DRY PACK MORTARS FOR SELF-LEVELLING FLOOR COMPOUNDS BASED ON Β-HEMIHYDRATE AND MODIFIED PHOSPHOGYPSUM BINDER

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Abstract

The paper considers the influence of β-hemihydrate and phosphogypsum binder on the properties of dry pack mortars for self-levelling floors. As preliminary research demonstrated that adding to phosphogypsum (PG) admixtures of lime and PA or PC superplasticizer permits to obtain binder with high compressive (up to 24 MPa) and bending strength (up to 12 MPa). The manufacturing of dry pack mortars for self-levelling floor underlayment was determined as one of the areas of PG application. There has been investigated composite binder consisted of PG, Portland cement and metakaolin.

There have been determined linear deformations of PG/ based binder comparing to β-hemihydrate calcium sulphate binder and bending to compressive strength ratio.

The method of planned experiment was applied for determination the optimal proportion of the components of the mortar. There were considered the sand / binder ratio, cement and metakaolin portions in binder, water-binder ratio and content of Tylose (water-retaining admixture). There was tested composite binder consisted of PG (or β-hemihydrate), PC and metakaolin. There were tested flow spread on Suttard's viscometer, time of application, and compressive strength at the age of 1, 3 and 28 days. The flow spread diameter of the mixtures was within the range of 180-200 mm, and time of application was between 30 and 45 min.

The compressive strength of mortar specimens is up to 4 MPa, at the age of 3 days is 10 MPa and at the age of 28 days reaches 17 MPa. The increasing of Portland cement dosage and metakaolin makes the most positive effect on strength parameters.

The mortar based on PG is applicable for underlayment of different flooring compounds types.

Keywords:

β-hemihydrate; phosphogypsum; self-levelling floor compound; dry pack mortar

1 INTRODUCTION

The gypsum containing by-products of chemical industry serve as alternative raw materials for calcium sulphate binders' production [Dvorkin 2008, Khatib 2013, Rashad 2017, Dvorkin 2013]. According to the available data the estimated annual world generation of phosphogypsum as one of the most common chemical industry by-products, varies from 160-170 Mt [Haschke, 2016] to 200-280 Mt [Saadaoui 2017].

Regardless the fact that phosphogypsum is the most widespread calcium sulphate-containing by-products in Ukraine, it is not common in recycling. The main obstacles in wide application of PG are the need in preliminary treatment of by-products (neutralization, mineralization), high water consumption and low performance properties of the binder.

Along with high content of calcium sulphate (within the range of 80…98%), the need in neutralization of harmful admixtures in by-products increases energy consumption for manufacturing binders [Lushnikova 2016, Dvorkin 2017].

The low performance characteristics of such binders can be eliminated due to application of chemical and mineral admixtures. Preliminary researches determine the optimal parameters for binder manufacturing [Dvorkin 2017]. There have been determined the optimal parameters for manufacturing PG binder: calcination temperature from 150 to 160^{0}C, grinding fineness up to 5% residue on 02 sieve and lime as neutralizing admixture content 2 to 2.5%.

For W/G ratio reduction and increasing mechanical properties of PG binder polycarboxylate ethers-based superplasticizers (SP) are effective due to the dominance of steric effect. To create alkaline environment and involve electrostatic effect of SP the enough lime is about 2 to 2.5% by gypsum m mass comparing to 0.5 to 0.6% of SP is required [Dvorkin 2009, Marschetzky 2013, Dvorkin 2017].

There are suggested several main areas of application of PG binders [Lushnikova 2016, Dvorkin 2018]. One of the known is dry pack mortars manufacturing.

2 AIM AND SCOPE OF RESEARCH

The main aim of current research was revealing the potential of PG based dry-pack mortars as self-levelling floor comparing to β-hemihydrate based mortars.

The scope of the research is determination the properties of both natural gypsum-based and PG based binders (drying shrinkage and bending/compressive strength ratio) and determination the properties of floor self-levelling mortars.

3 MATERIALS AND METHODS

3.1 Materials

Aged (more than 20 years) dump phoshorgypsum from Rivne fertilizing plant was used in this investigation (Ukraine) (see Tab.1).

Tab. 1: Chemical composition of phosphogypsum (% by mass).

