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Investigation of physico-chemical processes forming the structure of water-resistant composite materials based on modified gypsum binders

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Abstract: The article presents the results of studies of the physico-chemical processes of forming the structure of the material and the development of effective compositions of modified gypsum-carbonate composite gypsum materials based on gypsum binder, microcalcite and modifying polymer additives, contributing to the improvement of their rheological and physico-mechanical properties and, accordingly, to increase the bending strength of building decorative ceiling suspended gypsum boards based on them. As a result, it became possible to organize the production of ceiling-mounted suspended composite gypsum boards with high water resistance and physico-mechanical properties and low cost, as well as the widespread use of this board in the construction of residential buildings and construction structures in the republic
Keywords: structure, rheological properties, micro filler, microcalcite, superplasticizer, modifying additives, composition, gypsum, ceiling tiles, composition, technology, products.

1. Introduction

Today, the use of modern technologies in the production of industrial products, the production of competitive and environmentally friendly materials obtained using resource- and energy-saving technologies, and the solution of environmental problems are becoming increasingly relevant in the world. Currently, one of the priority tasks of building materials production is to increase the volume of production of building materials based on gypsum materials [1-11]. In this regard, conducting research in the field of physico-chemical modification and technological processes that make it possible to develop effective formulations and optimal production modes for cheap modified composite gypsum materials and building products and improving the environmental situation is of particular importance.

2. The object and methods of research

The object of the study is calcium sulfate semihydrate; a highly dispersed carbonate micronutrient and chemical additives used to reduce the water:binder ratio and increase the water resistance and performance properties of composite gypsum materials. In the dissertation work, the physico-chemical properties and structure of the ingredients were studied by chemical, X-ray, and differential thermal analysis methods. The construction, technical and physico-mechanical properties of the developed modified water-resistant composite gypsum materials were determined by methods in accordance with the requirements of GOST standards.

3. The results of the study and their discussion

For the development of gypsum materials, calcium sulfate semihydrate obtained from natural gypsum stone from the Karnab deposit in the Samarkand region was used by dehydration in a digester, and microcalcite obtained from marble waste by grinding and separation was used as a highly dispersed carbonate filler, and Melment F10 superplasticizer was used as a water-reducing additive.

At the same time, we selected three samples of gypsum binder β-modifications obtained at different temperatures and studied their phase composition, physical and mechanical properties in accordance with GOST 23789-1018.The results of the determination of the chemical and phase composition of the samples are shown in Tables 1 and 2. The table shows that with increasing dehydration temperature, the content of calcium sulfate semihydrate decreases.

Table 1

Phase composition of gypsum binder β-modification

№	Binder number	T-ture of dehydration	Phase composition, mass.%				The amount
			$CaSO_4$ 0,5H_2O	$CaSO_4$	$CaSO_4$2H_2O	impurities	
1	№ 1	180	90	5,6	2,10	2,30	100
2	№ 2	175	93	2,1	2,80	2,10	100
3	№ 3	170	91,7	1,9	4,20	2,20	100

The chemical analysis of gypsum binders obtained by cooking in digesters was carried out in accordance with GOST 23981.1 – 23981.14-79 "Methods of chemical analysis". In terms of the content of semi-aqueous calcium sulfate, the samples meet the requirements of GOST 125-2018 for gypsum binders. The results of the chemical analysis of gypsum binders are shown in Table 2.

Table 2

Chemical composition of the starting materials

№	Material	Content of oxides, mass%								
		SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	R_2O	ППП	The amount
1	gypsum binder №1	1,61	0,38	0,20	37,63	0,62	53,49	0,05	6,02	100,0
2	gypsum binder №2	1,56	0,30	0,20	38,16	0,49	53,64	0,05	5,60	99,96

3	gypsum binder №3	1,62	0,40	0,21	37,66	0,60	53,66	0,05	5,50	99,96

To determine the grade of the selected gypsum binder samples, the normal density, setting time, and strength characteristics were determined. The results of the study are shown in Table 3.

Table 3

Physico-mechanical characteristics of gypsum binder β-modification

Name of the parameters	The value of the parameters		
	Test №1	Test №2	Test №3
Degree of grinding:the remainder on the sieve No. 02,%	5,2	5,1	5,2
Water/astringent ratio	0,59	0,60	0,60
Setting time, min–seconds – start – the end	8–00 14–30	8,0 11-30	6-30 9,30
Compressive strength, aged 2 hours, MPa	5,7	6,2	5,8
Flexural strength, MPa	2,9	3,2	3,0
Compressive strength, dry, MPa	12,8	12,9	12,6
Flexural strength, dry, MPa	5,6	5,7	5,6

The results of physico-mechanical studies have shown that calcium sulfate semihydrate has a G-6 II grade and a water resistance coefficient of 0.49.

