

# Features of Fine-Grained Concrete Mechanically Activated with Silicate Sodium Solution

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Abstract. This article discusses the issue of improving the performance of finegrained concrete by using composite binders. In particular, in this study, sodium silicate (water glass) is considered as an additive to Portland cement, which gives concrete products an increased thermal resistance. The article describes in detail the behavior of fine-grained concrete after its mechanical activation with sodium silicate solution, and also describes the processes occurring in the body of the concrete mixture during mechanical activation. It should be expected that the performance characteristics of fine-grained concrete depend on the concentration of water glass and the time of its mechanical activation. The research method used is analysis, synthesis, generalization of reference and scientific literature, followed by an experiment. To study the effect of mechanical activation of a liquid glass solution on the structure-forming processes occurring in cement stone, the method of differential thermogravimetric analysis was applied. The results of this analysis and its interpretation are presented. In conclusion, information is provided on the compaction of the structure of a concrete stone.

Keywords: Concrete · Portland cement · Mechanical activation · Sodium silicate  $\cdot$  Chemical activation  $\cdot$  Concrete stone

## 1 Introduction

The use of composite binders in order to improve the performance characteristics of fine-grained concrete has firmly entered the practice of modern construction [\[1](#page-5-0)]. Quite often, sodium silicate is used as an addition to Portland cement – water glass, which gives concrete products an increased thermal resistance.

Liquid glass interacts with clinker materials, forming calcium hydrosilicate and sodium aluminate.

The sodium aluminate formed in the composition of the cement stone is a very strong accelerator of its setting. In addition to the reaction described above, there is a reaction between water glass and lime. The reaction proceeds with the formation of a very dense and durable calcium silicate, which, being deposited in the pores of the hardening cement stone, clogs them, providing an increase in the density and water resistance of concrete products. Liquid glass is a powerful hardening accelerator, and the effect is manifested the stronger, the higher the concentration of sodium silicate solution. Dilution of water glass solutions is accompanied by some depolymerization of the silicate anions contained in them. When cement is mixed with a dilute aqueous solution of water glass, part of the lime is extinguished with the formation of calcium hydroxide. The presence of calcium ions in the solution leads to the formation of finely dispersed calcium silicates. The mixing of cement with an aqueous solution of sodium silicate causes a change in the pH of the solution and the concentration of calcium ions in it in such a way that supersaturation on the calcium base does not occur. Calcium ions formed in the process of clinker hydration are bound by silicate anions into a gel, which leads to an accelerated formation of the stone structure. When interacting between water glass and slaked lime, calcium hydrosilicates of low basicity are formed.

The disadvantages of a composite binder, including Portland cement and water glass, can be attributed to a slight decrease in compressive and bending strength, caused by inhomogeneities in the structure of the cement stone arising from incomplete hydration of cement grains during accelerated setting [[2\]](#page-5-0). In this regard, there is a need to reduce the proportion of sodium silicate in the mixture due to the use of technological methods that provide better contact of sodium silicate with the solid components of the mixture.

Consequently, to increase the uniformity of the interaction of an aqueous solution of sodium silicate with cement clinker, it is necessary to subject the water glass to some action, as a result of which the colloidal solution would become more uniform and homogeneous. One of the types of such action is the mechanical activation of an aqueous solution in a vortex impact rotor-pulse apparatus. Sharp local pressure drops occurring during the rapid passage of the solution through thin holes in the rotor when they are aligned with the holes in the stator, cause not only depolymerization of molecules, but also rupture of part of the hydrogen bonds between water molecules, water is enriched with hydroxyl ions, as a result which increases the pH of the water [[3\]](#page-5-0). In a weakly alkaline medium and in the presence of electrolytes,  $SiO<sub>2</sub>$  particles coagulate, forming loosely bound nanodispersed aggregates. Thus, it is possible that under the action of cavitation, the size of relatively large sodium silicate particles decreases, and the particles themselves are distributed more evenly in the solution.

