal to 109 mm. The introduction of super plasticizer $C-3$ into the composition of the cement paste greatly increases the mobility. Therefore, when choosing the
water-cement ratio (W/C) of cement pastes, taking into account the super plasticizer C-3, we proceeded from the fact that the melt of the cone of all compositions was close to each other.

To study the structure, samples were made of cement stone with a size of 40x40x40 mm. The samples were heat treated according to the 2 + 3 + 6 + 3 h regime at a temperature of 800°C in a laboratory steaming chamber with an automatic control mode.

The samples were examined 10 days after heat treatment. To study the structure of the cement stone using a scanning electron microscope, pieces of approximately 2x2x2 mm were selected. At the same time, some of the samples were tested for compressive strength (Table 1). To study the structure of the cement stone, we used a JSM-255 electron scanning microscope from the Japanese company JEO (“Jeol”).

3. Results and discussion

The research results show (Figure 1) that with an increase in cement consumption from 350 kg / m³ to 450 kg/m³, the water absorption of concrete with a complex additive decreases from 4.5 to 3.7%, and without additives - from 5.25 to 4.9%. And the indicator of the average size of open capillary pores (λ) for concretes with a complex additive S-3 + KE 119-215 decreases with an increase in consumption within the same limits from 1.12 to 0.9, for concretes without additives - from 1.16 to 1.06. The parameter of the indicator of uniformity of the size of open capillary pores (α) of concretes with a complex additive С-3 + КЭ 119-215 and without additives increases with an increase in cement consumption, i.e. its pore space becomes more uniform.

In addition, the study investigated the kinetics of water absorption of concrete with increased water resistance, depending on the consumption of cement.

![Figure 1](image)

**Figure 1.** Porosity of concrete with increased water resistance, depending on the consumption of cement:
1 - water absorption (Wm) in% by weight; 2 - indicator of the average size of open capillary pores (λ); 3 - indicator of uniformity of the size of open capillary pores (α)
Figure 2. Kinetics of water absorption of concrete with increased water resistance depending on the cement consumption:
1,3 and 5 – samples without chemical additives (control);
2,4 and 6 – samples with a complex additive $S_3 + KE_{119-215}$ with a dosage of 0.5 + 0.1% by weight of cement;
1 and 2 – cement consumption 350 kg/m$^3$;
3 and 4 – the same 400 kg/m$^3$;
5 and 6 – the same 450 kg/m$^3$.

As can be seen from the research results (Figure 1), with an increase in cement consumption from 350 kg/m$^3$ to 450 kg/m$^3$ for concretes with a complex additive $С_3 + КЭ_{119-215}$ and without chemical additives, a decrease in the kinetics of water absorption is observed. At the same time, the highest degree of water absorption is achieved by concrete without add-ons with a cement consumption of 350 kg/m$^3$, and the lowest - by concrete with a complex additive $S_3 + KE_{119-215}$ with a cement consumption of 450 kg/m$^3$.

At the beginning of the work, it was noted that the structure of the cement stone is being investigated with a complex additive $C_3 + KE_{119-215}$ and without additives with a scanning electron microscope. As can be seen from the test results (Table 1), the introduction of silicon organic polymer $KE_{119-215}$ into the composition of the cement stone provides an increase in strength by 35% compared to compositions without additives, and even more compared to compositions with KTP, i.e. by 36%. The highest results in terms of compressive strength are shown by concretes with a complex additive $S.Z + KE_{119-215}$.

| Additive type | Dosage in % by weight of cement | W/C | Spread of cement paste in mm | Compressive strength of cement stone in MPa |
|---------------|---------------------------------|-----|------------------------------|---------------------------------------------|
| -             | -                               | 0.30| 109                          | 63/100                                      |
| 0-3           | 0.5                             | 0.38| 130                          | 83/132                                      |
| KTP           | 0.01                            | 0.30| 316                          | 63/97                                       |
| CE 119-215    | 0.1                             | 0.30| 110                          | 85/135                                      |
| 0-3+KTP       | 0.5+0.01                        | 0.28| 335                          | 73/116                                      |
| 0-3+CE 119-215| 0.5+0.1                        | 0.28| 120                          | 90/143                                      |
To study the structure of the cement stone, we used a JSM-255 electron scanning microscope from the Japanese company JEO (“Jeol”).

As seen from the micrographs (Figure 3.a), in the structure of the cement stone, uneven distribution of very large small pores is formed. In (Figure 3.b) you can see crystals of Ca(OH)$_2$ and calcium hydrosulfoaluminate. In addition, chips of clinker grains are visible.