The highly dispersed filler was obtained by grinding carbonate rock (white marble waste) from the Kitab deposit. The chemical composition is shown in Table 4. On the X-ray of a highly dispersed carbonate filler, all the main reflections belong to calcite CaCO3, the presence of which is confirmed by the d/n lines. = 0,383; 0,305; 0,304; 0,281; 0,248; 0,226; 0,207; 0,189; 0,185; 0,162; 0,159 nm.

Table 4

Chemical composition of highly dispersed carbonate micron filler

Content of oxides, mass.%										
SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	MnO	SO_3	R_2O	P_2O_5	ППП	The amount
0,43	0,56	0,45	53,58	0,42	0,05	0,07	0,14	0,06	43,20	98,96

Physico-chemical studies have shown that the structure of carbonate components varies from fine (0.1–10 microns) to medium crystalline (20-30 microns). The main component of carbonate rocks is calcite, but its properties vary significantly depending on the type of rock, and on the other hand, carbonate rocks, even belonging to the same class of minerals, can differ significantly in their structure.

During the experimental studies on the preparation of gypsum-carbonate compositions, nine compositions of a gypsum-carbonate mixture were prepared (Table 5).

Table 5

The composition of the mixture for the production of gypsum-carbonate composite material

№	Components	Ratio of components, mass. %								
		1	2	3	4	5	6	7	8	9
1	Gypsum binder	100	97,5	95	92,5	90,0	87,5	85	82,5	80
2	Microcalcite	-	2,5	5,0	7,5	10,0	2,5	15	17,5	20

The results of the study showed that during the homogenization of components in a ball mill, the residue on the sieve No. 02 (900 rem/cm2) decreases sharply with increasing mixing time.

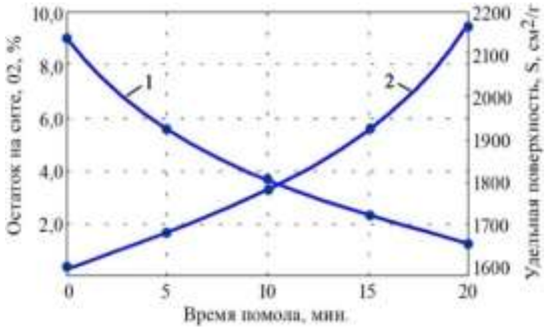

Fig. 1. Dependence of the fineness of the grinding (1) and the specific surface area of the gypsum composition (2) on the grinding time

Based on the conducted studies, it was found that mixing of the starting materials in a laboratory ball mill leads to mechanical destruction of the components and elements of their structure, partially disperses the grains of calcium carbonate. Due to this, additional grinding of calcium sulfate semihydrate took place, which led to an increase in the fineness of the grinding and the specific surface area of the gypsum-carbonate composition. This contributes to a significant increase in the number of active centers per unit volume of the material. This releases a significant amount of energy, which helps to increase the activity of the highly dispersed micro-filler.

Based on the conducted studies, it was found that the specific surface area of the composition increases with fine grinding of the gypsum-carbonate composition. The phase composition of the gypsum-carbonate composition was studied using X-ray phase analysis. The diffractogram of the gypsum-carbonate composition is shown in Figure 2.

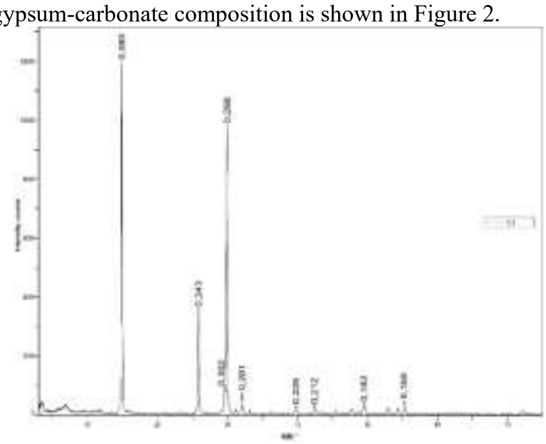

Fig. 2. Diffractogram of a gypsum-carbonate composition consisting of 80% calcium sulfate dihydrate and 20% highly dispersed carbonate filler microcalcite

It can be seen from the diffractogram (Fig.2) that the main phase is calcium sulfate semihydrate ((α/n = 0,590; 0,343; 0,3297; 0,278; 0,166 nm) and calcium carbonate (d/n= 0,281; 0,248; 0,226; 0,1853; 0,162; 0,159 nm).

Next, we will consider the properties of the developed composite gypsum materials obtained by carefully mixing gypsum binder and finely dispersed microcalcite. Table 6 shows the results of studies of rheological and physico-mechanical properties of gypsum-carbonate composite materials.