The quality of fine-grained concrete is determined not only by the strength and density of the cement stone, but also by good adhesion to the aggregate. In the process of crystallization, the surface of quartz grains acts as an active substrate for the formed nuclei of the solid phase, associated with the ability of quartz to adsorb lime with the formation of calcium hydrosilicates, on which other cement hydrates then grow. It is known that one of the ways to increase the adhesion strength of the cement stone to the aggregate surface can be the enrichment of the contact layer with substances that have chemical affinity for the aggregate material. It was found that as a result of wetting the quartz surface with liquid glass, the adhesion strength of the cement stone with the filler increases. During mechanical activation of a liquid glass solution under conditions of developed cavitation and subsequent mixing of a concrete mixture with this solution, an increase in contacts of uniformly distributed dispersed sodium silicate with a filler – quartz sand occurs.

It is also known that the treatment of solutions in a rotary-pulsation apparatus contributes to a change in viscosity [\[4](#page-5-0)]. By changing the structure of water systems, one can regulate their physicochemical properties [\[5](#page-5-0)]. Thus, fresh melt (more structured) water wets quartz much worse than ordinary (less structured) water. When mixing fine-grained concrete with water (or solution) restructured by mechanoactivation, water molecules better wet hard surfaces, including quartz, activating chemical reactions in the mixture.

#### 2 Materials and Methods

In order to determine the degree of influence of mechanoactivation of an aqueous solution of sodium silicate on the properties of cement compositions, the physicomechanical properties of cement stone and fine-grained concrete, mixed with activated solutions of  $Na<sub>2</sub>SiO<sub>3</sub>$  of various concentrations, as well as at various water-cement ratios were studied.

The following materials were used in the experiments: Portland cement grade M500 D0 (GOST 30515-97 "Technical description. Scope of application"), quartz sand (GOST 8736-85), distilled water (GOST 6709-72\* "Distilled water. Technical conditions"), liquid glass (GOST 13078-81 "Liquid sodium glass. Specifications").

The tensile strength of samples in bending and compression was established in accordance with the requirements of GOST 310.4-92 "Cements. Methods for determining the ultimate strength in bending and compression", GOST 10180-90 "Concrete. Methods for Determining Strength Using Control Samples". Heat resistance tests of the samples were carried out in a muffle furnace in accordance with GOST 20910-90. "Heat-resistant concretes. Technical conditions". Water absorption – according to GOST 12730.3-78 "Concrete. Method for determining water absorption".

To study the effect of mechanoactivation of a liquid glass solution used for mixing cement on the structure-forming processes occurring in a cement stone, the method of differential thermogravimetric analysis (DTGA) was applied, which was carried out on a derivatograph "Derivatograth Q-1500D" (Hungary). The heating rate of the samples during the experiment was 10 deg/min from room temperature (28...32 °C) to 1000 °C. The temperature curve and the weight loss curve were recorded. With the help of DTGA, the change in the crystalline structure of the cement stone was determined. As objects of analysis, we used prototypes with different ratios of water - Portland cement, kept under normal conditions for 28 days after mixing. The study of hydration products was carried out on samples taken from the inner layer of destroyed samples after strength tests and ground to a particle size of 0.8 mm.

The mechanical activation of liquid glass solutions was carried out on a laboratory setup, the diagram of which is shown in Fig. [1.](#page-3-0) A rotary-impulse apparatus, the rotor of which consisted of eight blades and was driven by an electric motor 5 rotating at a frequency of 3000 rpm, was connected to a water tank 6 by connecting hoses. Water with liquid glass circulated in a closed loop, the number of passage cycles along which determined the activation time – one cycle corresponded to 15 s.

<span id="page-3-0"></span>

Fig. 1. Laboratory plant (1 - inlet pipe; 2 - activator; 3 - outlet branch pipe; 4 - belt drive; 5 electric motor; 6 - container for liquid; 7 - unit for adjusting the rotor speed).

### 3 Results

To reduce the number of experiments and systematize the experimental data, the experiment planning method was used. The factor space consisted of the water-cement ratio, the time of mechanical activation and the concentration of water glass in the solution. When constructing a mathematical model, a three-level second-order Box – Benken plan was used.

