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Influence of synthetic calcium silicates on the strength properties of fine-grained concrete

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Abstract. The effect of additives based on acicular calcium hydrosilicates (xonotlite and tobermorite) and wollastonite, obtained from boric acid production waste in autoclave synthesis at a temperature of 220 °C, on the strength of fine-grained concrete, has been studied in this paper. It was shown that when the calcium hydrosilicates and wollastonite are introduced, an increase in the strength characteristics of concrete is observed. After heat and moisture treatment, the maximum increase in strength is observed with the addition of 4% of mass content of calcium hydrosilicates and 6% of mass content of wollastonite. After 28 days of hardening under normal conditions, the maximum increase in strength of concrete is observed with the addition of 4% of mass content of both types of additives. It was shown that the water absorption of concrete decreases with a maximum when 4% of mass content is added, as in the case of the introduction of calcium hydrosilicates, and wollastonite. With a further increase in the number of additives, the amount of water absorption increases, but these values remain below the values for the control sample without additives.

1. Introduction

Calcium hydrosilicates $n\text{CaO}\cdot m\text{SiO}_2\cdot p\text{H}_2\text{O}$ and wollastonite $\text{Ca}_6\text{Si}_6\text{O}_{18}$ have found wide application in the production of building materials, paper, paints, plastics, composite polymeric and metal-ceramic materials, sorbents for water purification. Scientists from different countries are conducting research to develop optimal methods for obtaining calcium silicates from available natural and technogenic raw materials.

Analyzing the prospects for the use of calcium silicates in the building materials industry, it is necessary first of all to focus on the variety of assortment of building materials and products based on cement, other binding substances and solidifying dispersed systems [1, 2].

A number of Russian and foreign studies have been devoted to the study of the effect of natural and synthetic wollastonite on the functional properties of building materials such as cement and concrete



[3–13]. It is shown that when using wollastonite as a filler, the strength characteristics, frost resistance, durability of building materials increase, and in some cases their resistance to corrosion increases, too.

Since 2007, the Institute of Chemistry of the Far-Eastern Branch of the Russian Academy of Sciences, in conjunction with a number of Russian universities and Armenian scientists, has been actively involved in the development of the physico-chemical basis for the complex processing of boric acid production waste (borogypsum) to produce calcium hydrosilicates, wollastonite and potassium fertilizers [14]. The possibility of using an additive based on wollastonite and pseudo-wollastonite obtained from borogypsum during the production of concrete has been shown [15, 16]. It is established that the additive under study contributes to the increase in strength, to a decrease in water absorption and to an increase in the frost resistance of concrete. The work continued with the production of calcium hydrosilicates and wollastonite with needle-like shape of particles by autoclave method in the temperature range 180–220 °C [17, 18].

The aim of this work is to study the effect of additives based on acicular calcium hydrosilicates (xonotlite and tobermorite) and wollastonite, obtained from borogypsum in autoclave synthesis (220 °C), on the strength of fine-grained concrete.

2. Experimental part

For the production of calcium hydrosilicates, borogypsum was mixed with the potassium hydroxide solution *p.a.* in the stoichiometric ratio. The synthesis was carried out in an autoclave made by Parr Instrument (USA) 4848 at a temperature of 220 °C for 3 hours. The reaction rate was monitored for the residual concentration of potassium hydroxide in the solution. To obtain wollastonite, the precipitate after autoclave treatment was calcined in the temperature range 900–1000 °C for 1 hour.

X-ray patterns of the samples were taken on an automatic D8 ADVANCE diffractometer with rotation of the sample in Cu K α radiation. X-ray phase analysis was carried out using the EVA search program with the PDF-2 powder data bank.

The specific surface was determined by the method of low-temperature adsorption of nitrogen using Sorbtometr-M instrument.

The morphology of the samples was studied on a high resolution scanning electron microscope (SEM) Hitachi S 5500 (Japan).

