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Effect of the supplement based on calcium hydrosilicates on the resistance of lime coatings

Влияние добавки на основе гидросиликатов кальция на стойкость известковых покрытий

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Ключевые слова: добавка; гидросиликаты кальция; сухие строительные смеси; диатомит

Abstract. It was proposed to use synthesized calcium hydrosilicates in lime finishing dry mixes as a modifying supplement. The effect of substances containing amorphous silica, which are used in the synthesis, on the activity of the modifying supplement was established. The effect of the synthesis mode of supplement on the structure formation of lime compositions was illustrated. It was found that the injection of supplements of hydrosilicates accelerates the increase of mechanical strength. The efficiency of the using of modifying supplements of amorphous silica, such as diatomite, in the synthesis was shown. The evaluation of hydrophysical properties of coatings based on lime dry mix with the using of supplements synthesized in the presence of calcium hydrosilicates was provided. It was shown that the injection of supplements of hydrosilicates accelerates the increase of water resistance and frost resistance of coatings. It was found that the adhesion of the lime compositions with the supplement based on calcium hydrosilicates, synthesized in the presence of diatomite, is higher than that of the compositions with the addition of calcium hydrosilicates synthesized without diatomite.

Аннотация. Предложено применять в качестве модифицирующей добавки в известковых отделочных сухих строительных смесях синтезированные гидросиликаты кальция. Установлено влияние веществ, содержащих аморфный кремнезем, используемых при синтезе, на активность модифицирующей добавки. Показано влияние режима синтеза добавки на структурообразование известковых составов. Выявлено, что введение добавок гидросиликатов способствует ускорению набора механической прочности. Показана эффективность применения при синтезе модифицирующих добавок аморфного кремнезема, в частности, диатомита Инзенского месторождения. Приведена оценка гидрофизических свойств покрытий на основе известковой сухой строительной смеси с применением добавок, синтезированных в присутствии гидросиликатов кальция. Показано, что введение добавок гидросиликатов способствует повышению водостойкости и морозостойкости покрытий. Установлено, что адгезия известковых составов с добавкой на основе гидросиликатов кальция, синтезированной в присутствии диатомита, выше чем у составов с добавкой гидросиликатов кальция, синтезированной без диатомита.

Introduction

For the restoration and finishing of the walls of buildings and structures, lime compositions, including dry mixes, are used. Coatings based on lime dry mixes have high values of vapour permeability and bioresistance, which allow using them as traditional mixes for finishing architectural monuments and renovating structures in places of historical buildings. However, lime mixes are characterized by low values of strength and durability, which limiting their using. To avoid the destruction of lime dry mixes, modifying supplements are injected to their recipe [1, 2].

Previous studies has confirmed the efficiency of the injection of mineral supplements, based on synthesized calcium hydrosilicates, in the recipe of finishing lime dry mixes, that increase resistance of lime coatings, [3, 4, 5].

Synthesis of supplement was the deposition of soluble glass in the presence of calcium chloride solution [6, 7, 8]. Lime compositions with the using of synthesized calcium hydrosilicates (CHS) form coatings increased water resistance (softening coefficient is K_{soft} = 0.61).

In addition, it has been found that the disadvantages of the above methods for the obtaining of hydrosilicate supplements are the formation of high basic and low basic hydrosilicates during mixing, where the high basic hydrosilicates predominate to a greater degree. It has been established that high basic calcium hydrosilicates have less water resistance and strength compared to low basic hydrosilicates, so it is important to obtain a hydrosilicate supplement, which will contain more low basic calcium hydrosilicates. [9, 10, 11].

The aim of the work is to develop a technology of the synthesis of a supplement containing low basic calcium hydrosilicates.

To achieve this aim, it is necessary to solve the following:

- to reveal patterns of structure formation of lime compositions in the presence of supplements based on calcium hydrosilicates synthesized in the presence of diatomite;
- to develop a recipe for a lime dry mix, coatings based on it have increased operational resistance;
- to establish technological and operational properties of lime dry mixes and coatings based on it.

Given that low basic calcium hydrosilicates have higher strength, compounds, containing amorphous silica, such as diatomite of Inza deposit, were used in synthesis of supplement in further research [12, 13, 14].

Methods

Two CHS synthesis modes were used in work:

- mode 1 –the deposition in the presence of 15% $CaCl_2$ solution in an amount of 50% by mass of soluble glass;
- mode 2 –the deposition in the presence of 10% $CaCl_2$ solution in an amount of 50% by mass of soluble glass with the addition of diatomite, wherein the ratio of liquid:solid phase (L:S) was (L:S) = 1:2.

The resulting sediment was dried at a temperature of 100^{o}C.

The synthesized supplements were used to develop the recipe of lime dry mix [15, 16].

