Assessment of the effect nitrites and fluorides on the change in the strength of concrete

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Abstract. The article describes examples of using inorganic salts and their mixtures for accelerate of hardening cement concretes in practice of modern construction. The results of the research of a complex mixed additive based on inorganic fluorides and nitrites, which accelerate the process of hardening of cement concrete are presented. The composition of the additive allows to accelerate the process of hardening the cement stone, due to the formation of dense and insoluble compounds in the pores of the cement stone. The experimental part included the process of determining the optimal amount of fluorides and nitrites in the additive. The results of the research showed a reduced additive consumption per 1m3 of heavy concrete. The advantages and disadvantages of the additive, which identified at experiments, allow us to recommend its use for accelerating the hardening of concrete in wet neutral and slightly acidic environments. The methods of tests that were carried out using certified and calibrated measuring instruments are described in detail. According to the results of the tests, the dependence of the concrete hardening rate on the amount of additive used was revealed, and the ways of practical use of the additive in the production technology of concrete and reinforced concrete were proposed. Also, according to the results of destructive and non-destructive testing, an increase in the strength of concrete by >15% compared to conventional concrete was found.

1. Introduction
Cement concrete hardening accelerators are used to provide increased speed of concrete strength gain and are mainly inorganic salts and their mixtures. Cement concretes (concrete on cement binders) are the main material for the construction of buildings and structures with a monolithic reinforced concrete frame and elements, as well as the basis for the production of precast concrete structures and products.

Due to the fact that concrete work in the construction of monolithic frames of buildings and structures can be carried out in the open air and at negative temperatures, it is not always possible to observe optimal hardening conditions, which is why the project strength of concrete may not be typed [1,2]. For this purpose, various additives are used in concrete one of which is additives - accelerators
of hardening [3]. When preparing the article, various sources of domestic and foreign authors on the topic of hardening accelerators were analyzed. Table 1 provides examples of additives based on ammonium salts, nitrites, and silicates [5-7]. In addition to these compounds, sulfates, chlorides, phosphates, fluorides, carbonates, and other additives can be used as hardening accelerators [8-10]. Most of these substances are inexpensive reagents, which justifies high economic efficiency [8-10].

Table 1. Additives - accelerators of hardening based on ammonium salts, silicates and nitrites.

| Additives          | Accelerators                                                                 |
|--------------------|------------------------------------------------------------------------------|
| Ammonium salts     | CO(NH$_2$)$_2$, NH$_4$NO$_3$ (in small concentrations)                        |
| Silicates          | Solutions of liquid glass (nNa$_2$SiO$_3$ + mH$_2$O or nNa$_2$OxSiO$_2$ + mH$_2$O) |
| Nitrites           | NaNO$_2$, Ca(NO$_3$)$_2$                                                     |

The quantity of salts and their mixtures used as cement concrete hardening accelerators has increased over the past decades, due to the search for effective and cost-effective additives. However, it also happens that some of the salts in the mixture are aggressive towards the cement stone of concrete or reinforcement steel, which results in negative effects on the hardening concrete.

These disadvantages are including:
- high hygroscopicity (the ability to absorb excess moisture, which leads to cracking and shrinkage of concrete, especially in dry conditions, figure 1a) [11,12];
- salting on the surface and in the depth of concrete pores (figure 1b) [13];
- corrosion of rebar inside the concrete body (for chloride additives) [14,15];
- presence of an unpleasant smell due to the release of reaction by-products (for example, when using ammonium salts or carbamide – smell of ammonia) [16].

Additional negative effects include the aggressiveness of the additive components to the skin, as well as to the respiratory and visual organs [17].

That’s why the search for perspective salt mixtures, which can be used as accelerators-hardening without these negative effects and at the same time with a low consumption per 1m$^3$ of finished concrete is actual problem of construction materials science.

Figure 1. a. Cracking of fine-grained concrete due to the hygroscopicity of the additive components. b. Salting out in pairs on the section of the surface layer.

The process of accelerated strength gain of concrete is related to the ability of components of additives - accelerators of hardening to react with one of the main components of hardening concrete – "free calcium hydroxide" (in the terminology of academician RAACS Sergey V. Fedosov) [18]. Such
processes cause the binding of "free calcium hydroxide" into strong and insoluble products or making the acceleration of its diffusion from the pores [19, 20]. An illustration of this process is the use of solutions of inorganic acids as hardening accelerators [21].

