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Improvement of mechanical characteristics of mortar by using of wollastonite

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Abstract. Geomimetic (nature-like) principles are proposed for optimizing the strength properties of cement mortar, consisting in the integrated effect of wollastonite on the processes of structure formation of cement paste. At the same time, wollastonite (2, 4, 6 and 8 % of the weight of cement) in the composition of mortar has a double function: it serves as a filler (silica-containing component) and microfiber. It was revealed that the incorporation of wollastonite into the mortar mix makes the material easier without sacrificing strength. It has been proven that in the initial period of hardening (3 and 7 days), hydration processes are accelerated, and early strength for all formulations developed with the addition of wollastonite is higher than for the control specimen. An analysis of the structure formation of cement paste from the standpoint of geomimetics reveals the similarity of wollastonite CaSiO3 to the main minerals of the Portland cement clinker Ca2SiO4 (belite) and Ca3SiO5 (alite). This similarity leads to the creation of a chemically uniform and, accordingly, strong microstructure. Micro reinforcement of the mortar matrix with wollastonite occurs due to the elongated shape of the microfiber and its good adhesion to the mortar matrix. The results can be used by technologists in the design of mortar mixes for the construction of buildings and structures for various purposes.

1. Introduction

The trend of building materials science is aimed at reducing the amount of cement in building materials, which is achieved by using various mineral raw materials of natural and technogenic origin in binders in the preparation of mortars and mortar mixes [1–3]. To control the structure formation of the cement composite, it is necessary to use new components. At the same time, the prospect of using

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components increases if they are extracted from industrial waste. Moreover, from the standpoint of geomimetics (the science of creating materials from the standpoint of studying geological processes), the mineral composition of the binder components should be identical to the minerals of Portland cement clinker [4].

The use of such raw materials as various natural pozzolanic ones, as well as industrial waste (fly ash, blast furnace slag, rice husk ash, etc.), has been studied quite well [5–7]. These materials make it possible to obtain high-performance mortars with compressive strength above 100 MPa, flexural strength above 15 MPa and a diverse range of high exploitation characteristics. This allows the creation of materials of construction for a variety of special applications. At the same time, the use of waste of various genesis is a priority in the production of building materials.

A promising material is wollastonite, which is formed as waste from boron production. It was previously proved that wollastonite $CaSiO_3$ ($Ca_6Si_6O_{18}$) due to the micro-reinforcing effect is able to give mortar enhanced characteristics of strength (tensile, flexural and compression) and impact resistance [8–10]. Natural wollastonite is characterized by an elongated crystal structure, upon cleaving of which grains of needle-shaped form are formed [11–12]. The needle-shaped form of wollastonite grain determines the main direction of its use as a micro-reinforcing filler with the ratio of fiber length to its diameter from 3 : 1 and higher [13–14]. The micro-reinforcing properties of wollastonite ensure non-shrinkage of materials manufactured with its use. In the production of composite building materials and cement-based products, it was found that its physicochemical affinity with cement-containing raw materials, active selective adsorption of binder hydration products, has a significant effect on the rheological parameters of concentrated suspensions and pastes, the formation of structure, strength and deformation properties hardened composites.

According to the law of similarity, as an integral part of geomimetics [15–16], the selection of composite components should be carried out from the point of view of similarity of their characteristics, such as adhesion, for example, coefficient of linear thermal expansion, deformation properties, etc. Mortars with added wollastonite in closed form are very plastic, easy to apply and have good adhesion to various surfaces [17–18]. Wollastonite increases the water retention capacity of closed mixtures, enhances their structure formation and almost completely eliminates shrinkage during hardening. Possessing good adsorption properties, wollastonite eliminates salt formation. Mortar with the addition of wollastonite have good weather- and frost resistance [19–23].

The leading five countries for the production of wollastonite are shown in Fig. 1 [24]. In Russia, mining of wollastonite was previously carried out only in the Altai, but now mining has been discontinued [25]. Therefore, a search for new sources of wollastonite is necessary. Especially promising is the disposal of production waste.