The first synthesis mode was used in accordance with the data given in [17]

The developed recipe includes lime powder of ClassII, quartz sand 80 % of fraction 0.63–0.315, 20 % of fraction 0.315–0.14. Plasticizer Kratasol PFM in an amount of 1 % by mass of lime, redispersible powder Neolith P-4400 in an amount of 0.3 % by mass of lime, hydrophobisator Zincum-5 in an amount of 0.5 % by mass of lime were injected in the recipe to control structural and mechanical characteristics of the lime composite. The content of CHS supplement was 30 % by mass of lime. Mixes were prepared with water: lime ratio W/L = 1.2.

Water holding capacity of finishing composition was determined in accordance with Russian State Standard GOST 5802-86 "Mortars. Test methods". Before the test, 10 sheets of filter paper of 150 × 150 mm each were weighed with an error of up to 0.1 g, placed on a glass plate of 150 × 150 mm and 5 mm thick, one layer of cheesecloth was put on the filter paper, a metal ring with an inside diameter 100 mm and a height of 12 mm was installed on top, and the whole installation was weighed again. Then the thoroughly mixed solution was put flush with the edges of the metal ring, weighed and allowed to stand for 10 minutes. After that, the metal ring with the solution was carefully removed along with the cheesecloth, and the filter paper was weighed with an error of up to 0.1 g.

The water- holding capacity of the finishing mixt was determined by the percentage of water content in the sample before and after the experiment, according to the formula:

$$V = 100 - \left(\frac{m_2 - m_1}{m_4 - m_2}\right) * 100, \quad (1)$$

where m_1 – mass of filter paper before the test, g; m_2 – mass of filter paper after the test, g; m_3 – mass of installation without solution mix, g; m_4 – mass of the installation with a solution mix, g.

The water holding capacity of the dry mix was determined twice for each sample and was calculated as the arithmetic mean of the results of the two determinations, differing by no more than 20 % from the smaller value.

To evaluate the operational resistance of coatings based on lime dry mix, frost resistance tests were carried out by cyclic freezing and melting of the finishing layer applied to the cement-sand base after 28 days of air-dry hardening. Appearance of coatings was evaluated according to Russian State Standard GOST 6992-68 "Lacquers and paints. Method for determination of coating weather-resistance". As the "failure", the state of the coating was taken, estimated of III.4 points (Table 3).

The kinetics of water absorption of coatings based on the dry mix was determined in accordance with Russian State Standard GOST 5802-86 "Mortars. Test methods".

Samples, previously dried to constant mass, were placed in a container filled with water. The water temperature in the container was (20 ± 2) °C. Samples were weighed at interval of 1 hour on conventional scales with an error of not more than 0.1 %. When weighing, samples taken from water are wiped with a damp cloth previously. The mass of water that leaked from the sample pores to the weighing pan was included in the mass of the saturated sample. The test was carried out until the results of two consecutive weighings differed by no more than 0.1 %. The water absorption of a single sample by weight W_m in percent was determined by the formula:

$$W_m = \frac{m_2 - m_1}{m_1} * 100\%, \quad (2)$$

where m_1 – mass of the dried sample, g; m_2 – mass of the water-saturated sample, g.

The ultimate compression strength of samples was determined in accordance with Russian State Standard GOST 5802-86 "Mortars. Test methods". As a equipment for testing the compression strength of samples, a test machine of the type "IR 5057-50" was used. Depending on the type of used power sensor of "IR 5057-50", the force measuring range was from 50 to 50000 N with an accuracy of 1 N (0.1 kgs). Built-in cross-bar speed controllers allow to set the speed of application of the load from 1 to 100 mm/min (in terms of displacement). The compression strength (MPa) of the samples was determined by the formula:

$$R_{\text{com}} = \frac{P}{F}, \quad (3)$$

where P – destructive force, N; F – cross-sectional area of the sample before the test, m^2.

Results and Discussion

It was found that after 28 days of air-dry hardening the compression strength R_{com} of the lime samples with supplement based on CHS, synthesized by the 2nd mode, is higher and is R_{com} = 5.5 MPa, while the compression strength of the lime samples with supplement based on CHS, synthesized by the 1st mode, is R_{com}= 2.86 MPa. Compression strength of control sample is R_{com}= 1.475 MPa.

Frost resistance was tested by cyclic freezing and melting of the finishing coat, applied to the cement-sand base, after 28 days of air-dry hardening to evaluate the operational resistance of coatings, based on lime mix. Evaluation of the appearance of the coatings was carried out according to Russian State Standard GOST 6992-68 "Lacquers and paints. Method for determination of weather-resistance of coatings". State of coating was assumed as the "failure", if it estimated at III.4 points (Table 1).It was found that the lime samples with supplement, based on CHS, have passed 35 cycles of the test. While the state of the coating after 35 test cycles is estimated at V.5 points that corresponds to the state of coating with loss of gloss to 5 %, with a slight change in color and the absence of blushing, bronzing, dirt retention, peeling, cracking, bubbling.