Table 6

Influence of highly dispersed filler on rheological and physico-mechanical properties of gypsum-carbonate composite

№	Composition, mass. %		Normal density	Setting time, min-sec		Strength, MPa	
	gypsum	micro calcite		start	the end	$R_{из}$	$R_{сж}$
1	100,	-	0,61	4-00	6-00	3,0	6,1
2	97,,5	2,5	0,61	4-00	6-00	3,1	6,4
3	95,0	5,0	0,62	4-30	6-30	3,3	6,9
4	92,5	7,5	0,62	5-30	7-00	3,5	7,5
5	90,0	10,0	0,63	6-00	8-00	3,6	7,6
6	87,5	12,5	0,64	7-00	9-00	3,7	7,6
7	85,0	15,0	0,65	7-00	9-00	3,9	6,0
8	82,5	17,5	0,67	7-00	10-00	4.0	5,8
9	80,0	20,0	0,67	8-00	11-00	4,0	5,6

Laboratory studies of the physico-mechanical properties of the gypsum carbonate composition have shown that a finely dispersed carbonate filler in the amount of 10-15% acts as a plasticizer. With a higher degree of filling (20%), the water consumption of the binder increases slightly, and the strength characteristics of the gypsum-carbonate composition improve by 15-18%. The increase in strength is explained by the fact that highly dispersed microcalcite particles fill the voids between coarser grains during the formation of the calcium sulfate dihydrate structure.

The use of fast-setting composite gypsum materials in construction is largely due to a combination of positive properties. First of all, it is the absence of shrinkage deformations, high adhesive strength and water resistance. As shown by the results of studies on the use of gypsum-carbonate compositions for the manufacture of building finishing products, the structure of calcium sulfate dihydrate is formed during the hydration process for 120 minutes.

Further, to improve the physico-chemical properties and water resistance of sulfate-containing composite modified gypsum-carbonate materials, we used a polymer additive - superplasticizer Melment F10. The mixtures were prepared by mixing a polymer additive with a gypsum-carbonate composition in a laboratory mixer for 15 minutes. The superplasticizer content was 0.0; 0.25; 0.50: 0.75 and 1.0% by weight of gypsum binder. In the prepared mixtures, the normal density was determined according to GOST u 23789-2018.

From the data obtained in Table 7, it can be seen that with an increase in the content of the superplasticizer additive to 0.75%, the water consumption of the gypsum-carbonate composition decreases sharply. At the same time, the softening coefficient of the gypsum-carbonate composition was estimated by the water resistance of standard samples measuring 4x4x16 cm made on the basis of a mortar mixture of normal gypsum material density, and it ranges from 0.59 to 0.74 with a content of SP "Melment F10" from 0.0 to 1.0%.

Table 7 shows the technological characteristics of the modified composite gypsum materials.

Table 7

The effect of the Melment F10 joint venture on the technological characteristics of the gypsum-carbonate composition

№	Content Joint Venture Melment F10, %	В/Т	Setting time, min		Softening coefficient (Cr)
			start	the end	
	0,0	0,61	4-00	6-00	0,59
	0,25	0,51	4-00	6-00	0,62
	0,50	0,42	5-00	7-00	0,66
	0,75	0,40	5-00	8-00	0,73
	1,0	0,37	6-00	8-00	0,74

The study found that reducing the water gypsum ratio from 0.7 to 0.4 makes it possible to increase the strength of gypsum products by 2.5-3.0 times. The compressive and flexural strength of the hardened gypsum binder and products made from it largely depends on their moisture content. In particular, even sorption humidification to 0.5-1% of a dry gypsum sample in air with a relative water vapor content of 80-100% reduces its strength to 60-70% of the strength in the dried state.

It has been established that the plasticizing effect in solution mixtures makes it possible to reduce the water-gypsum ratio (V/G) while maintaining the required mobility, which can be explained by the action of electrostatic and spatial stabilization.

Thus, based on the conducted studies of the superplasticizer on the physico-mechanical properties of the gypsum-carbonate composition, it was found that the strength characteristics increase. The introduction of water-reducing additives into the gypsum-carbonate composition helps to increase the bending strength and increase the water resistance of products made on the basis of a modified gypsum-carbonate composition.

4. Conclusion

Based on the conducted research, it has been established that the principle of action of the superplasticizer is to create conditions for the formation of waterproof compounds, the formation of a more dense structure of the solidified material in the system with a low water content.

Thus, the effectiveness of increasing water resistance and improving the strength characteristics of composite gypsum materials by modification using highly dispersed fillers and a polymer modifier has been scientifically substantiated and experimentally confirmed.

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