Wollastonite has a wide range of applications: the high-performances cements, paints, varnishes, household and technical ceramics, molding materials, car brake pads, plastics, additives for special glasses against electromagnetic radiation, composite materials against radioactive radiation, in medicine and others. Wollastonite is included in the list of strategic raw materials in the USA, Great Britain and China [8–14]. In this work, it was studied the use of wollastonite in mortar, the resulting waste from boron production.

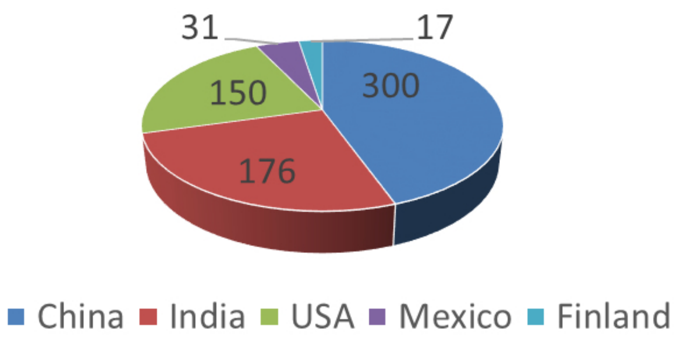


Figure 1. The largest countries for the production of wollastonite (thousands tons).

However, the use of wollastonite as a silica-containing additive that affects the hydration of clinker minerals is not well understood. Thus, the aim of the work is to analyses the effect of wollastonite on the characteristics of mortar, both as a binder filler and reinforcement as microfiber.

To achieve this goal, the following tasks were solved:

– study of the microstructure of wollastonite;

- selection of the optimal amount of wollastonite as a binder filler;
- study of fresh and hardened properties of the modified composite
- comparation of hydration schemes of pure cement and cement-wollastonite binder.

2. Materials and methods

2.1. Materials

Portland cement CEM I 42.5R (Spasskcement, Russia) was used as a binder. The chemical and mineralogical composition of the Portland cement is listed in Table 1.

Table 1. Chemical and mineralogical composition of Portland cement CEM I 42.5N.

Chemical composition (%)							Mineralogical composition (%)			
CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O	C_3S	C_2S	C_3A	C_4AF
65.94	21.70	5.02	4.20	1.25	0.40	0.78	61.0	16.3	6.2	12.8

The ratio of the content of the main components of the presented cement sample indicates its compliance with the Russian standard GOST 31108-2016 (Table 2). The microstructure of used Portland cement grains is shown in Fig. 2.

Table 2. Physical and mechanical properties of Portland cement used.

Compressive strength, MPa		Setting time, min		Fineness of grinding (passed through a sieve 008), *%*	Specific surface area, m^2/kg	Standard consistency, *%*
2 days	28 days	start	end			
14.0–18.8	38.0–47.0	130–240	225–360	88–90	290	22.25–26.25

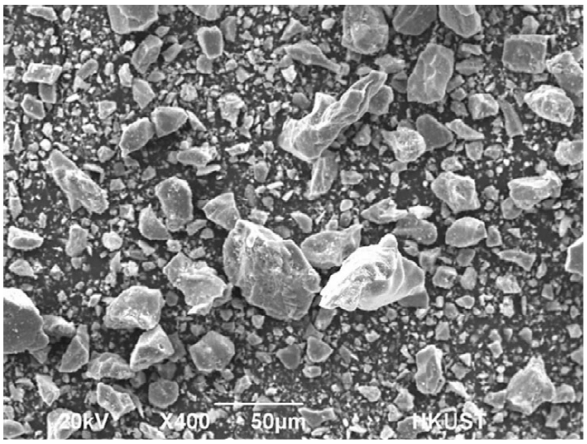


Figure 2. Microstructure of Portland cement grains used.

