Water-resistant fiber-reinforced gypsum cement-pozzolan composite

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Abstract. Gypsum and gypsum-cement-pozzolan composite are of significant interest as materials and products for building decoration. The current tendency to reduce the consumption of gypsum-based materials and products in the context of growth and development of the finishing materials market depends on the decrease in their competitiveness compared to peers. This leads to significant interest in improving the quality of products based on gypsum and gypsum-cement-pozzolan binder. Dispersed reinforcement is one of the ways of improving performance characteristics. The role of the type of reinforcing fibers in the formation of the gypsum-cement-pozzolan composite structure and properties is studied in article. The influence relations of the cellulose fibers content with varying grinding degrees on the relative flexural and compressive strength of a gypsum-cement-pozzolan matrix are obtained. It was found that the optimal content of cellulose fibers in the modified gypsum-cement-pozzolan matrix is 0.5-1% by weight of the binder, the best indicators of flexural and compressive strength are achieved by grinding cellulose fibers to 30°SR. Using scanning electron microscopy, it was found that the microstructure of a disperse-reinforced gypsum-cement-pozzolan matrix is characterized by a uniform cellulose fibers distribution, the predominance of directionally frame reinforcement with a fibers linear orientation in the sheet plane. In this way the right choice of the type, crushing degree and cellulose fibers content can improve the quality and expand the application area of the produced gypsum-cement-pozzolan composite.

1 Introduction

Nowadays, industrial and civil buildings construction rates and structures are at a fast pace. Various materials and products, such as fiber cement boards, dry building mixes, gypsum plasterboard and gypsum fiber board, etc, are used for interior decoration of newly constructed and reconstructed buildings and structures. Products based on gypsum binder, which has a developed mineral resource base, low cost and environmental friendliness of production are of a particular interest. However, it should be noted that there is a tendency

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to reduce the consumption of dispersively reinforced gypsum composites. An analysis of finishing materials market revealed that the consumption of these products since 2006 has decreased by 43%, which in our opinion is due to the absence of gypsum-based sheet construction products in the market with high performance properties. At the same time, market of finishing materials, including sheet materials grows and develops.

So a significant interest in improving the quality and volume of manufactured and consumed products based on gypsum binder has quickened. In this regard, at the first stage of research the reinforcing fibers type role in the structure and properties formation of gypsum-cement-pozzolan composite, as one of the ways to increase their effectiveness, was studied. At the second stage, experimental studies of the cellulose fibers effect on the properties of the gypsum-cement-pozzolan matrix (GCPM) were performed. At the third stage, the microstructure formation features of the dispersed-reinforced GCPM are studied.

To eliminate the disadvantages of thin-walled sheet products based on cement, gypsum and mixed binders - low bending strength, significant fragility, high shrinkage / swelling deformations, various reinforcing materials are introduced into their composition [1–5]. The efficiency of reinforcement depends on both the type of reinforcing fibers and their orientation in the volume of the material [6,7], and the matrix enveloping material. The work [8–11] shows the experience of using polypropylene and cellulose reinforcing fibers for gypsum building materials.

The matrix in the reinforced composite materials gives the product necessary shape, creates a monolithic material and combining numerous fibers into a single unit that allows the composition to absorb various kinds of external loads. In addition, the matrix takes part in creating the load-bearing capacity of the composite material by ensuring the transfer of forces to the fibers. To select fibers for dispersed reinforcement it is necessary to take into account not only manufacturability, strength, economy, but also durability, which is closely related to the fibers resistance in the matrix and their adhesion to it. The relative tensile strength of the fibers increases with their diameter decreasing, so the minimum thickness fibers are used for reinforcement.

According to the classification of I.I. Burney, there are two main varieties of sheet products dispersed reinforcement: with a scattered or associated arrangement of fibers., the fibers with a scattered arrangement are located at such distance, which let them function independently. In sheet products with an associated arrangement of fibers, adhesion between them in the contact zones, as a result of the binder hardening, creates the conditions for the joint function of the fibers in material. Scattered reinforcement can go into associated one by increasing length and number of fibers per volume unit.

