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Case study

Structure and properties of synthesized additive based on amorphous aluminosilicates

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ABSTRACT

The article contains data on the structure and properties of synthesized additives for lime finishing compounds. It is observed that the samples, based on lime combined with synthesized additive, show rapid growth of durability at the initial stage.

1. Introduction

As a rule, lime compositions are used for historical buildings restorations. Given that lime compositions are characterized by slow curing periods and have insufficient water resistance, the use of nanosized additives – synthesized hydrosilicates, aluminum silicates, silica sol, organo-mineral additive (Loganina et al., 2014a,b,2015a,b) – is suggested. The research shows that such synthesized additives combined with lime based finishing compounds increase water- and frost-resistance of finishing coatings.

2. Results of researches

During further research, we found the possibility of using synthesized aluminosilicates in the formula of lime compositions. Aluminosilicates are synthesized by adding microfine aluminum powder to sodium silicate at 60 °C for 90 min.

The synthesized additive is lightweight powder of light gray colour (powder size 2–20 μm), with bulk density of 0.55 ± 0.05 g/cm^3. A large amount of gaseous molecular hydrogen appears during the process of additive synthesis. As a result, pores of different size and forms appear in the additive. Yield of the product is 90%. The chemical composition of the additive is shown in Tables 1 and 2.

The analysis of the data presented in Table 1 shows that the prevailing elements are C, O, Al, Si, Na.

X-ray diffraction analysis (XRD) showed that the mineralogical composition of the additive, is mainly represented by crystalline aluminum hydroxide—bayerite – α-$Al(OH)_3$ and boehmite – γ-AlO(OH). The amorphous phase is represented by sodium aluminosilicates.

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Table 1
The chemical composition of the synthesized additive.

Variation interval	Elements (%)												
	C	O	F	Na	Mg	Al	Si	S	Cl	K	Ca	Fe	Cu
Max	16.35	47.48	0.51	29.10	0.02	28.13	19.94	0.06	0.06	0.03	0.08	0.07	0.25
Min	12.12	34.86	0.08	6.15	0.00	3.55	3.26	0.00	0.02	0.00	0.03	0.0	0.08

Table 2
Oxide content in the additive composition.

Oxide name	Content (%)
Al_2O_3	51.03
SiO_2	36.36
Na_2O	11.89
Fe_2O_3	0.110
CaO	0.107
MgO	0.105
SO_3	0.0290
TiO_2	0.0124
K_2O	0.0112
Σ	99.6546

Fig. 1 shows electron micrograph of the additive. The picture analysis shows that the structure is represented by lamellar and needle-shaped formations 0.11–10.49 μm in size.

In addition, the mineralogical composition was estimated by differential thermal analysis done with "Termoskan-2". Thermal analysis (TA) of the samples was carried out at the temperature range of 20-1000 °C in air, at the heating rate of 10 °C/min. Fig. 2 shows the thermogram of the additive.

The additive thermogram analysis shows, that the heat effect at temperatures 100–135 °C results from free water loss. The moisture loss makes 5%. Small heat effect at temperatures 200–240 °C, (0.42 J) results from the beginning of bayerite $Al(OH)_3$ dehydration; the sample weight change makes 11%. The heat effect at temperature 310–350 °C results from partial dehydration of bayerite turning into boehmite AlO(OH). The sample weight change makes 15.5%.

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The curves of differential-thermal analysis (DTA) have exothermic effect with a maximum at 689 °C, caused by formation of Al_2O_3. The thermal effect makes 12.83 J. At temperature 850 °C we can see a diffused peak, showing the exoeffect and transition of γ-Al_2O_3 into α-Al_2O_3. The weight loss makes 18%.

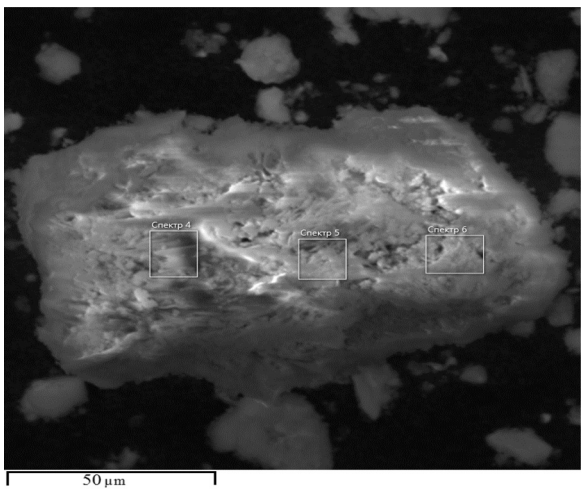

Fig. 1. Electron micrograph of the additive.