Wollastonite (calcium silicates $CaSiO_3$) obtained from the laboratory of protective coatings and marine corrosion of the Institute of Chemistry of the Far Eastern Branch of the Russian Academy of Sciences from borogypsum of technogenic origin (Primorsky Krai) was used as a partial replacement for Portland cement. Among natural minerals, wollastonite is highly resistant to aqueous solutions and solutions of chloride salts; therefore, it was chosen as a filler and aggregate for the developed corrosion-resistant fine-grained concrete. Substitution of Portland cement was carried out in an amount of 2, 4, 6 and 8 % by weight. River sand with medium size modulus (fraction 20–40 mm) was used as a filler (Razdolnoye, Russia). The water absorption of the sand is 5 % and the bulk density is 1,180 kg/m^3. Table 3 lists the determination of grain size composition of sand.

For mixing composite binders and making mortars, a water from city pipelines was used that meets the requirements of the Russian standard GOST 23732-2011. The water does not contain harmful impurities and has a pH of 6.52. Its total hardness is 0.41–0.60 mg-eq/l. Mixing water does not contain dissolved acids or alkalis, which hinder the normal setting or hardening of binders, harmful impurities, decomposing plant substances, which can have a harmful effect on mortar hardening. To increase the plasticity of the mortar mixture, S-3 superplasticizer (SP) in liquid form was used (Vladimirsky ZBK, Russia).

Suplasticizer S-3 is an organic synthetic substance based on the condensation product of naphthalenesulfonic acid and formaldehyde with a specific ratio of fractions with different average molecular weight – sodium polynaphthalene methylene sulfonate or methylenebis (naphthalene sulfonate) sodium.

Table 3. Determination of grain size composition of sand.

Content of coarse-grained impurities,%		Residues on sieves,%	Granulometric composition Sieve sizes, mm						Fineness module
> 10 mm	> 5 mm		2.5	1.25	0.63	0.315	0.16	< 0.15	
–	–	partial	2.0	6.5	34.5	41.5	13.0	2.5	2.4
		full	2.0	8.5	43.0	84.5	97.5	100	

2.2. Mix design

Variation of the addition of wollastonite was carried out in the range from 0 to 36 kg per 1 m^3 of mortar mix (Table 4).

Table 4. Results of the selection of the optimal composition of CB (per 1 m^3 of mortar mix).

Mix ID	CEM I, kg	Wollastonite, kg	Sand, kg	Water, l	SP, l
1	450	-	1500	270	38
2	441	9	1500	270	38
3	432	18	1500	270	38
4	423	27	1500	270	38
5	414	36	1500	270	38

Cement was poured into a bowl of a laboratory planetary mixer (Testing, Germany) and water was added to it, then the mixer was turned on at 140 rpm, stirred for 120 seconds, turn on the sand supply, after another minute switch to accelerated mode, namely 285 rpm min, and interfere with another 150 seconds. Fig. 3 shows the flow chart of specimens preparation.

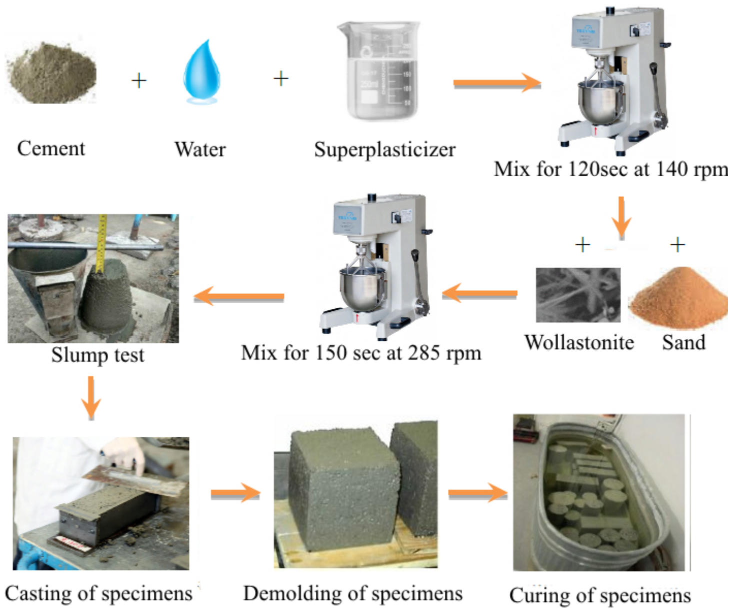


Figure 3. Flow chart of specimens preparation.