The results of the studies showed that the supplement based on calcium hydrosilicate has high activity, which is determined by the value of solubility in a 20 % KOH solution [18].

Table 1. Impact of the synthesis mode on the activity of the synthesized calcium hydrosilicate

Supplement	Solubility M, %	Activity A
Control composition	65	260
Lime composition with supplement, based on calcium hydrosilicates synthesized by 1st mode	68	350
Lime composition with supplement, based on calcium hydrosilicates synthesized by 2nd mode	70	370
Diatomite	61	299

The injection of a supplement based on calcium hydrosilicates, synthesized in the presence of diatomite, in the lime composition contibutes to an increase in water holding capacity. Table 2 shows the values of water holding capacity of lime samples.

Table 2.Water-holding capacity of the lime composites

Composition	Water-holding capacity, %
Control composition	95.5
Lime composition with supplement, based on calcium hydrosilicates synthesized by 1st mode	97.2
Lime composition with supplement, based on calcium hydrosilicates synthesized by 2nd mode	97.9

Thus, the water holding capacity of the composition based on the supplement synthesized in the presence of diatomite is 97.9 %, while of the composition based on the supplement synthesized without the use of diatomite – 97.2 %. The water holding capacity of the control composition is 95.5 %.

From the experimental data (Table 2) it follows that lime compositions with additives of calcium hydrosilicates are characterized by a sufficient water holding capacity of 97.2–97.9 %.

It was found that there is a difference in the state of coatings after 24 cycles of freezing and melting. Thus, state of the coatings, based on composition with CHS (1st synthesis mode), was estimated at V. 6 points (Table 3), and state of the coatings, based on composition with CHS (2nd synthesis mode), was estimated at V. 7 points after 30 cycles (Table 3).

Table3. Quality of the appearance of the coatings

Composition	Number of cycles	Points
Lime composition with supplement based on calcium hydrosilicates, synthesized on 1stmode	before test	V. 8
	6	V. 8
	12	V. 8
	18	V. 7
	24	V. 6
	30	V. 6
	35	V. 5
	40	IV. 4
Lime composition with supplement based on calcium hydrosilicates, synthesized on 2nd mode	before test	V. 8
	6	V. 8
	12	V. 8
	18	V. 7
	24	V. 7
	30	V. 6
	35	V. 5
	40	V.4
	45	IV. 4

Higher resistance to cyclic freezing and melting of the coatings based on composition with CHS (2nd synthesis mode) is due to a change of the porous structure, in our view. It was found that the injection of supplements, based on CHS, in the recipe of lime compositions reduces porosity. Thus, porosity of the samples with supplement, based on CHS synthesized by 1st mode, was estimated at P = 29.7 % (Table 4), and porosity of the samples with supplement, based on CHS synthesized by 2nd mode, was estimated at P = 26.7 % (Table 4).

Table 4. Porosity of the lime composites

Composition	Porosity, %		
	P_{cl}	P_{op}	P_{ov}
Control composition	7.1	28	35.1
Lime composition with supplement, based on calcium hydrosilicates synthesized by 1st mode	5.4	24,3	29.7
Lime composition with supplement, based on calcium hydrosilicates synthesized by 2nd mode	4.5	22.2	26.7

Adhesion of the lime samples aged 28 days of hardening with a supplement based on calcium hydrosilicates, synthesized in the presence of the diatomite, is R_{adg} = 0.89 MPa, while adhesion of the samples with the addition of CHS, synthesized without diatomite, is R_{adg} = 0.8 MPa [19, 20].

It was found that lime coatings based compositions with the addition of calcium hydrosilicates are characterized by higher water resistance. Thus, softening coefficient of lime samples with the supplement, based on CHS synthesized by the 1st mode, is K_{soft} = 0.61, while softening coefficient of lime samples with the supplement, based on CHS synthesized by the 2nd mode, is K_{soft} = 0.73. Softening coefficient of the control samples is K_{soft} = 0.29.