Depending on the orientation of the fibers, materials can have several types of reinforcement. There are some types of the reinforcement with the scattered arrangement of fibers, this is volumetric, directional-volumetric, planar and directional-planar reinforcement, and types of reinforcement with an associated arrangement of fibers are: frame, directional-frame, mesh and directional-mesh reinforcement. There is no definite fibers orientation for volume and frame types of reinforcement. The fibers in planar or mesh reinforcement are oriented in parallel planes. If the reinforcing fibers become oriented in any direction, type of reinforcement will be called directional.

The types of reinforcement indicated in this classification are not found in their pure form. As a rule, the planar orientation of the fibers is imperfect, and the direction of the fibers is not strictly maintained. Therefore, considering the structure of dispersed reinforced material we can only speak about the predominance of some reinforcement kinds.

Technical characteristics of the various fiber types, which were considered as dispersed reinforcement of gypsum-cement-pozzolan systems, are shown in Table 1.

Table 1. Technical characteristics of fibers for dispersed reinforcement of gypsum-cement-pozzolanic systems

Type of fiber	Density, g / cm3	Tensile strength, MPa	Modulus of elasticity, MPa
Polypropylene	0,9	400-770	3500-8000
Polyethylene	0,95	700	1400-4200
Nylon	1,1	770-840	4200
Acrylic	1,1	210-420	2100
Polyester	1,4	730-780	8400
Asbestine	2,6	910-3100	68000
Cellulose	1,2	300-500	10000
Glass	2,6	3850	75000
Carbon	2,0	2000	245000
Carbon	1,63	7800	380000
Polyamide	0,9	720	1900
Viscose	1,2	660	5600
Basalt	2,6	1600-3600	100000
Wollastonite	2,9	200-400	10000
Kevlar	1,45	3600	150000
Polyacrylonitrile	1,2	900	20000

Considering these types of fibers, it should be noted, that nylon, polyamide, carbon and kevlar fibers have many positive properties, such as elasticity, wear resistance, high strength, but these types of fibers have a high cost [12]. In this regard, using these fibers for the dispersed reinforcement of GCPM will lead to a significant increase in the cost of finished products.

Acrylic and polyacrylonitrile fibers are quite cheap to manufacture, but they have a tendency to be rolled and clumped, that complicates their using as dispersion reinforcing material due to the impossibility of their uniform distribution in a gypsum cement matrix.

The effectiveness of wollastonite as a reinforcing component has been proven for the material cement matrix. However, there is no information about this component's effect on the structure and properties of the gypsum-cement-pozzolanic binder (GPCB). It is known that the surface of wollastonite is hydrolyzed in case of contact with water, forming calcium hydroxide. It can be expected that the addition of wollastonite to GCPB apart from the micro-reinforcing effect will lead to an increase in the alkalinity of the liquid phase and to the creation of conditions for the formation of highly basic calcium hydrosulfoaluminates.

The use of basalt and glass fibers for reinforcing GCPM is limited due to the available information [13–15] about their low resistance to chemical corrosion, including the environment of gypsum-cement-pozzolanic stone. Despite their good properties using of asbestos fibers in special activities is not investigated in the article because of a decrease in demand for products containing asbestos fibers, which is associated with conflicting information about their carcinogenicity.

The high specific surface of carbon fibers [16] leads to their conglomeration in the process of dispersed reinforced products' manufacturing and to the irregular allocation in the volume of the material. Using of ultrasonic dispersants for the preparation of carbon fibers suspensions in the environment of surfactants allows to level this disadvantage. However, this complicates the technology of preparing a gypsum-fiber mixture based on dispersed carbon fibers.

Polypropylene fibers are chemically inert, fiber molecules do not contain reaction groups. In this regard, these fibers are not subject to chemical corrosion [12,17]. This type of fiber is hydrophobic, which positively affects the water resistance of products reinforced with polypropylene fibers. The advantage of this fiber is also in its low cost compared to analogues. However, this type of fiber has low fire resistance (it begins to soften at 140°C, its melting point is 175°C).