![Figure 3](image1.jpg)  
**Figure 3.** Microstructure of cement stone without additive  
a) zoom x45; b) zoom x1500.

The introduction of superplasticizer S-3 into the composition of the cement stone (Figure 4.a) contributes to the uniform distribution of small pores to the volume of the material. Crystals of Ca(OH) and clinker grains are observed (Figure 4.b) no neoplasms are observed in the pores. (Figure 4. a) pores are clearly visible, which are formed as a result of air entrainment.

![Figure 4](image2.jpg)  
**Figure 4.** Microstructure of cement stone with superplasticizer S-3.  
a) zoom x45; b) zoom x1000.

The introduction of the KTP additive into the composition of the cement stone (Figure 5.a) promotes air entrainment, as evidenced by the pores in the photo. No neoplasms are observed in the pores (Figure 5 b); crystals of Ca(OH) on calcium hydrosulfoaluminate (ettringite) are visible.
Figure 5. Microstructure of cement stone with the addition of KTP.
   a) zoom x45; b) zoom x1000.

Figure 6. Microstructure of cement stone with the addition of CE 119-215.
   a) zoom x1000; b) zoom x3000.

The most interesting picture is observed when an organosilicon polymer such as olegoethoxy-2-ethylhexoxysiloxane is introduced into the composition of the cement stone (Figure 6a) and (Figure 6b). As seen from photomicrographs, in addition to crystals of Ca(OH)$_2$ and clinker grains, crystals of new formations are observed in the pores (possibly crystals of polyorganocalcium siloxanes). Y.A Savvina in her work [12] shows the possibility of the formation of such crystals in the interaction between cements and the addition of alkoxysiloxanes.

With the introduction of complex additives S-Z + KTP into the concrete composition, uniformly distributed pores formed as a result of air entrainment are observed in the structure of the cement stone (Figure 7 a).
Figure 7. Microstructure of cement stone with a complex additive S-3 + KTP. 
   a) zoom x45; b) zoom x1500.

The introduction of the complex additive S-Z + KE 119-215 into the composition of the cement stone is formed in the structure (Figure 8a) with more uniform and smaller pore sizes in comparison with the samples with the complex additive S-Z + KTP. In addition, one can see (Figure 8b.) Ca(OH)$_2$ crystals and clinker grains.

Thus, the introduction of superplasticizer S-3, an air-entraining additive KTP and an organosilicon polymer KE 119-215 into the composition of the cement stone contributes to the improvement of its structure. Especially their complex combination (i.e. S-3 + KTP and S-3 + KE 119-215), which increase water resistance and improve the physical and mechanical properties of concrete.

To study the phase composition of the cement stone, pieces were selected from some of the manufactured samples with dimensions of $40 \times 40 \times 40$ mm, from which the powders were made. The powders were passed through a No. 004 sieve. Then they were washed with alcohol and a series of
ether, after which they were dried in an oven at a temperature of t=05 °C. The studies were carried out on a DRON-3 diffractometer of Russian production.

In the process of studying the phase composition of the cement stone by the X-ray method, the degree of hydration was determined depending on the type of chemical additives.

**Table 2. The degree of hydration of cement stones, depending on the type of additive**

| Additive type   | Dosage and% by weight of cement | W/C | Spread of cement paste in mm | The degree of hydration of the cement stone in% |
|-----------------|---------------------------------|-----|-----------------------------|-----------------------------------------------|
| -               | -                               | 0,30| 109                         | 60                                           |
| C-3            | 0,5                             | 0,28| 110                         | 65                                           |
| KTP            | 0,01                            | 0,30| 116                         | 69                                           |
| KE 119-215     | 0,1                             | 0,30| 110                         | 71                                           |
| C-3+KTP        | 0,5+0,01                        | 0,28| 135                         | 61                                           |
| C-3+KE 119-215 | 0,5+0,1                         | 0,28| 120                         | 68                                           |

As can be seen from the results of studies (table 2.), the introduction and composition of concrete superplasticizer S-3, the degree of cement hydration at the age of 14 days after heat treatment (mode 2 + 3 + 6 + 3 .. at t = 80°С) is 65%, i.e. 5% more compared to samples without additives. The introduction of KTP into the composition of the cement stone, the degree of hydration increases by 9%. The highest degree of gyration of cement in cement stone (7%) is observed when an additive of organic silicon polymer is added to its composition, i.e. 1% more compared to samples without additives. The introduction of the complex additive S-3 + KTP into the composition of the cement stone: the degree of hydration increases by only 1%, and with the introduction of the complex additive S-3 + KE 119-215 - by 8%. The reason for the decrease in the degree of cement hydration by 3% (table 2) with the introduction of the complex additive S-3 KE 119-215, compared with the additive KE 119-215, may be explained by the formation of an absorption layer of the superplasticizer S-3 on the surface of the cement particle, which prevents the active interaction of the additive KE-119 215 and cement.