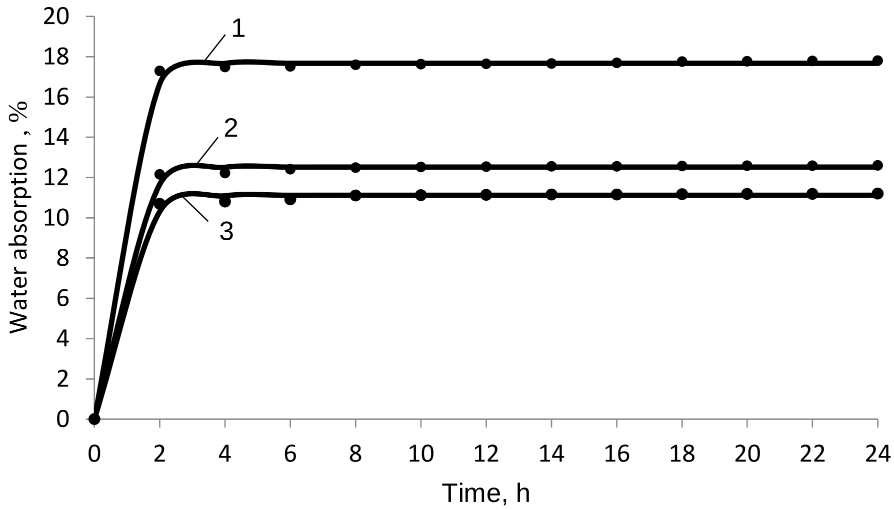

Figure 1. Water absorption by mass of lime composites:
1 – control composition with lime binder;
2 – composition with supplement, based on CHS synthesized by the 1st mode;
3 – composition with supplement, based on CHS synthesized by the 2nd mode

Figure 1 shows the curves of water absorption by mass of lime composites. Approximation of data was performed using software CurveExpert 1.3. Based on the results of approximation and experimental studies (Figure 1), it follows that lime samples with supplement, based on CHS synthesized by the 2nd mode, have lower water absorption than lime samples with supplement, based on CHS synthesized by the 1st mode.

The curves shown in Figure 1 were described by the exponential equation

$$y = a\,(1 - e^{-bx}), \qquad (4)$$

where *a* – constant, taking into account the highest possible water absorption; *b* – water absorption rate constant; *x* – time.

The values of the constants *a* and *b* shown in Table 5.

Table 5. The values of the constants of water absorption equation

Composition	*a*	*b*
Control composition	17.671	1.902
Lime composition with supplement, based on calcium hydrosilicates synthesized by 1st mode	12.517	1.754
Lime composition with supplement, based on calcium hydrosilicates synthesized by 2nd mode	11.117	1.512

The calculation results have shown that the water absorption rate constant of the samples based on compositions with addition of CHS is significantly lower, in the range of 1.512 to 1.754 $hour^{-1}$.

Compression strength of lime samples aged 28 days air-dry hardening has been estimated. For comparison, lime samples were made only using diatomite in an amount of 30% by weight of lime.

It is found that the compression strength lime samples made with using calcium hydrosilicate supplement synthesized without diatomite is 4.7 MPa (Table 6), while at the lime samples made with using calcium hydrosilicate supplement synthesized in the presence of diatomite – 7.59 MPa (Table 6). Compression strength of control mix is 2.12 MPa.

Table 6. Compression strength of lime samples

Supplement	Compression strength, [MPa]
The control mix (no supplement)	2.12
Lime composition with supplement, based on calcium hydrosilicates synthesized by 1st mode	4.7
Lime composition with supplement, based on calcium hydrosilicates synthesized by 2nd mode	7.59
Diatomite	3.25

Table 7 presents the technological and operational properties of the finishing composition and coatings based on it, which were based on the developed dry mix and the prototype composition.

Table 7. Technological and operational properties of the finishing composition

Parameter name	Parameter value for developed composition	Prototype
Compression strength, MPa	5.5	2.5
Adhesion strength, MPa	0.89	0.7
Frost resistance, cycles	35	35
Water-holding capacity, %	97.9	97
Water absorption, %	10.15	12
Water resistance	0.73	-
Shrinkage strain, %	0.024	-
Water vapour permeation μ, mg/m·t·Pa	0.049	0.01
Viability, h	1.5	2-3
Presence of cracks due to shrinkage	no cracks	no cracks

The technological and operational properties of the developed dry mix were compared with the properties of the lime plaster mix "Kreps Antique", produced by the company "Kriks".

Thus, coatings based on the developed dry mix have higher operational properties such as compression strength, adhesion strength, frost resistance, etc.

Conclusions

The possibility of increasing the resistance of coatings by applying a supplement based on calcium hydrosilicates synthesized in the presence of diatomite, has been substantiated, which reduces the total porosity, accelerates the curing of coatings, increases the strength and water resistance due to the formation of low basic calcium hydrosilicates.

It was found that the supplement based on hydrosilicates synthesized in the presence of diatomite, is characterized by high activity, A = 370 mg / g.

It has been established that the injection of a calcium hydrosilicate supplement, synthesized in the presence of diatomite, in composition of the dry mix contibutes to accelerate in curing of the coatings.

Thus, compositions with the supplements, based on CHS synthesized in the presence of diatomite, form coatings with improved water resistance and resistance to cyclic freezing and melting. Lime mixes with synthesized supplement are characterized by high compression strength, vitality of 3–4 hours, adhesive strength of 0.9–1.1 MPa.

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