Specimens of 40×40×160 mm in size (for flexural studies) and 100×100 mm (for compression studies) were made. Six specimens of each composition and size were made. The prepared mixture was manually layered in layers in molds and vibrated on a vibrating platform (SMZ-539, Russia) for 5 sec. The mold with the specimens was covered with glass, and after 1 day, the formwork was carried out. The prepared specimens were placed in a normal hardening chamber (model KPU-1M) on pads and stored for 27 days (Fig. 4). The temperature in the chamber is 20 °C, the relative humidity is 95 %. After 28 days from the date of manufacture, the samples were removed from the chamber. For 4 hours, the specimens were in the natural conditions of the room in which they were subsequently tested, i.e., at an air temperature of 20±5 °C and a relative humidity of at least 55 %.

Figure 4. Loading samples into the steam chamber.

2.3. Methods

The density of mortar specimens was determined by dividing the mass of specimens with a size of 100×100 mm by their volume. To determine the average density of concrete, three cubic samples hardened under the same conditions and having the same hardening age (60 days). Saturated samples were weighed to prevent drying cracks. The mass was measured after 2 hours of boiling, followed by immersing the samples in ionized water in an evacuated state for another 24 hours. Then the samples were kept at a temperature of 105 °C for 24 hours to remove water and obtain a dry weight.

The determination of the slump of the mortar mix was carried out using the Abrams cone as follows. First, the cone was filled with a mortar, which was pierced to seal and remove voids, after which it was supplemented with the mix. Then the cone was removed (lifted vertically) and positioned next to the mortar. After that, a plasticity test was carried out.

The compressive and flexural strength of the specimens was tested on a Testing hydraulic press (Germany) according to Russian Standard GOST 310.4-81. The compressive strength of an individual specimen was calculated as the quotient of dividing the value of the breaking load on the working area of the plate. Compressive strength was calculated as the arithmetic mean of the four largest test results on six specimens.

The possibility of using wollastonite, the manufacture of samples and their testing was carried out at the Department of Building Structures and Materials of Far Eastern Federal University by Dr. Kozin. The morphological features of the wollastonite microstructure were studied using a Hitachi S5500 scanning electron microscope (Japan).

Differential thermal studies were carried out on a Shimadzu DTG-60H thermogravimetric analyzer. The programmed heating of the furnaces from 20 to 1500 °C is carried out by an electronic thermal heater at a rate of 20 °C/min. A platinum thermocouple with an accuracy of 5 °C measures the temperature (T), while the signal is recorded on paper with a sweep speed of 2.5 mm/min. The temperature difference (ΔT) between the test substance and the reference, proportional to the thermal effect, is recorded as a DTA curve (sensitivity 500 μV). Simultaneously with the DTA curve, the weight loss curve (TG) and its derivative (DTG) are recorded (sensitivity 500 μV). Sample weight 113 mg. The weighing accuracy was 0.05 mg.

3. Results and Discussion

To understand the mechanisms of the influence of wollastonite additives on the physicomechanical characteristics, the microstructure of wollastonite was considered (Fig. 5). Wollastonite is a white powder with a density. It is characterized by the presence of elongated plate and needle crystals, when cracked which form needle-shaped grains. The elongated structure of microfibers will obviously contribute to the hardening of the cement matrix.