The results of the study of neoplasms of the cement stone by the X-ray method showed that in the samples without chemical additives, calcium hydroxide Ca(OH), calcium hydroycylates / C-S-H / and C₂AH₆ are formed.

The study of cement stone with the addition of C-3 showed that the formation of calcium carbonate CaCO₃ is observed, and in the samples with additives, KTP - the formation of calcium carbonate CaCO₃ hydrosulfoferrite of calcium C₃FGH₂₂ (similar to ettringite).

In cement stone with organosilicon polymers, the formation of minerals calcium hydroxide Ca(OH)₂, calcium hydrosilicate C-S-H, calcium carbonate CaCO₃ and calcium hydroaluminate was found, C₆AH₁₉ in samples with complete additives C-3 + KTP Ca (OH)₂ is formed, C-S-H and calcite-CaCO₃.

As noted, the additive KE 119-215 has a hydrophobic property. To study the hydrophobicity of concrete with a complex additive S-Z + KE 119-215, its wetting angle was determined.

The device for determining the angle of contact of concrete samples consists of a radiation source (which was used as a projector) and a screen (a sheet of blank paper). A concrete sample is placed between the radiation sources and the screen. Then we drop a drop of water onto the surface of the concrete sample and at the same time turn on the directional beam source. At this time, a projection of a water drop appears on the screen, which can be drawn on the screen. The drawing will make it possible to determine the concrete wetting angle (α).
For the study, concrete samples were used with a complex additive S-Z + KE 119-215 and without additives.

The results of determining the hydrophobicity showed that the wetting angle of concrete with С-3 + КЭ 119-215 is 440, and without additives - 300, i.e. 140 lower.

4. Conclusion
Thus, according to the results of the study, the structure of concrete with increased water resistance, according to the results of the study of X-ray structural analysis, the following conclusions can be drawn:

1. The result of the study of the physical and mechanical properties of concrete, depending on the amount of introduced plasticizing and air-entraining additives, showed that the most optimal dosage of complex additives S-Z + K E 119-215 to increase water resistance is 0.5 + 0.1% of the mass of cement.

2. The result of studying the water resistance of concrete, depending on the cement consumption, showed that an increase in cement consumption from 350 kg/m$^3$ to 400 kg/m$^3$, water resistance increases by two marks, and an increase in cement consumption from 400 kg/m$^3$ to 450 kg/m$^3$ - by one brand.

3. Frost resistance of concrete at a cement consumption of 350 kg/m$^3$ with a complex additive S-3 + K M9-215 in an amount of 0.5 + 0.1% of the cement mass reaches the F250 grade, i.e. does not meet the requirements of the trays. An increase in cement consumption up to 400 kg/m$^3$ increases frost resistance up to B300.

4. The strength of concretes with increased water resistance at the age of 28 days after TBO treatment with preliminary exposure for an hour is 88% of the brand strength, and at 2-hour exposure it is 27% more, i.e. 5% off the vintage.

5. An increase in the rate of temperature rise from 20°C/hour to 27°C/ hour - the strength of concretes with a complex additive C-3 + K M9-215 at the age of 28 days after TVO decreases by 7%, i.e. from 38% to 31% of the brand.

6. An increase in the duration of isothermal heating from 6 h to 8 h during heat treatment of concretes with a complex additive C-3 + K 149-215 contributes to an increase in strength at day-old age by 17%, i.e. from 55% to 72% of the brand.

7. An increase in the temperature of isothermal heating from 60°C to 80°C at TVO of concretes with a complex additive С-3 + K 149-215, its strength in one day increases from 77% to 99% of the brand strength.

8. The structure of concrete with a complex additive is exposed. The results of an electron microscopic study showed that evenly distributed micropores are formed in its structure.

9. The highest degree of cement hydration is observed in samples with additives К 119-215 and with complex additives С-3 + К 149-215, which, respectively, is 7% and 68% at the age of 14 days after TVO.

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