The obtained regression equations formed the basis for constructing a number of surfaces displaying the dependences of the model responses on two factors at a fixed value of the third factor, which made it possible to determine the most rational values of technological parameters. It was found that the best characteristics were possessed by the samples, mixed at  $W/C = 0.29...0.31$  with an aqueous solution of sodium silicate of 5% concentration, activated for 45…60 s. According to the experimental data, the mechanical activation of an aqueous solution of liquid glass did not have a significant effect on the setting time of the cement paste. At the same time, samples of cement stone, mixed with mechanically activated 4…6% aqueous solutions of sodium silicate with water-cement ratios of 0.29…0.31, had increased strength characteristics in comparison with the samples, closed under other conditions.

The increase in the strength of the cement stone in compression and in bending is due to a decrease in the proportion of portlandite due to the binding of calcium into hydrosilicates, which is confirmed by DTGA data. Thermal decomposition of all samples was accompanied by three endothermic effects having a complex shape associated with the superposition of individual thermal effects. The first endothermic effect at temperatures up to 350  $\degree$ C is associated with the loss of free water and part of the water that is part of the hydroaluminosilicates and calcium sulfohydroaluminate.

The second endothermic effect associated with portlandite dehydration was observed at temperatures from 450 to 520 °C. The complex shape of the peak, corresponding to this endothermic effect, confirmed the interaction of sodium silicate with both CaO and Ca  $(OH)_{2}$ . In the form of the third endothermic effect, there were two maxima: at temperatures of 680 … 690 °C and 820 … 825 °C, formed during the dehydration of calcium hydrosilicates of different basicity and decomposition of calcite, respectively.

The least weight loss in the temperature range  $450-520$  °C, corresponding to the dehydration of calcium hydroxide, was characteristic of the cement compositions prepared on 5% sodium silicate solutions, however, an increase in the treatment time from 45 to 60 s led to a decrease in the amount of formed crystalline hydrates with 9.7 to 6.8%. This fact, as well as a decrease in the amount of crystalline hydrates with a simultaneous increase in the amount of portlandite in a cement stone, mixed on an inactivated solution of water glass of the same concentration, indicates the conditionality of the process of structure formation by activation modes. A decrease or an increase in the concentration of water glass  $(0.1\% \text{ or } 10\%)$ , as well as a change in the activation time, led to a weaker binding of calcium to calcium hydrosilicates. As a result, the ultimate strength in compression of a cement stone, activated for 45…60 s. 5% sodium silicate solution, increased up to two times in comparison with the control composition on non-activated distilled water.

#### 4 Discussion

Samples of concrete, mixed with a mechanically activated 5% solution of water glass, had the best performance. It should be noted that the most of calcium hydrosilicates and the least of portlandite were formed in the cement stone of the compositions obtained under similar conditions. In addition, these compositions showed the smallest loss of mass of physically bound water, which, in our opinion, may indicate a lower porosity compared to other compositions. The increased water absorption by concrete samples prepared on mechanically activated 0.1% and 10% sodium silicate solutions, respectively, can serve as an indirect confirmation of the formation of a larger volume of open pores in these samples and indicate a less dense structure. A higher content of chemically bound water, in our opinion, may be associated with the formation of a larger amount of ettringite-like phase, the needle-like dendric structure of the crystals of which reduces the density of concrete. Concrete on mechanically activated 0.1% and 10% sodium silicate solutions turned out to be less durable than concrete on 5% activated solutions, but more durable than control samples on non-activated 5% solution  $Na<sub>2</sub>SiO<sub>3</sub>$  and in non-activated distilled water.

When comparing the one can notice the presence of a correlation between the weight loss during thermal destruction of cement stone samples and the properties of fine-grained concrete. The strongest and most heat-resistant concrete specimens were obtained in compliance with the operating parameters at which the ratio of crystalline hydrates in cement stone to portlandite is the highest. Consequently, the possibility of controlling the structure formation in the cement stone of concrete is determined not only by the choice of the concentration of liquid glass in the mixing water, but also by the mode of mechanoactivation of the solution in the rotor-pulsating apparatus.

<span id="page-5-0"></span>In addition, the mechanoactivation of sodium silicate solution promoted an increase in the adhesion of the cement gel with the aggregate, which also improved the performance characteristics of fine-grained concrete.

# 5 Conclusion

Thus, the mechanical activation of aqueous solutions of sodium silicate, used for mixing concrete, promoted, firstly, the compaction of the structure of the cement matrix, and, secondly, a decrease in the amount of liquid glass in the mixture.

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