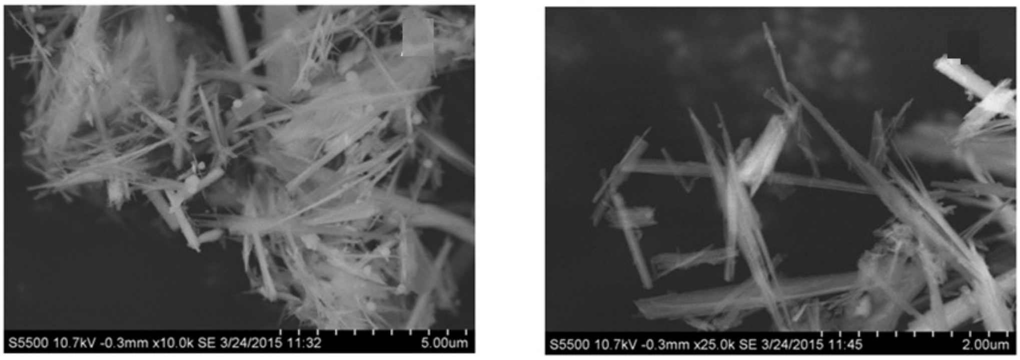


Figure 5. Microstructure of wollastonite fibers.

The slump of the developed mixes is shown in Fig. 6. It is seen that the introduction of wollastonite makes the mix more rigid. This is due to the reinforcing effect of wollastonite. At the same time, even with the maximum introduction of the addition of wollastonite 8 %, the mix shows sufficient fresh characteristics.

Figure 6. Slump test results.

At the same time, a decrease in the density of mortar at the age of 28 days is observed, depending on the increase in the percentage of replacement of Portland cement with wollastonite (Fig. 7), which is explained by a lower density of wollastonite compared to Portland cement. It should be noted that the dependence is not linear, with successive replacement for every 2 %, the density decreases by 25, 29, 40 and 38 kg/m^3. This fact, obviously, can be explained by the formation of less dense calcium hydrosilicates due to the secondary hydration of calcium hydroxide released during hydration of alite with wollastonite minerals. Moreover, the positive effect of reducing the specific gravity of the structural material does not adversely affect the strength characteristics. Accordingly, even considering the lower density, there are prerequisites to count on the best characteristics of weather resistance and frost resistance.

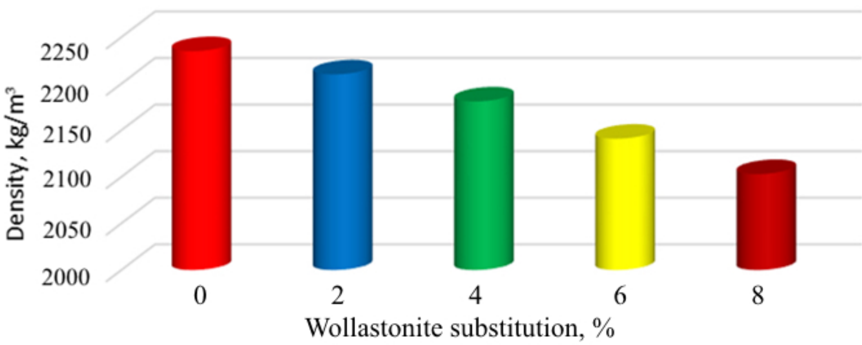


Figure 7. Effect of wollastonite addition on mortar density.

As a result of studying the compressive strength of mortar specimens, it was found that all the studied doses of wollastonite (2–8 %) give an increase in this parameter on days 3, 7 and 28 (Fig. 8).

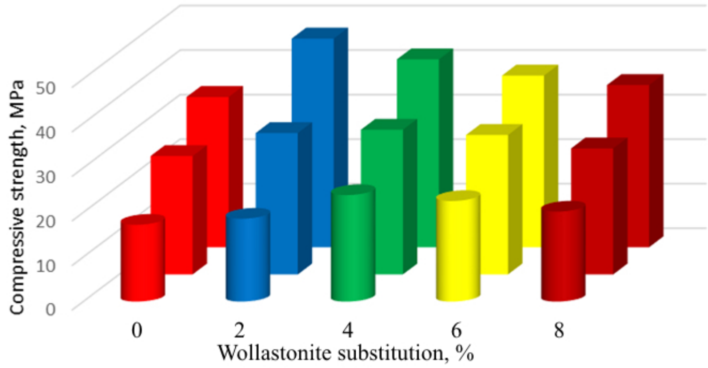


Figure 8. Effect of wollastonite addition on mortar compressive strength (3, 7 and 28 days).