Cellulose fiber is a traditionally used material for products dispersed reinforcement based on gypsum-cement-pozzolanic binder. Cellulose fibers are biopositive, have high adhesive, in particular cement-retaining, and strength properties [18]. However, in the literature there are no experimental studies of the cellulose fibers' degree effect of grinding on the physicomechanical properties of products based on low-quality gypsum binder with a decreased Portland cement content under their modification with of chemicals. In addition, the type of dispersed reinforcement of products whose molding is performed using the foundry method has not been studied.

Based on the analysis of various types of fibers that we considered for dispersed reinforcement of GCPM based on low-quality GCPB, cellulose fibers are adopted for further studies.

High technical indices, chemical resistance, and low mass of cellulose fibers cause considerable interest in studying them as the dispersion-reinforcing material of a gypsum-cement-pozzolanic matrix. In addition, home industry has high production volumes of this type of fiber. It determines the need of studying the formation features of the gypsum-cement-pozzolanic matrix structure and properties reinforced with this fiber type.

2 Methods

For the preparation of GCPB were used:

- building gypsum brand G6BII manufactured by LLC "Arakchinsky gypsum";
- Portland cement grade PC500-D0-N from Belgorod cement plant;
- mineral supplement - metakaolin with hydraulic activity of 1238 mg/g, selected from a wide range of natural and man-made AMD, taking into account previous studies [19–21].

The ratio of components in the composition of the composite binder was - gypsum: PC: AMD - 76: 20: 4 parts by weight.

To prepare the developed multifunctional complex additive (CD) [22–24] the Odolit-K SE produced by Service-Group LLC (TU 5745-01-96326574-08); The Best-TB joint venture manufactured by Innovative Technologies LLC, the hydrophobizing additive Ethylsilicate-40 produced by Khimprom OJSC (TU 2435-427-05763441-2004) were used.

Coniferous sulphate unbleached pulp NSK-0, produced by OJSC Solombalsky Pulp and Paper Mill according to STO 00279189-2-2007, previously fluffed up to 20, 30, 50° SR, was used as a fibrous material.

At the first stage the influence of the grinding degree and the content of cellulose fibers on the strengths under bending of GCPM based on low-grade GB (gypsum binder) with a low content of PC was studied. The amount of mixing water was selected so, that the gypsum-cement-fiber mixture reached the ND (normal density). At the second stage, the microstructure of the cellulose fibers reinforced and a modified developed multifunctional CA gypsum-cement matrix was studied.

Strength tests in bending of gypsum fiber boards samples were carried out on models of 400x300x10 mm according to the method of GOST R 51829-2001 "Gypsum fiber sheets. Technical conditions".

Optical studies of the obtained GCPM models were carried out using a Philips XL-30 scanning electron microscope. Optical studies were carried out in order to study the

morphology of hydrated neoplasms, the structure surface of the investigated gypsum-cement compositions and the changes occurring in them, as well as the features of the distribution of fibers in the matrix.

3 Results

As you know, during the dissolution of cellulose fibers, they are grinded, this process affects both the physicomachanical characteristics of the fibers and the properties of dispersively reinforced products based on them. The strength of cellulose fibers is characterized by their breaking length. Modification of the GPCB of the developed multifunctional CA studied composition makes it possible to significantly increase the flexural strengths (by 61.9%), water resistance (up to 0.93), and frost resistance (up to 100 cycles) of GCPM. In this work, we studied the dispersed reinforcement of the developed modified matrix with cellulose fibers. The results of grinding degree effect of experimental studies and the content of cellulose fibers on the relative flexural strength and relative compressive strength of GCPM are given in tables 2 and 3, respectively.