Component, %	Phosphogypsum
SiO_2	-
Al_2O_3	0.34
Fe_2O_3	0.16
CaO	38.30
MgO	0.004
SO_3	59.1
P_2O_5 total	0.69
P_2O_5 water soluble	0.04
F^-	0.14
Cl^-	0.01
L.O.I.	–

There is observed prevalence of grains size 0.1 to 0.4 mm, 0.05 to 0.1 mm and less. It is purified with rains and thawed snow in dumps. The content of water soluble P_2O_5 at the age of 20 years is at least 10 times lower compared to the fresh one [Dvorkin 2017]. The radioactivity of PG does not exceed 370 Bq/kg. Dump PG was ground with subsequent neutralization of acid admixtures.PG meets the following Ukrainian standard specifications:
- calcium sulphate content is less than 90 %;
- total amount of phosphates content (in terms of P_2O_5) is less than 1.5 %;
- water soluble phosphates content (in terms of P_2O_5) is less than 0.15 %;
- fluorides content (in terms of F) is less than 0.4 %.

As an alkaline admixture slake lime with 87% activity (CaO+MgO content) was used.

The properties of superplasticizer (SP) used are shown in Tab. 2.

Tab. 2: Properties of superplasticizer.

Name (trade name)	Melflux 1641F
Manufacturer	BASF Construction Polymers, Trostberg, Germany
Chemical base	modified polycarboxylic ether
Average water reduction (for cement-based concrete), %	up to 50%
Average strength increasing at water-to-binder ratio decreasing (for cement-based concrete), %	up to 60%
Dosage recommendation, % by cement weight	0.05…1.5 (for alumina cement) 0.05…0.5 (for ordinary PC)
Physical shape	powder
Density, g/cm^3	-
Dry content, %	-
Bulk density, kg/m^3	400…600
Colour	yellowish
pH value (solution)	6.5…8.5

For comparing the properties there was used β-hemihydrate calcium sulphate binder with compressive strength at the age of 2h 5 MPa.

As the components of drypack floor mortars following components were applied: Portland cement CEM I 52,5 EN-196 (Tab. 3.), metakaolin, calcined at 700^{0}C as active mineral admixture (Tab. 4 and 5), Tylose as water-retaining admixture and citric acid as setting time retarder.

Tab. 3. Physical-mechanical properties of cement.

No.	Property, units	Value
1	Fineness (008 sieve fraction content), %	5
2	Specific surface by Blaine, cm^2/g	3,300
3	Normal consistency, %	24%
4	Hardening - begins at - ends at	 1 h 35 min 3 h 45 min
5	Uniformity of volume change	Meets the requirements
6	Ultimate strength at 28 days, MPa: - flexural - compressive	 8.75 54.0
7	Gypsum content (SO_3), %	3.5

Table 4. Chemical composition of metakaolin, % by weight.

SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	MnO	Na_2O	K_2O	L.O.I.
52.5	42.2	0.34	0.70	0.30	0.25	0.01	0.10	0.90	0.50

Table 5. Physical and chemical properties of metakaolin.

No.	Property, units	Value
1	Specific surface by Blaine, cm^2/g	18,000
2	Density, g/cm^3	2.50
3	Bulk density, kg/m^3	350
4	Pozzolanic activity, mg/g (by CaO absorption)	25
5	Normal consistency of metakaolin paste, %	46

3.2 Methods

At the first stage of the research there have been investigated properties of PG and β-hemihydrate based binders.

There was determined linear deformations (ASTM C596-18) at the specimens of gypsum binder at the specimens 40×40×160 mm and bending ad compressive strength ratio. Gypsum pastes had normal consistency measured by Suttard cylinder. There were used standard physical methods ASTM C472 - 99(2014) for testing binders and drypack mortars.

At the second stage, there have been investigated the influence of modifying admixtures and proportion of the composition on flow spread diameter of Suttard cylinder, retention time, bending and compressive strength of mortars. For each composition 3 specimens were tested.

Dry pack mortars compositions have been prepared by mixing in laboratory ball mill.

There have been applied methods of statistical modelling for planning the experiments and description the results [Montgomery 2000, Sonebi 2004, Dvorkin 2012]. For solving technological problems, second order polynomial regression equations were applied as follows:

$$y = b_0 + \sum_{i=1}^{k} b_i x_i + \sum_{i=1}^{k} b_{ij} x_i x_j + \sum_{i=1}^{k} b_{ii} x_i^2 \quad (1)$$

where

y is the output parameter value;

b_0, b_i, b_{ij}, b_{ii} are the regression coefficients;

x_i, x_j, are the investigated factors;

k is the number of factors.