Particles of polymineral cement-wollastonite binder play a complex role in the structure formation of cement paste: firstly, filling the space of micropores, harden the cement paste; secondly, they form active crystallization centers; and, finally, participate in chemical reactions with the formation of new phases, due to which crystalline intergrowths of low-basic CSH are formed with a ratio C / S ≤ 1.0 instead of primary high-basic hydrosilicates of calcium and portlandite.

In the initial period of hardening (3 and 7 days), acceleration of hydration processes was revealed already in the initial period. The compressive strength on 3 and 7 days for all developed formulations with the addition of wollastonite has a higher value than for the control composition.

At the second stage of hydration of the composite binder, the role of chemical processes is growing, which contribute to a significant modification of the phase composition of the system: the balance shifts from primary crystalline hydrates (calcium hydroxide and highly basic CSH) to more stable secondary fine crystalline hydrates, represented by low-basic calcium hydrosilicates. Obviously, this conclusion is valid until an excessive amount of silica-containing active filler begins to cover the surface of new phases, and this prevents the formation of contacts and the coalescence of crystalline hydrates. Based on the foregoing, we obtain the hypothesis of the presence in the binder of the composition of the optimal volumetric concentration of silica filler, taking into account its pozzolanic activity. As for the inert properties of the filler, its effective dosage will depend directly on the total volume of capillary pore space of the hardening composite necessary for the clogging. The presence of secondary generation hydrosilicates, formed as a result of the binding of CSH with an active silica-containing additive in the composition of cement-wollastonite binder, is noted (Fig. 9).

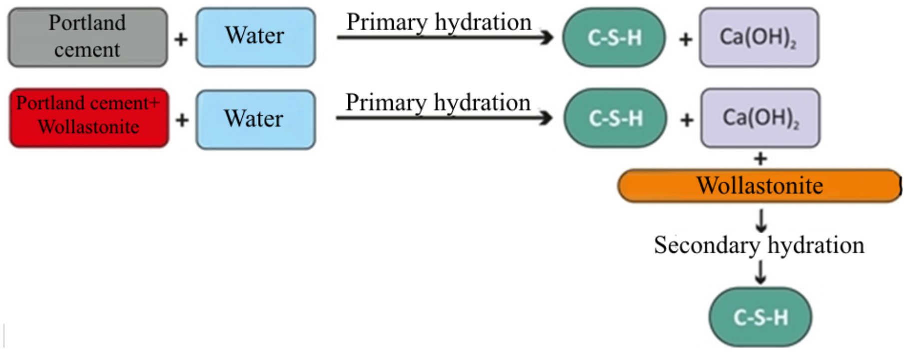
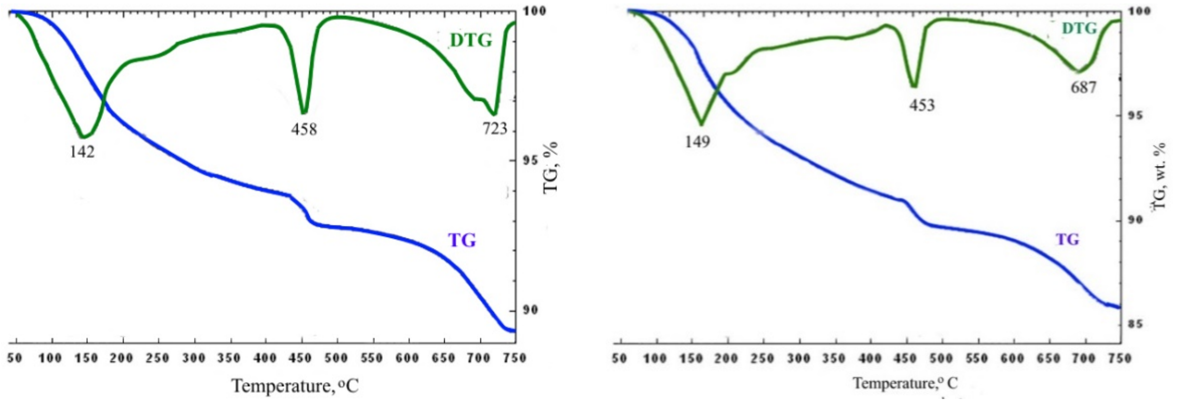