For the production of concrete beams and cubes, the following components were used (in relation to the weight part of the cement taken as 1): superplasticizer C-3 – 0.01; sand – 3; additive based on calcium silicates – 0.02–0.08, water – 0.42. The water-cement ratio was maintained constant in all compositions ($W / C = 0.42$). To the aqueous solution of the superplasticizer C-3, a calcium silicate-based material was added as a powder and mixed with a mixer for 2 minutes until a homogeneous suspension was obtained. The cement was poured into a bowl of a laboratory mixer (type 1.0203.01 made by Testing), water was poured into it, then stirred for 30 seconds. The resultant mass was added to the prepared suspension and stirred for 30 seconds. The next step was gradually adding sand to the mixture and stirring at 140 rpm for 2 minutes and at 285 rpm for 30 seconds. The prepared mixture was layered manually into the 3FK-70 (3FB-40) molds and vibrated on the vibrating pad (model SMZh-539) for 10 seconds. The mold with the samples was covered with glass, and after 1 day, stripping was done. The samples were placed in a normal hardening chamber (model KPU-1M) on liners and stored for 27 days. The temperature in the chamber is 20 °C, relative air humidity is 95%. After 3, 7 and 28 days from the date of manufacture, some samples were taken out of the chamber. Within 4 hours the samples were in the natural conditions of the room, in which they were subsequently tested, that is, at an air temperature of 20 ± 5 °C and a relative humidity of at least 55%. With heat and moisture treatment (HMT), the samples in the molds were placed in the KPU-1M steaming chamber and held for 2 hours at a temperature of 20 °C. Then there was an even rise in temperature to 80 °C for 3 hours and isothermal heating at a temperature of 80 °C for 6 hours. After isothermal warming, the molds with samples were cooling for no less than 2 hours, then the chamber cover was opened, 24 hours after production samples were molded and immediately tested. Samples were tested for bending and compression on a combined machine (type 1.0244 made by Testing).

Samples were tested for water absorption in accordance with the guidelines described in [19].

3. Results and discussion

According to X-ray diffraction analysis, the following were found in the synthesized sample: crystalline phases of unreacted anhydrous calcium sulfate CaSO_4 , those of tobermorite 11 \AA $\text{Ca}_5(\text{OH})_2\text{Si}_6\text{O}_{16}\cdot 4\text{H}_2\text{O}$ of orthorhombic modification (PDF-2, 00-019-1364) with crystal cell parameters: $a=11.27000$; $b=7.35000$; $c=22.74000$; $\alpha = 90.000$; $\beta = 90.000$; $\gamma = 90.000$ and of those of xonotlite $\text{Ca}_6[\text{Si}_6\text{O}_{17}](\text{OH})_2$ of monoclinic modification (PDF-2, 00-029-0379) with the parameters of the crystal cell: $a=17.02900$; $b=7.35600$; $c=7.00700$; $\alpha = 90.000$; $\beta = 90.340$; $\gamma = 90.000$. The specific surface area of the obtained precipitate is $30.7 \text{ m}^2\cdot\text{g}^{-1}$. After annealing at $900 \text{ }^\circ\text{C}$, crystalline phases of unreacted anhydrous calcium sulfate CaSO_4 and wollastonite of the triclinic modification (PDF-2, 01-084-0654) were found in the sample with crystal cell parameters: $a=7.92580$; $b=7.32020$; $c=7.06530$; $\alpha = 90.055$; $\beta = 95.217$; $\gamma = 103.426$. The specific surface of the sample obtained is $4.5 \text{ m}^2\cdot\text{g}^{-1}$. As a result of annealing at $1000 \text{ }^\circ\text{C}$, only the wollastonite phase of the triclinic modification with the same parameters of the crystalline cell is present in the sample composition (figure 1).