4 RESULTS AND DISCUSSION

4.1 Properties of β-hemihydrate and PG binders

Compositions of binders and water -gypsum ratio (W/G) to achieve normal consistency are shown In Tab 6.

There are have been determined linear deformations at the stages of hydration and hardening up to 15 days (Fig. 1).

The proportions of binder and admixtures have been determined by preliminary researches [Dvorkin 2013, Dvorkin 2017].

Table 6. Composition of the binder.

#	Composition of the binder, % by weight				W/G
	β-hemihydrate	PG	SP	Lime	
1	100	-	-	-	0.60
2	97.40	-	2	0,60	0.34
3	-	100	-	-	0.80
4	-	96.40	3	0.60	0.34

As it is known the final volume of $CaSO_4{\cdot}2H_2O$ is significantly lower than total volume of $CaSO_4{\cdot}0.5H_2O$ and $1.5H_2O$. However, the observed expansion at hardening is caused by its recrystallization with transformation of rhombic crystals into monoclinic [Abdel-Aal 2004, Yu 2009]. The expansion starts when firm spatial structure form during intensive crystallization. Dihydrate crystals fixed in the places of contact form framework.

At early stages of hardening when gypsum paste has high plasticity and doesn't have firm structure, shrinkage is mostly caused by the processes of sedimentation.

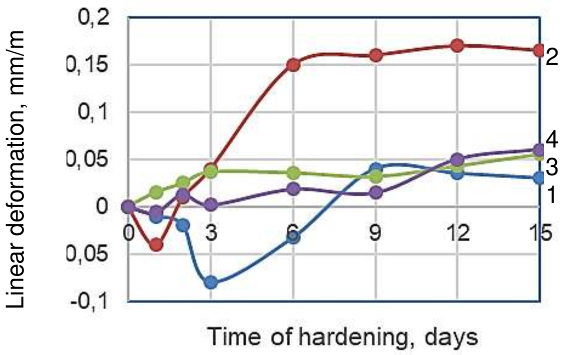

Fig. 1. Linear deformations of the binder specimens (according to Tab. 6)

According to the data obtained for β-hemihydrate binder with SP and lime has low deformations started with low shrinkage in the initial time up to 0.04 mm/m with subsequent expansion up to 0.17 mm/m in late period. Whereas control specimens based on hemihydrate binder has higher level of shrinkage (up to 0.08 mm/m) with subsequent achieving expansion up to 0.03 mm/m at the age of 8-9 days, which meets the known data for β-hemihydrate [Yu 2009, Lushnikova 2016]. The difference between the specimens #1 and 2 is primary caused by higher water consumption of control binder (Tab. 6). The higher expansion of specimens #2 can be explained by earlier time of formation of structural framework and intensive growth of crystals.

Linear deformations of PG based β-hemihydrate specimens has significantly lower ranges and absolute values of deformations comparing to β-hemihydrate (Fig. 1 , specimens #3 and 4). The deviations of the deformations can be explained by the variations of grain distribution of phosphogypsum and presence of impurities and recrystallisation of new formations.

Modified PG specimens with SP and lime deformations are almost equal to those without admixtures and are around 0.05 mm/m. The value insignificantly exceeds expansion for β-hemihydrate specimens.

The low expansion deformations stabilised in 9-12 days of hardening make possible to use such binders for floor mortars as if accordingly to Ukrainian standard

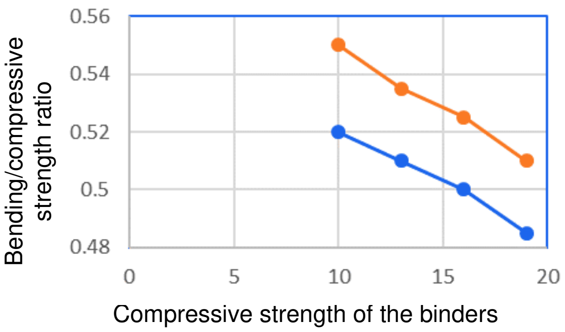

Fig. 2. Dependence of bending-compressive strength ratio on compressive strength of modified binders (# according to Tab. 6)

requirements the deformations of mortars must not exceed 2mm/m [DSTU 2011].

As one of characteristic of structure imperfection of hardened binder is relationship between bending and compressive strength [Rossi 2005] (Fig. 2). In general, the ratios of the both types of binders are similar.

For modified β-hemihydrate binder the ratio is 0.48…0.53, whereas for modified PG binder it is slightly higher (0.51….0.55). It reveals the lower structure imperfection of the hardened PG binder.