Figure 9. Comparation of hydration schemes of pure cement and cement-wollastonite binder.

The use of the developed composite binder makes it possible to compact the structure of the hardening composite, thus, the controlled structure formation of a rigid matrix with reduced porosity is carried out, and this, accordingly, entails the strengthening of the composite. Clinker grains, as well as individual clinker minerals and wollastonite mineral additives, are large fragments of active solids that form nodes of the spatial lattice of cement paste during hydration. In the course of this, crystalline hydrates, which, depending on the activity of the mineral, are formed simultaneously or sequentially, filling the free space, by partial ingrowth, bind to the already existing framework. Thus, the described processes lead to the formation of a mechanical mixture of crystals, differing in composition, size and shape, and connected by a gel-like mass.

Particles of a polymineral binder play a complex role in the structure formation of a cement-wollastonite paste: firstly, filling the space of micropores, they compact and harden the cement stone; secondly, they form active centers of crystallization; and, finally, they participate in chemical reactions with the formation of new phases, due to which crystalline intergrowths of low-basic CSH with a C / S ratio of ≤ 1.0 are formed instead of primary highly basic calcium silicate hydrates and portlandite.

The strengthening of the cement composite is confirmed by the results of the DTGA patterns of the specimen, which has 8 % of wollastonite at the age of 3 and 28 days (Fig. 10).



a) b)

Figure 10. DTA and TG patterns for a specimen with 8 % wollastonite at the age of 3 (a) and 28 (b) days.

As the 3-day specimen, the 28-day one shows a loss of water at all stages with the greatest intensity at temperatures of 70–190 °C. After 200 °C, a decrease in the amount of removed water is also observed. The endothermic peak associated with dehydration below 200 °C is associated with the partial removal of water from ettringite and weakly crystallized and amorphous phases of calcium hydrosilicates. A weak endothermic peak with water loss in the range of 320–410 °C corresponds to the dehydration of weakly crystallized boehmite AlO(OH) or other products of ettringite decomposition, such as $CaO \cdot Al_2O_3 \cdot 6H_2O$ hydrates. A decrease in the amount of $Ca(OH)_2$, which is an indicator of the pozzolanic reaction, occurs due to its binding during the formation of second generation hydrosilicates.

Analyzing the above from the standpoint of geomimetics, we reveal the similarity of wollastonite $CaSiO_3$ with the main minerals of the Portland cement clinker Ca_2SiO_4 (belite) and Ca_3SiO_5 (alite), which allows one to obtain a chemically uniform and, accordingly, strong structure of cement paste.

As a result of varying the amount of applied wollastonite additive, it is possible to control the number and dimension of ettringite crystals, which further determines the properties of solutions. In turn, carbonate structures are in close contact with cement stone, which is explained by the occurrence of epitotoxic bonds between the products of cement hydration and wollastonite minerals. Thus, the addition of finely dispersed wollastonite is a chemical factor in increasing the activity of interaction between the active additive and sand. It increases the reactivity of the surface of polymineral additives to cement and therefore activates the hydration process. In the hydration products of composite binders, a redistribution of crystalline phases occurs, both unhydrated clinker minerals (alite, belite, tetracalcium alumoferrite), quartz and calcite, and hydration products (CH – portlandite, $Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$ – ettringite).