Also for some additives, the accelerated hardening process is associated with the binding of excess moisture at the hardening stage. An illustration of the effect of such additives in concrete can be the use of chlorides or carbonates [22, 23].

Based on these theoretical concepts, the purposes and objectives of the research was formed.

Purpose of research – to reveal regularities of influence of the additive-accelerator of hardening based on a mixture of inorganic fluorides and nitrites on the change of strength characteristics of cement concrete.

Research problem – based on theoretical and practical data, select and justify the effectiveness of a mixed composition based on inorganic fluorides and nitrites with the addition of a liquid glass solution, by studying the positive and negative effects, as well as the flow rate per 1m3 of concrete.

2. Methods

The following equipment was used in the production of concrete samples and weighing of additive components: hand electric vibrator model Zitrek Z-35-1,5, electronic scales Mucheng 0,1-500 (weighing accuracy 0.1 to 500g).

The following devices and equipment were used for testing control samples in the experimental part: a testing press model Matest C055N (certificate of the State register of measuring instruments in the Russian Federation № 65079-16, maximum load 2000 kN, the verification certificate from 16.05.2019, Figure 2a), the nondestructive shock-pulse device ONYX-2.5 by brand Interpribor (certificate of the State register of measuring instruments in the Russian Federation №30252-10, the verification certificate from 28.04.2019, Figure 2b), Canon 1200D digital SLR camera.

Determination of the strength of control samples was performed in parallel by methods of destructive and non-destructive testing to ensure the accuracy and reliability of the results obtained [24-26]. Processing of the received numerical data and plotting were performed in the Microsoft Excel 2010 software package.

![Figure 2. a) Test press model Matest C055N; b) the nondestructive shock-pulse device ONYX-2.5.](image)

As destructive testing method has been used to test concrete strength of control samples according to GOST 10180-2012 «Concretes. Methods for determining the strength by control samples». The essence of the method consists in the destruction of the concrete sample on the test press, thanks to this data will be obtained on the actual destructive load for concrete, which determines the compressive strength of concrete [24, 25].

As a nondestructive testing method was used the shock-pulse method according to GOST 22690-2015 «Concretes. Determination of strength by mechanical methods of non-destructive testing». The
essence of the method lies in the relationship between the strength of concrete and the impact energy (and its changes) at the time of impact of the tip with sensor with the surface of concrete [25, 26].

The procedure for conducting research is divided into theoretical and practical parts.

The theoretical part of the study included selection of initial reagents based on theoretical knowledge and calculation of the optimal ratio of these reagents in the composition to ensure the required characteristics. Based on the results of theoretical studies, tables were compiled for 3 variants of combinations of additive components (tables 2 and 3), which were clarified during the practical part.

**Table 2. Initial reagents in the compositions.**

**Core components**
- Distilled water, according to GOST 6709-72
- The potassium fluoride (KF) 2-water; (c.p.* quality), according to GOST 20848-75
- The ammonium fluoride (NH₄F); (c.p.* quality), according to GOST 4518-75
- Sodium nitrite (NaNO₂); (c.p.* quality), according to GOST 4197-74

**Auxiliary (functional) components**
- Liquid glass (solution of Na₂SiO₃) – it is introduced into the concrete at the stage after hydration with a mixed additive
- Functional additive FP-4MD

c.p.* - chemically pure

**Table 3. Variants of reagent combinations in mixed compositions.**

| The recipe (composition) | Composition, per 1 kg of additive |
|--------------------------|----------------------------------|
| C-1                      | H₂O (distilled): 90.0 %;         |
|                         | KF: 0.8%;                        |
|                         | NH₄F: 1.6%;                      |
|                         | NaNO₂: 0.4%;                     |
|                         | (liquid glass solution) Na₂SiO₃: 3.2% |
|                         | FP-4MD: 4%                       |
| C-3                      | H₂O (distilled): 90.0 %;         |
|                         | KF: 0.8%;                        |
|                         | NH₄F: 0.4%;                      |
|                         | NaNO₂: 0.8%;                     |
|                         | Na₂SiO₃: 2%                      |
|                         | FP-4MD: 6%                       |
| C-6                      | H₂O (distilled): 90.0 %;         |
|                         | KF: 1.2%;                        |
|                         | NH₄F: 0.2%;                      |
|                         | NaNO₂: 2%;                       |
|                         | Na₂SiO₃: 1.6%                     |
|                         | FP-4MD: 5%                       |

The practical part of the research included testing the additive for the effectiveness of the concrete strength set. To do this, the resulting composition in the form of a solution was added to the water in the manufacture of control samples in each batch.