4.2 Properties of floor mortars

According to the local standards, compressive strength of the mortars must be 15…35 MPa, flow spread must be more than 170 mm [DBN 2001, DSTU 2011]

There are series of researches devoted to the floor drypack mortars [Barluenga 2010, Zhi 2017, Pereira 2018]. Basic components for them to provide technological and performance properties apart of mineral binders, aggregates and mineral admixtures, are chemical admixtures such as superplasticizers, water-retaining admixtures, setting and hardening retarders and so on.

Modified β-hemihydrate and phoshogypsum binders can be applied for manufacturing self-levelling floors. To provide fast hardening of β-hemihydrate and PG at initial stage and at later time (14...28 days) – structure forming at hardening Portland cement at presence of metakaolin, several admixtures have been selected.

For selection of technological parameters for manufacturing drypack floor mortars three-level five-factors plan Ha_5 [Montgomery 2000, Dvorkin 2012] was used in the experiment. The equation (1) has following form:

$$y=b_0+b_1x_1+b_2x_2+b_3x_3+b_4x_4+b_5x_5+b_{11}x_{11}^2+b_{22}x_{22}^2+b_{33}x_{33}^2+b_{44}x_{44}^2+b_{55}x_{55}^2++b_{12}x_1x_2+b_{13}x_1x_3+b_{14}x_1x_4+b_{15}x_1x_5+b_{23}x_2x_3+b_{24}x_2x_4+b_{25}x_2x_5+b_{34}x_3x_4+b_{35}x_3x_5+b_{45}x_4x_5 \quad (2)$$

Tab. 7 presents the terms of planning the experiments. Matrix of planned experiment and results of testing of the specimens for β-hemihydrate based mortars are shown in Tab. 8.

Table 7. Terms of experiment planning.

Factors designation		Variation levels			Range of variation
Symbolic	Natural	Bottom (-1)	Mean (0)	Upper (+1)	
x_1	Ratio of sand and binder*, by weight (S/B)	0.7	1	1.3	0.3
x_2	Portland cement content, % by binder weight (PC)	5	10	15	5
x_3	Metakaolin content, % by binder weight (MTK)	5	15	25	10
X_4	Tylose content, % by binder weight (T)	0	0.03	0.06	0.03
X_5	Water-binder ratio (W/B)	0.45	0.50	0.55	0.05

- Under binder total amount of PG (β-hemihydrate), Portland cement, metakaolin and Tylose is meant

Regression equations have been obtained for regression equations for the properties. The equations for compressive strength are as following:

$$f_c^{1day} = 4.0 - 0.88x_1 + 0.16x_2 + 0.26x_3 - 0.13x_4 - 0.28x_5 - 0.09x_1^2 - 0{,}08x_2^2 - 0.06x_3^2 + 0.01x_4^2 + 0.24x_5^2 \quad (3)$$

$$f_c^{3days} = 6.77 - 1.47x_1 + 0.26x_2 + 0.44x_3 - 0.22x_4 - 0.47x_5 - 0.15x_1^2 - 0{,}13x_2^2 - 0.11x_3^2 + 0.02x_4^2 + 0.39x_5^2 \quad (4)$$

$$f_c^{28days} = 12{,}53 - 2.77x_1 + 0.44x_2 + 0.85x_3 - 0.37x_4 - 0.9x_5 - 0.28x_1^2 - 0.25x_2^2 - 0.21x_3^2 - 0.03x_4^2 + 0.72x_5^2 \quad (5)$$

Compositions for the matrix (Tab. 8) was calculated per 1l of the mixture taking into consideration $\rho_{binder.} \approx \rho_{sand} \approx$ 2.75 kg/l, where ρ - is real density of gypsum binder Portland cement, metakaolin and Tylose. Set of equations was used:

$$\begin{cases} Water + (Sand + (Binder)/2.75 = 1л \\ Sand/(Binder) = x_1 \\ Water/(Binder) = x_5 \end{cases} \quad (6)$$

Taking the ratios (x_1) and (x_5) from Tab. 8, it is possible to calculate sand, water and binder consumption. Taking into consideration x_2, x_3 i x_4 values it is possible to calculate weight of all components of binder.

Table 8. Matrix of planned experiment and results of tests of mortars for self-levelling floor based on β-hemihydrate binder.