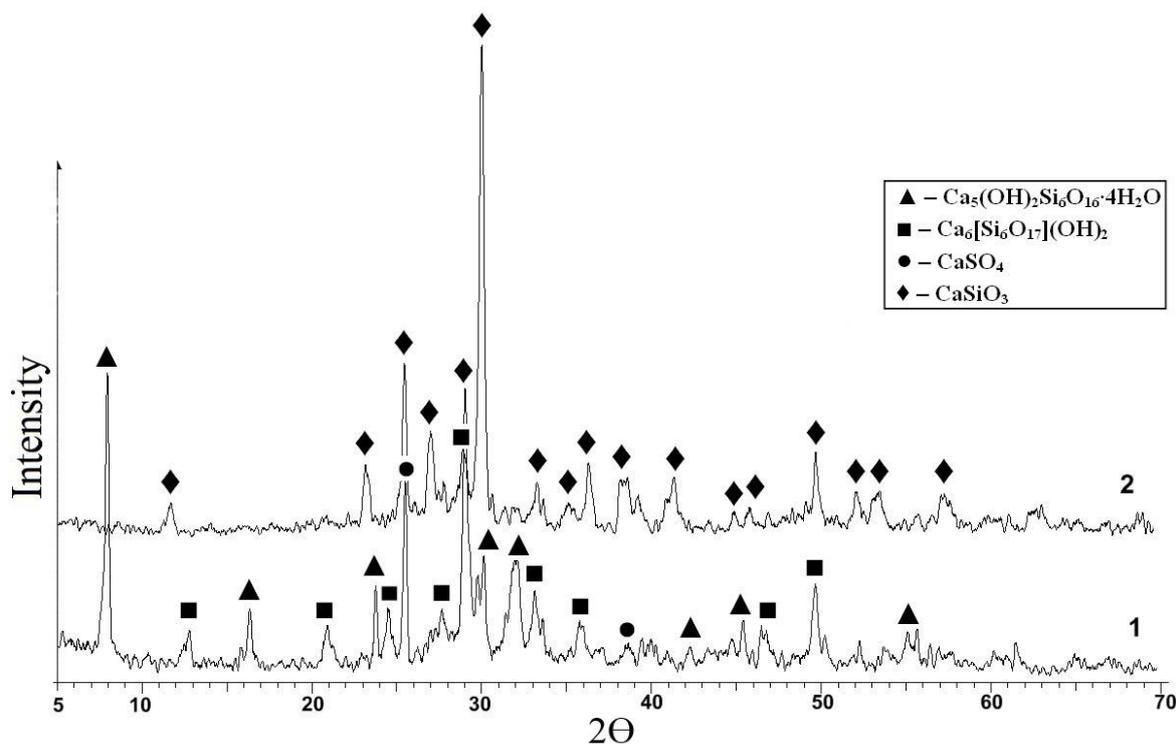


Figure 1. Diffractograms of samples obtained as a result of autoclave borogypsum treatment: 1 – initial sample (before annealing); 2 – after annealing at $1000 \text{ }^\circ\text{C}$.

According to the data of scanning electron microscopy, the samples obtained, both initial and after annealing, consist of agglomerates of nano-sized particles having a predominantly acicular shape (figure 2). However, after annealing, the pinching of the needle particles is observed (figures 2c, d). After annealing at 900 and $1000 \text{ }^\circ\text{C}$, no significant differences are observed in the particle morphology.

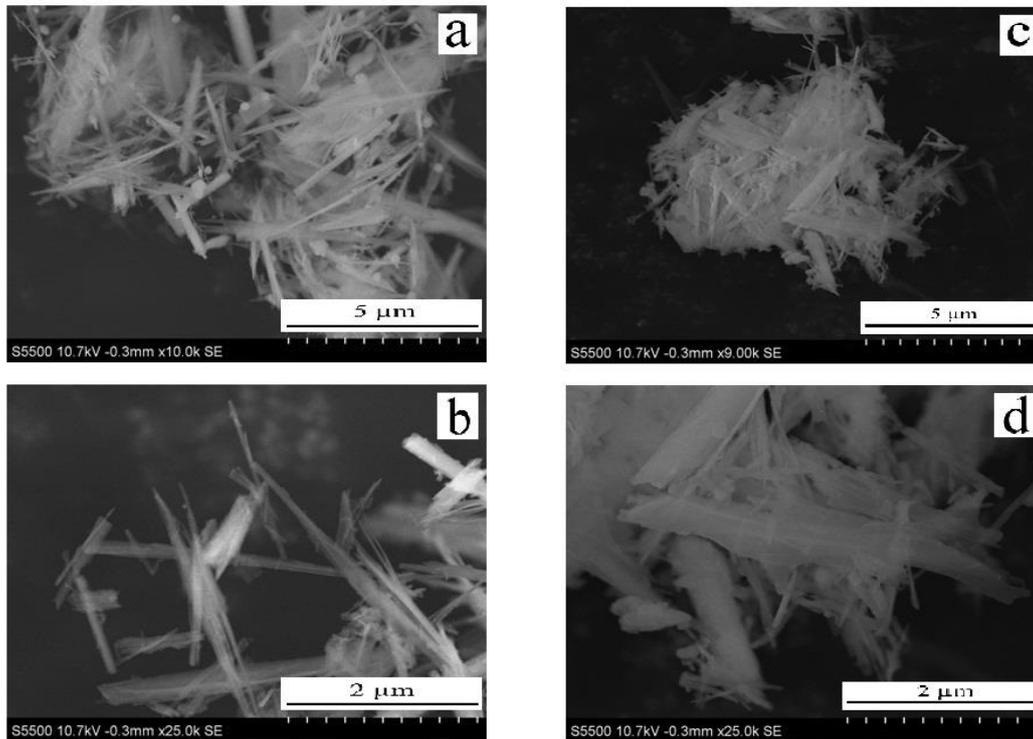


Figure 2. SEM images of microparticles of the sample obtained as a result of autoclave synthesis, after drying at 85 °C (a, b) and after annealing at 1000 °C for 1 hour (c, d).

The table 1 gives data on the strength characteristics of concrete obtained with the addition of calcium hydrosilicates and wollastonite in an amount of 2–8% by mass content.

Table 1. Strength characteristics of concrete with the addition of calcium hydrosilicates and wollastonite.