Table 2. The grinding degree effect and the content of cellulose fibers on the relative tensile strength in bending GCPM

The content of cellulose fibers in the composition of GCPM, % by weight of binder	The relative flexural strength of the GCPM (%) at the degree of grinding of cellulose fibers (°SR)		
	20	30	50
0	100	100	100
1	72,1	100,5	57,7
2	36,8	69,6	28,2
3	26,3	41,3	20,1
4	19,5	33,6	14,6
5	16,1	20,9	13,5

The dependences of the effect of the content of cellulose fibers on the relative flexural strength of the GCPM at different degrees of grinding are described by polynomials of the third degree of the following form:

$R_{flex}(20^{\circ}SR) = -0,0861x^3 + 4,869x^2 - 39,09x + 101,45;$ (1)

$R_{flex}(30^{\circ}SR) = 1,7222x^3 - 12,438x^2 + 3,3587x + 102,02;$ (2)

$R_{flex}(50^{\circ}SR) = -0,9111x^3 + 12,226x^2 - 55,729x + 100,43.$ (3)

Table 3. The effect of the degree of grinding and the content of cellulose fibers on the relative compressive strength

The content of cellulose fibers in the composition of GCPM, % by weight of binder	The relative compressive strength of the GCPM (%) at the degree of grinding of cellulose fibers (°SR)		
	20	30	50
0	100	100	100
1	72,9	92,9	63,4
2	36,6	55,1	27,5
3	22,6	35,3	19,1
4	15,5	19,5	12,9
5	9,9	10,9	8,6

The dependences of the cellulose fibers content effect on the relative compressive strength of the GCPM at different degrees of grinding are described by polynomials of the third degree of the following form:

$$R_{\text{comp}}(20^\circ\text{SR}) = 0,0679x^3 + 3,4963x^2 - 37,289x + 101,54; \quad (4)$$

$$R_{\text{comp}}(30^\circ\text{SR}) = 1,3657x^3 - 8,8056x^2 - 8,0626x + 101,94; \quad (5)$$

$$R_{\text{comp}}(50^\circ \text{ SR}) = -0,6472x^3 + 9,8595x^2 - 51,519x + 101,27. \quad (6)$$

The analysis of dependences 1-6 shows that cellulose fibers with different degrees of grinding have a significant impact on the flexural and compressive strength of GCPM. The optimal coefficient of gypsum-cement-pozzolanic matrix fiber reinforcement with cellulose fibers according to the criterion for achieving the required tensile strength in bending in accordance with GOST R 51829-2001 comprises 0.5-1% by weight of the binder. A further increase in the content of cellulose fibers in the mixture leads to a significant decrease in strength indicators, which, in our opinion, is associated with an increase in the mixture water demand, clumping of fibers and their irregular distribution in the gypsum-cement-pozzolanic matrix. The best indicators of tensile strengths in bending and compression are achieved when grinding cellulose fibers are to 30 ° SR. So, when the fiber content in the amount of 1% by weight of the binder, the flexural strengths of the GCPM reinforced by cellulose fibers with a grinding degree of 30 ° SR are 47.3% and 28.4% higher than that of the similar compositions reinforced by fibers with a grinding degree of 50 ° SR and 20 ° SR, respectively. Comparable results in the study of the disperse-reinforced GCPM compressive strengths were obtained. A further increase in the grinding degree, according to the obtained experimental information, leads to a decrease in the strength characteristics of the finished products, which our opinion depends on the fact that the damage cellulose fiber degree increases disproportionately to the increase in bond strength.

It is of considerable interest in this case is the study of the microstructure formation features of the modified gypsum-cement matrix dispersed-reinforced by cellulose fibers with an optimal degree of grinding.

The microstructure of the gypsum cement matrix reinforced by cellulose fibers modified by the developed multifunctional CA using scanning electron microscopy was studied. Electronic images of the samples are presented in figures 1-3.