Test no. U	Factors (symbolic)					Factors (natural)					Output parameters				
	x_1	x_2	x_3	x_4	x_5	X_1	X_2	X_3	X_4	X_5	Flow spread diameter, mm	Retention time, min	Compressive strength, at the age of, days, MPa 1	3	28
1	+1	+1	+1	+1	+1	1.3	15	15	0.06	0.55	200	45	3.63	6.13	11.36
2	-1	-1	+1	+1	+1	0.7	5	15	0.06	0.55	205	43	4.42	7.47	13.83
3	-1	+1	-1	-1	-1	0.7	15	5	0	0.45	195	35	5.02	8.44	15.63
4	+1	-1	-1	-1	-1	1.3	5	5	0	0.45	182	32	2.85	4.82	8.92
5	-1	+1	-1	+1	+1	0.7	15	5	0.06	0.55	201	42	4.48	7.57	14.01
6	+1	-1	-1	+1	+1	1.3	5	5	0.06	0.55	202	45	2.31	3.89	7.21
7	+1	+1	+1	-1	-1	1.3	15	15	0	0.45	195	33	4.03	6.76	12.51
8	-1	-1	+1	-1	-1	0.7	5	15	0	0.45	185	32	5.77	10.74	18.04
9	-1	+1	+1	+1	-1	0.7	15	15	0.06	0.45	198	41	5.42	9.12	16.88
10	+1	-1	+1	+1	-1	1.3	5	15	0.06	0.45	180	43	3.29	5.57	10.31
11	+1	+1	-1	-1	+1	1.3	15	5	0	0.55	182	35	2.78	4.71	8.11
12	-1	-1	-1	-1	+1	0.7	5	5	0	0.55	190	34	4.77	8.06	14.92
13	-1	+1	+1	-1	+1	0.7	15	15	0	0.55	200	32	4.89	8.24	15.25
14	+1	-1	+1	-1	+1	1.3	5	15	0	0.55	195	33	2.94	4.97	9.21
15	+1	+1	-1	+1	-1	1.3	15	5	0.06	0.45	180	42	3.13	5.29	9.80
16	-1	-1	-1	+1	-1	0.7	5	5	0.06	0.45	180	43	4.47	7.55	13.98
17	+1	0	0	0	0	1.3	10	10	0.03	0.50	190	38	3.16	5.34	9.89
18	-1	0	0	0	0	0.7	10	10	0.03	0.50	195	39	4.65	7.86	14.55
19	0	+1	0	0	0	1	15	10	0.03	0.50	180	37	4.05	6.84	12.66
20	0	-1	0	0	0	1	5	10	0.03	0.50	190	38	3.79	6.39	11.83
21	0	0	+1	0	0	1	10	15	0.03	0.50	195	36	4.01	6.76	12.52
22	0	0	-1	0	0	1	10	5	0.03	0.50	190	37	3.86	6.51	12.05
23	0	0	0	+1	0	1	10	10	0,06	0.50	180	40	3.79	6.41	11.85
24	0	0	0	-1	0	1	10	10	0	0.50	195	32	4.23	7.13	13.21
25	0	0	0	0	+1	1	10	10	0.03	0.55	180	37	3.61	6.06	11.23
26	0	0	0	0	-1	1	10	10	0.03	0.45	180	32	4.87	8.21	15.21
27	0	0	0	0	0	1	10	10	0.03	0.50	180	35	4.11	6.93	12.83

As it is seen form equations (3)- (5) the increasing in ratio of sand and binder as well as increasing water-binder ratio and Tylose content due to prevention of sedimentation of hardening.

Compressive strength of the mortars specimens increases from 2.78...5.77 MPa at the age of 1 day to 7.21...18.04 MPa at the age of 28 days. The strength growth in later age is explained by common effect of such components as PC 15% and metakaolin 15%. The sufficient retaining time is provided due to retarding effect of citric acid.

Suggested proportions of the drypack mortars meet the requirements of local standards [DBN 2001, DSTU 2011]. Water consumption of such mortars is 0.45...0.48.

The research prove that such types of mortars can be obtained on the PG binder. As results of testing according to the plan (Tab. 7 and Tab. 8) demonstrate, that values of properties of mortars are like those on hemihydrate (Tab. 9).

In the case of application of PG the sand-binder ratio to meet the standard requirements must equal to 0.7, water-binder ratio not more than 0.45, content cement must be at maximum level 15% by binder weight, due to high water consumption MTK content is 5…15%.