Additive	Mass content of additive (%)	HMT		Normal hardening conditions (28 days)	
		Bending (MPa)	Compression (MPa)	Bending (MPa)	Compression (MPa)
Based on calcium hydrosilicates (xonotlite and tobermorite)	No additive	3.4	21.7	5.0	27.4
	2	4.3	23.0	5.1	31.6
	4	4.4	25.9	5.2	33.5
	6	3.4	22.3	5.1	31.3
	8	3.3	20.4	5.0	28.5
Based on wollastonite obtained after calcining at 900 °C	No additive	3.3	21.2	5.07	28.3
	2	3.7	23.1	5.5	34.6
	4	4.3	24.9	5.6	36.6
	6	4.8	27.4	5.3	33.1
	8	4.3	22.9	5.07	31.8

As can be seen from the table, with the addition of calcium hydrosilicates and wollastonite, the strength characteristics of concrete are increased. After HMT, the maximum increase in strength is observed when 4% of mass content is introduced for calcium hydrosilicates and 6% of mass content

for wollastonite. In this case, the strength increases with bending by almost 30% for both additives, with compression – by 19 and 24% for calcium hydrosilicates and wollastonite, respectively. After 28 days of hardening under normal conditions, the greatest increase in strength is observed with adding 4% of the mass content of both additives. When calcium hydrosilicates are introduced, the flexural strength increases insignificantly, when compressed – by 22%. In case of the addition of wollastonite, the strength characteristics of bending are increased by 25%, while compression is increased by 21%. With an increase in the amount of both types of additives up to 8% of mass content, there is a decrease in strength. However, in case of the addition of wollastonite in an amount of 8% by mass content, the values of bending strength and compression remain above the control values (without additives).

Figure 3 shows the dependences of the water absorption of concrete on the amount of the additive based on calcium hydrosilicates and wollastonite.

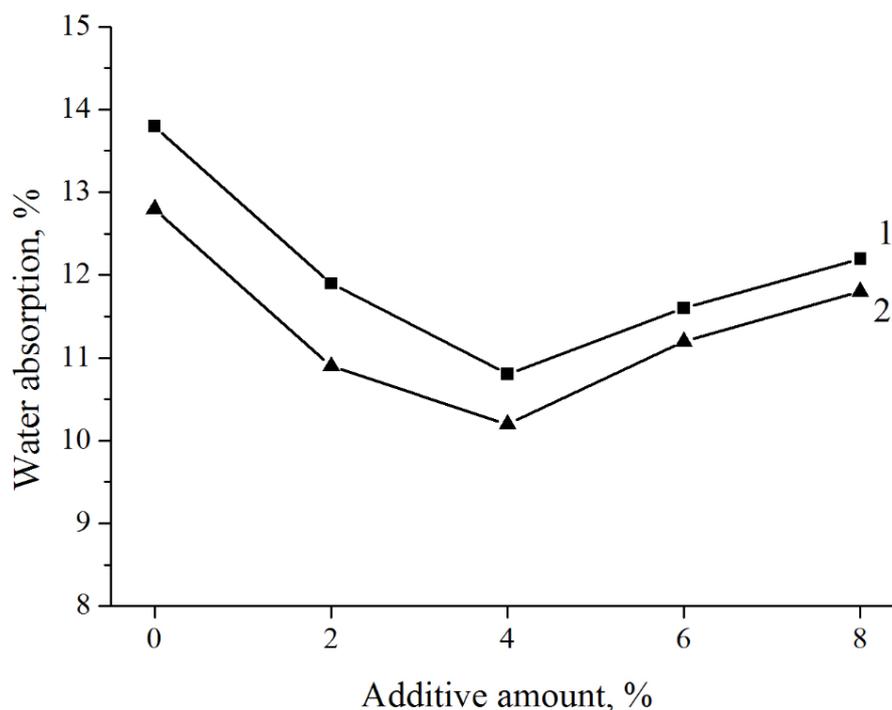


Figure 3. Dependences of the water absorption of concrete on the amount of the additives based on calcium hydrosilicates (xonotlite and tobermorite) (1) and wollastonite (2).

As can be seen from the above dependences, when the additives are introduced, a decrease in the water absorption is observed with a maximum of 4% of mass content. With a further increase in the amount of the additives, the amount of water absorption increases, but these values remain below the values for the control sample without additives.

The authors continue to actively study the relationship between the conditions for obtaining calcium silicates, their structure, the shape and size of the particles and the functional properties of concrete with a different amount of described additives.

4. Conclusions

Thus, studies on the effect of additives based on acicular calcium hydrosilicates (xonotlite and tobermorite) and wollastonite, obtained from boric acid production waste in autoclave synthesis at a temperature of 220 °C, have shown that when they are introduced, the strength characteristics of fine-grained concrete are increased. After heat and moisture treatment, the maximum increase in strength is observed when 4% of mass content is introduced for calcium hydrosilicates and 6% for wollastonite.

After 28 days of hardening under normal conditions, the greatest increase in strength is observed with the addition of 4% of mass content for both types of additives. It is shown that the water absorption of concrete decreases with a maximum when 4% of mass content is added, as in the case of the introduction of calcium hydrosilicates, and wollastonite. With a further increase in the number of additives, the amount of water absorption increases, but these values remain below the values for the control sample without additives.

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