Fig. 1. Electron-microscopic image of GCPM, dispersively-reinforced by cellulose fibers, at a magnification in 100 times

Fig. 2. Electron-microscopic image of GCPM, dispersively-reinforced by cellulose fibers, at a magnification in 1000 times

Fig. 3. Electron-microscopic image of GCPM, dispersively-reinforced by cellulose fibers, at a magnification in 5000 times

As it follows from the electron microscopic images, the structure of the dispersed-reinforced GCPM is a strand of thin cellulose fibers that are connected in a monolith by the hydration products of GCPB. The presence of cellulose fibers determines the strength and rigidity of the conglomerate, the binder protects the fiber from aggressive environmental influences, provides interaction between the fibers during mechanical stresses. Fig. 1 shows the general picture of the dispersion-reinforced GCPM structure at the fracture site. The structure is characterized by a relatively uniform, chaotic distribution of cellulose fibers. The fibers are predominantly parallel to the molding plane, this explains the increased flexural strength. Over a magnification of 5000 times (Fig. 3) clusters of shapeless submicrocrystalline structures are visible, these structures are low-basic calcium hydrosulfoaluminates, characterized by a high degree of amorphism.

At magnification in 100 (Fig. 2) the areas of contact between the reinforcing fibers are clearly visible, therefore, it is possible to draw a clear conclusion about the associated location of the fibers. At the same time, when considering the structure of dispersed-reinforced GCPM, the location and orientation of the fibers in different planes are observed. Thus, it is possible to draw a conclusion about the absence of directional mesh reinforcement with linear-planar orientation and a mesh reinforcement with in-plane orientation. Next, the frame reinforcement, which includes the materials under consideration will be observed. Electron microscopic images show that the linear orientation of the fibers in the plane of the sheet prevails. Thus, according to the type of reinforcement of dispersed-reinforced GCPM directional-frame reinforcement with linear orientation of the fibers in the plane of the sheet, which indicates a uniform distribution of

fibers in the matrix and causes increased physical and mechanical characteristics of the finished products prevails.

With a magnification in 100 times (Fig. 2) the contact zones between the reinforcing fibers are clearly visible, therefore, we can clearly conclude that the fibers are connected. In this case, when considering the structure of the dispersion-reinforced GCPM, the arrangement and orientation of the fibers in different planes are observed. Thus, we can conclude that there is no directional-mesh reinforcement with linear-planar orientation and mesh reinforcement with a planar orientation. Next, we consider the frame reinforcement, to which the studying materials belong. Electronic microscopic images show that the linear orientation of the fibers in the plane of the sheet prevails. Thus, by the type of reinforcement of the disperse-reinforced GCPM, the directional-frame reinforcement with a linear orientation of the fibers in the plane of the sheet predominates, which indicates a uniform distribution of fibers in the matrix and causes increased physical and mechanical characteristics of the finished products.

The performed studies show a positive effect of the studied fibers type on the structure and properties of the modified GCPM. Thus, cellulose fibers right type choice, degree of clumping and their content in matrix will improve the quality of the products based on gypsum binder and expand their application field.

4 Conclusions

1. The analysis of literature data indicates the crucial role of reinforcing fibers in the formation of the composite materials structure and properties, a correct choice of fibers can significantly improve the physical and mechanical characteristics of finished products. A review of the experience with the use of fibers currently available on the construction market and suitable for dispersed reinforcement of gypsum-cement compositions made it possible to single out cellulose ones as one of the most effective types of reinforcing component for GCPM.

2. The performed experimental studies made it possible to determine the optimal content and degree of cellulose fibers grinding in the composition of the modified GCPM. The best indicators of tensile strengths when using cellulose fibers are achieved when they are contained in the mixture in an amount of 0.5-1% by weight of the binder and the degree of grinding of 30°SR. When the fiber content in the amount of 1% by weight of the binder, the bending strengths of GCPM reinforced with cellulose fibers with a grinding degree of 30°SR are 47.3% and 28.4% are higher than similar compositions reinforced with fibers with a grinding degree of 50°SR and 20°SR, respectively.

3. The microstructure of the dispersion-reinforced GCPM is characterized by a uniform distribution of cellulose fibers, the predominance of directionally frame reinforcement with a linear orientation of the fibers in the sheet plane, which leads to increased physical and mechanical characteristics of the finished products and confirms the optimal content of cellulose fibers in the GCPM.

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