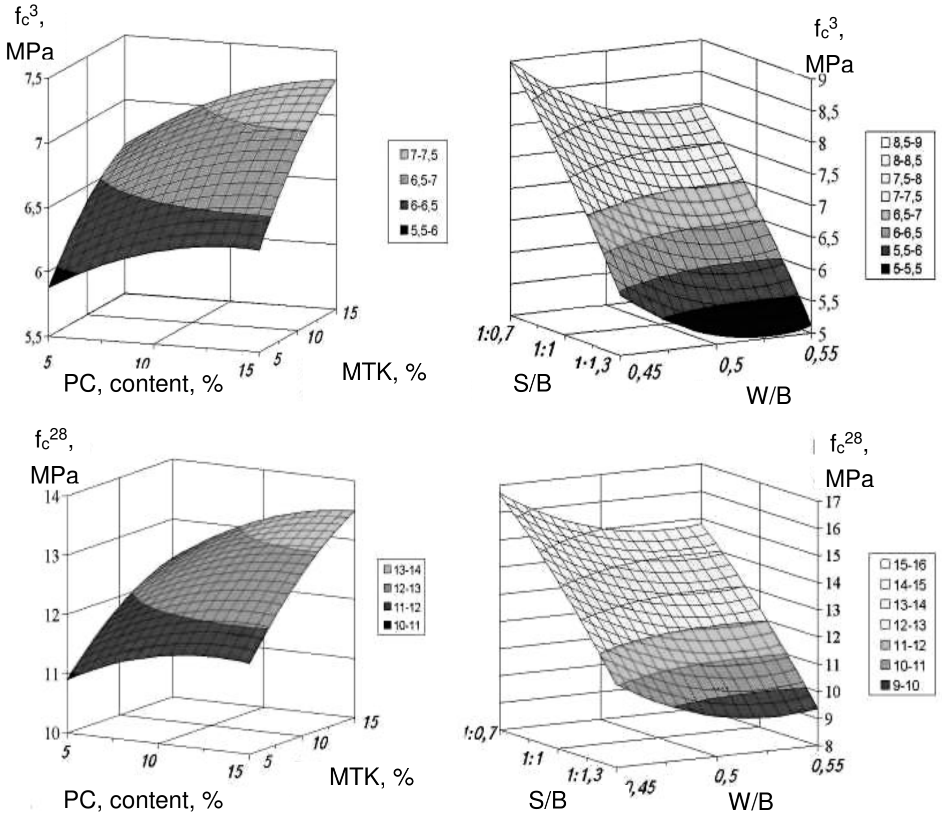

Fig. 3. Dependence of compressive strength of the mortars for self-leveling floors based on β-hemihydrate binder at the age of 3 and 28 days on the technological factors

However, there slower strength growth of PG based mortars comparing to hemihydrate-based mortars should be considered. It limits terms of construction works. Moving on such floor can be started not earlier than in 18…20 h after placing and finishing.

Table 9. Matrix of planned experiment and results of tests of mortars for self-levelling floor based on phosphogypsum binder.

Test no.	Factors (natural)					Output parameters				
	X_1	X_2	X_3	X_4	X_5	Flow spread diameter, mm	Reten-tion time, min	Compressive strength, at the age of, days, MPa 1	3	28
1	1.3	15	15	0.06	0.55	200	45	3.53	6.02	11.12
2	0.7	5	15	0.06	0.55	205	43	4.35	7.29	13.47
3	0.7	15	5	0	0.45	195	35	5.22	8.31	15.28
4	1.3	5	5	0	0.45	182	32	2.80	4.66	8.57

It is possible to use such mortars based on hemihydrate and PG for floor underlayment (thickness 10…80 mm), layers covering the insulating layers (with thickness not less than 30 mm) and self-levelling mortars for horizontal surfaces for different types of flooring (thickness 2…15 mm, compressive strength not less than 15 MPa).

Strength kinetic parameters meet the standard requirements: compressive strength at 24 h - 4...5 MPa, 3 days – 10...11 MPa, 28 days - 15...18 MPa.

5 CONCLUSIONS

The research summarizes the results of studying the basic properties of hemihydrate and phosphogypsum based binders and discovers one of the most potential area of application of PG – drypack mortars for flooring.

The results of research demonstrate that basic properties of PG based mortars are close to those based on hemihydrate based. The difference in setting time must be considered during flooring works.

β-hemihydrate and PG based mortars are appropriate for floor bottom layers (thickness 10…80 mm), upper layers covering the insulating layers (with thickness not less than 30 mm) and self-levelling mortars for horizontal surfaces for different types of flooring (thickness 2…15 mm, compressive strength not less than 15 MPa).

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