During the study of the flexural strength of the developed compositions, the microreinforcing effect of wollastonite (Fig. 11, 12), which has an elongated shape and has increased adhesion to cement stone, was confirmed. However, when the optimal dosage of 4 % is exceeded, a decrease in the strength characteristics of mortar specimens at the age of 3, 7 and 28 days is observed.

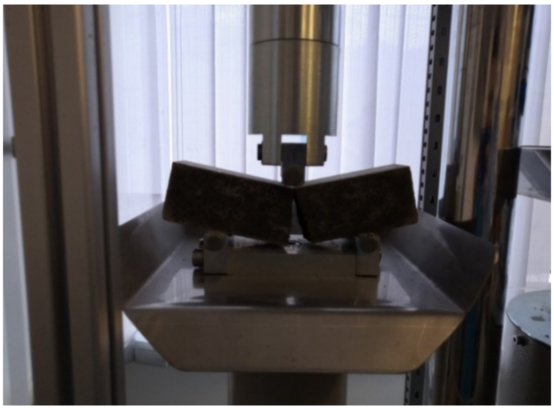


Figure 11. Flexural strength test.

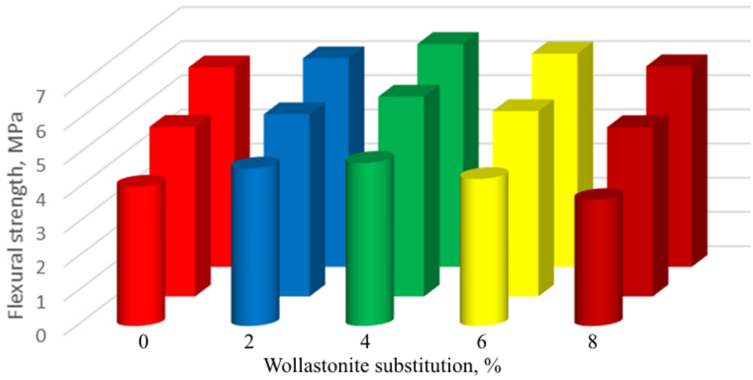


Figure 12. Effect of wollastonite addition on mortar flexural strength (3, 7 and 28 days).

Comparing with articles by other authors who developed mortar with wollastonite [13–17, 24–25], we note that the compressive strength increased by 12–26 %, and the flexural strength – by 24–68 % depending on the dosage of wollastonite.

4. Conclusion

As a result of the study of the physicomechanical characteristics of fine-grained concrete with the replacement of Portland cement with wollastonite in an amount of 2, 4, 6, 8 wt. % revealed the following:

1. Wollastonite in mortar has a dual function: it acts as a filler (silica-containing component) and as microfiber.
2. With an increase in the amount of cement replaced with wollastonite, a decrease in the density of the structural material is observed, which, however, does not adversely affect the strength characteristics. Accordingly, even considering the lower density, there are prerequisites to count on the best characteristics of weather resistance and frost resistance.
3. In the initial period of hardening (days 3 and 7), acceleration of hydration processes was revealed. The compressive strength on days 3 and 7 for all developed compositions with the addition of wollastonite has a higher value than for the control composition.
4. Analyzing the structure formation of cement paste from the standpoint of geomimetics, it was revealed the similarity of wollastonite $CaSiO_3$ with the main minerals of the Portland cement clinker Ca_2SiO_4 (belite) and Ca_3SiO_5 (alite), which makes it possible to obtain a chemically uniform and, accordingly, strong microstructure.
5. The micro-reinforcing effect of wollastonite is ensured by the elongated shape of the microfiber and its good adhesion to the cement paste.

5. Prospects for further development of the topic

It is advisable to consider interdisciplinary approaches to solving the urgent problems of building materials science, for the development of technologies for the production of mortar with additives of natural calcium silicates for a wide range of building composites, including for the development of the Arctic. The algorithm used in the paper can be used in the development of composite binders to expand the range of production of mortar for various purposes, including to improve a comfortable human environment in architectural and construction design and production of mortar composites.

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