Adding liquid glass to concrete serves to give it acid-resistant properties.
For destructive testing samples of 10×10×10 cm (Figure 3) were made from cement dough of normal consistency (water-cement ratio=0,3) made by mixing cement grade М500D0 with a solution of the additive. When the smell of ammonia appeared and then disappeared, a liquid glass additive was added to the cement dough. The concrete was compacted using a manual electric vibrator during mixing. After initial hardening, the samples were placed in a wet hardening chamber at atmospheric pressure (air humidity 99÷100%). The total number of control samples and samples with additives was 50 PCs (25 + 25 PCs). After a certain period of hardening the samples were determined by the ultimate compressive strength by destruction on a certified testing press (Figure 4).

![Figure 3. General view of test samples for destructive testing.](image)

![Figure 4. The destruction of the sample during the tests on the press.](image)

For nondestructive testing, samples of 3×3×3cm (Figure 5) were made from cement dough of normal consistency (water-cement ratio=0,3). The cement dough was prepared in the same way as in the case of preparing samples for destructive testing. The total number of control samples (samples without additives and samples with additives) was 100 PCs (50 + 50 PCs). After a certain period of hardening, the compression strength of the samples was determined by the shock-pulse method. The number of strikes on each face of the sample was not more than 1. The average value of compressive strength was determined then.

![Figure 5. General view of test samples for non-destructive testing.](image)
All the results obtained were averaged and entered in tables. The tables were used to plot the graphs of kinetics cement stone hardening.

3. Results

The results of determining the compressive strength of concrete by destructive testing are included in table 4.

According to the results of data analysis (table 4) graphs of comparative kinetics of concrete samples hardening in the period from 3 to 28 days are constructed (figures 7, 8, 9).

Table 4. Results of destructive testing.

| Test samples | Test method | Average compressive strength of cement stone, MPa |
|--------------|-------------|--------------------------------------------------|
|              |             | 3 days   | 7 days   | 14 days  | 21 days  | 28 days  |
| Without      | Destructive testing (testing of control samples) according to GOST 10180-2012. | C   | 30.98    | 34.25    | 37.01    | 39.96    | 42.02    |
| additives    |             |          |          |          |          |          |
| (samples is 10*10*10 cell cm) |             | C-1     | 22.14    | 28.44    | 33.71    | 39.94    | 43.77    |
| With         | Destructive testing (testing of control samples) according to GOST 10180-2012. | C-3  | 28.76    | 35.67    | 39.29    | 44.62    | 49.69    |
| addition     |             |          |          |          |          |          |
|              |             | C-6     | 24.48    | 30.78    | 37.33    | 41.23    | 44.81    |

Figure 6. Graph of comparative kinetics of cement stone hardening of the control sample (C) and the C-1 composition according to the data of the destructive control.
Figure 7. Graph of comparative kinetics of cement stone hardening of the control sample (C) and the C-3 composition according to the data of the destructive control.

Figure 8. Graph of comparative kinetics of cement stone hardening of the control sample (C) and the C-6 composition according to the data of the destructive control.

The results of measurements of concrete strength by nondestructive testing are included in table 5. According to the results of data analysis (table 5) graphs of comparative kinetics of concrete samples hardening in the period from 3 to 28 days are constructed (figures 10, 11, 12).

| Test samples | Test method | Average compressive strength of cement stone, MPa |
|--------------|-------------|--------------------------------------------------|
|              |             | 3 days   | 7 days   | 14 days  | 21 days  | 28 days |
| Without      | Non-destructive testing of shock-pulse method according to GOST 22690-2015. | C-1 | 16,40 | 22,71 | 29,43 | 36,99 | 41,11 |
| With addition| Non-destructive testing of shock-pulse method according to GOST 22690-2015. | C-3 | 19,82 | 28,19 | 37,84 | 43,38 | 48,55 |
|              |             | C-6 | 16,91 | 26,49 | 34,93 | 39,59 | 42,94 |
4. Discussion

According to research on the topic of corrosion of cement concretes, it was found that one of the main components of hardening concrete is "free calcium hydroxide" Ca(OH)$_2$. The character of the reaction of "free calcium hydroxide" with the components of the additive-accelerator of hardening will determine the speed and degree of strength gain [27-29].

The work of the tested compositions is based on the principle of initial dissociation and hydrolysis of individual components of the additive, after which a chemical reaction of binding of "free calcium
Hydroxide \((\text{Ca(OH)}_2)\) by fluoride ions occurs with the formation of slightly soluble calcium fluoride salts \((\text{CaF}_2)\) by direct reactions (1) and (2):

\[
\begin{align*}
\text{KF} + 4\text{H}_2\text{O} & \leftrightarrow [\text{K(H}_2\text{O)}_4]^+ \text{ (alkaline medium)} + \text{F}^- \\
\text{F}^- + \text{H}_2\text{O} & \rightarrow \text{HF} + \text{OH}^- \\
2\text{HF} + \text{Ca(OH)}_2 & \rightarrow \text{CaF}_2\downarrow + 2\text{H}_2\text{O};
\end{align*}
\]

\[
\begin{align*}
\text{NH}_4\text{F} + \text{H}_2\text{O} & \rightarrow \text{NH}_4\text{OH} + \text{HF} \text{ (acidic medium)} \\
2\text{NH}_3\text{OH} & \rightarrow 2\text{NH}_3\uparrow \text{ (smell of ammonia)} + 2\text{H}_2\text{O} \\
2\text{HF} + \text{Ca(OH)}_2 & \rightarrow \text{CaF}_2\downarrow + 2\text{H}_2\text{O};
\end{align*}
\]

Certain substances of the composition, for example, ammonium fluoride can increase the saturation limit of this solution with cement hydration products. The resulting calcium fluoride \((\text{CaF}_2)\) it is a slightly soluble compound and is in a nanodispersed state, which explains the increased rate of intergrowth of cement grains and increases the rate of hardening. Also, calcium fluoride is adsorbed in the pores of the cement stone, which prevents premature carbonization of concrete at the stage of its hardening.

The functional addition of a liquid glass solution (sodium silicate) was made after the concrete was mixing with water and additive, for the absence of the possibility of a side reaction (3), as a result of which the silica gel falls out:

\[
\begin{align*}
2\text{NH}_4\text{F} + \text{Na}_2\text{SiO}_3 & \rightarrow 2\text{NaF} + 2\text{NH}_3\uparrow + \text{H}_2\text{SiO}_3\downarrow
\end{align*}
\]

Based on the results of tests and graphs, it can be noted that the most effective hardening accelerator is the C-3 composition, which is associated with the highest increase in the strength of the cement stone. Also, the test results show that the use of the additive (C-3) most intensifies the strength set of cement stone in the period of hardening from 7 to 28 days.

The difference in the values of the initial average compressive strength of cubes of different sizes (3 days for samples of 10x10x10cm and 3x3x3cm) is explained by the difference the content of cement binder in them, when the cement is sealed with water.

\[
\begin{align*}
X = \left(\frac{R_{\text{concrete1}} - R_{\text{concrete2}}}{R_{\text{concrete1}}}\right) \times 100\%,
\end{align*}
\]

where: \(X\) – the resulting strength gain from the use of additive; \(R_{\text{concrete1}}\) – actual maximum compressive strength of concrete samples without additive at the last date of the calculation period; \(R_{\text{concrete2}}\) – actual maximum compressive strength of concrete samples with additive at the last date of the calculation period.

According to the value of the comparative kinetics of hardening of cement stone in the period from 3 to 28 days the increase in the strength of concrete in the case of the proposed additive C-3 was calculated by equation (4) and averaged:

1) for the destructive testing – 18,25%;
2) for the non-destructive shock-pulse testing – 20,68%.

5. Conclusions

Since the average increase in concrete strength was more than 15% based on the results of direct and indirect tests, we can conclude that the C-3 composition based on a mixture of nitrites and fluorides is effective as a hardening accelerator.

The additive is recommended to accelerate the curing of cement concretes in wet neutral or slightly acidic environments, due to the presence of hygroscopic components (potassium and ammonium fluorides). Using an additive for curing concrete in dry conditions can lead to increased cracking. Additive consumption for 1m3 of concrete was determined experimentally and is approximately 0,014–0,015 by weight of concrete. By comparison, the consumption of sodium chloride (often used as an additive-hardening accelerator) is up to 4% (0.04) by weight [30,31]. All this indicates a lower consumption of the additive. Large amounts of the introduced additive caused increased cracking and fragility of the samples.
Summing up the conclusion of the article it can be noted, that various combinations of inorganic fluorides and nitrates are promising mixtures as accelerators of hardening cement concretes, which is confirmed by experimental results.

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