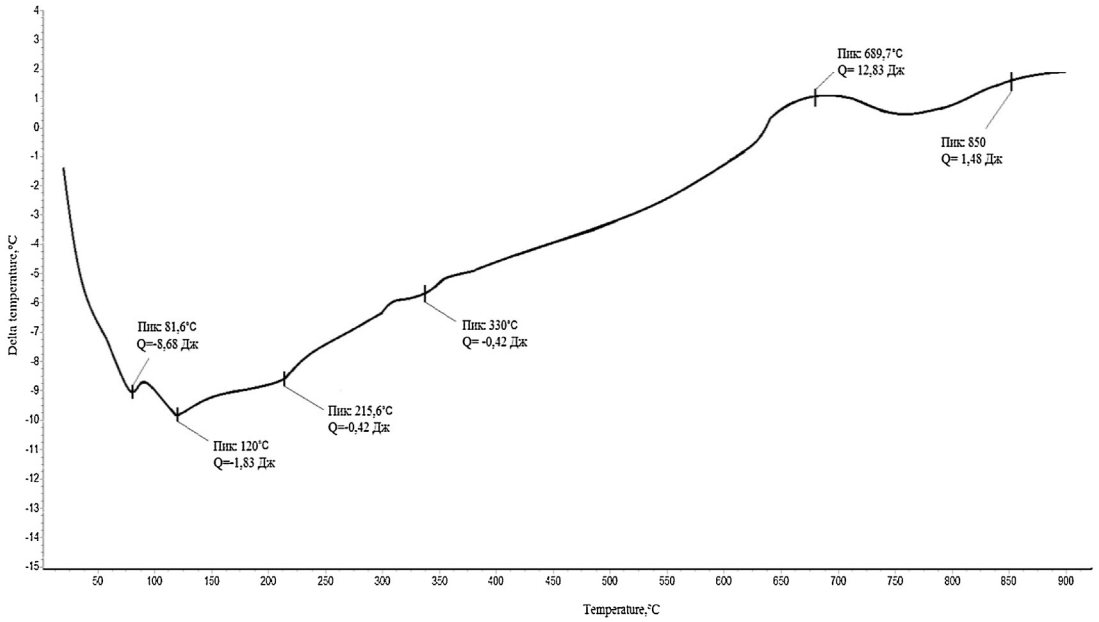

Fig. 2. Thermogram of the synthesized additive.

The paper examines the impact of the additive on the physical and mechanical properties of lime samples (Table 3). To control the properties of the additive varied temperature conditions of the synthesis were used (Table 3). The experiment involved the use of sodium silicate solute with silica modulus of 2.9. The development of synthesis technology was conducted with the following ratio of the components (aluminum powder: sodium silicate solute: water): Condition 1 – 1:4:7; Condition 2 – 1:8:14.

As a criterion for optimization of the synthesis, the indicator of lime composite durability was taken. The experiment involved powder lime made of lime with activity of 84%. Water-to-lime ratio was 1. The samples hardened in air-dry conditions at temperature of 18–20 °C and relative air humidity of 60–70%. Preliminary studies have shown that the optimal ratio of the additive is 5–10% by weight of lime.

Analysis of the data shown in Table 3 shows that the samples based on lime composite binder (LCB) possess higher porosity than samples with lime binder. For example, the porosity of control samples was 50.00%, and of those based on LCB – 66.10-73.29%. Despite the increase in porosity samples based on SCR are characterized by higher durability of 1.71–2.36 MPa (with 5% of the additive) and 2.21–2.86 (with 10% of the additive).

Reduced density of samples based on LCB can occur due to the use of the additive that has been synthesized for 1.0 h at 60 °C. Further increase in the temperature and time of heat treatment of the additive results in some further increase of lime samples density.

Despite the increase in the porosity of the samples based on LCB, they are characterized by higher durability, which, in our opinion, is caused by the chemical interaction of lime with synthesized aluminosilicate.

Lime samples based on LCB are characterized by high softening index (SI), depending on the content of additives and their mode of synthesis SI = 0.41-0.72. The additive synthesized with lower aluminum ratio, contributes to greater increase of water resistance of lime composites SI of which equals 0.49–0.72

XRD data shows that the mineralogical makeup of the samples based on LCB is represented by hydrocarboaluminates calcium, d, A (4.613; 2.5289), hydroaluminates calcium d, A (4.099; 3.948; 3.6187; 2.8432), hydroaluminosilicates calcium, d, A (5.016; 3.1816), calcite, d, A (3.0079; 2.7542), calcium hydroxide, d, A (3.1816; 2.6433), sodium hydroaluminosilicates, d, A (3.6896; 2.9214; 2.6708).

The analysis of lime based composite combined with synthesized additive showed that thermal effect at 80–130 °C (1.227 J) is due to free water loss (Fig. 3, curve 2). Thermal effect at 150–210 °C is due to consecutive dehydration of hydroaluminate calcium and hydrocarboaluminate calcium (2.17 J). Thermal effect at 479–550 °C (13.77 J) is due to portlandite dehydration.

The thermal effect at temperature 800–1000 °C is 16.82 J. It characterizes calcite dissociation; the weight loss is 9.93%.

Analyzing the thermograms of the lime based combined with synthesized aluminosilicate composition and control samples (without additives) we can see, that the thermal effect resulting from portlandite dehydration in control samples is higher (25.63 J) and is, evidently, caused by a high content of portlandite (Fig. 3a curve 1). This is confirmed by data on the content of non-combined lime. It was found that after 28 days of dry curing the control samples have 47.67% of free lime, while samples with synthesized aluminosilicate have 31.41% of free lime.

Table 3
Properties of lime composite depending on the synthesis conditions of aluminosilicate additive.

Synthesis conditions	Water adsorption (%)	Durability (MPa)	Density (g/cm^3)	Porosity (%)			Softening index
				General	Open	Closed	
1	2	3	4	5	6	7	8
The contents of the additive of 10%							
Control (no additives)		1.0	0.94	50.00	38.9	11.1	0.37
Composition 1. Temperature 60 °C, heat treatment time 30 min.	67.4	2.21	0.809	68.86	54.58	14.28	0.67
Composition 1. Temperature 60 °C, heat treatment time1 h.	74.2	2.63	0.694	73.29	51.54	21.75	0.42
Composition 1. Temperature 60 °C, heat treatment time 2 h.	65.6	2.7	0.747	71.24	49.06	22.18	0.62
Composition 2 Temperature 60 °C, heat treatment time 30 min.	66.7	2.31	0.841	71,00	56.92	14.08	0.67
Composition 2 Temperature 60 °C, heat treatment time 1 h.	67.2	2.66	0.791	69.55	53.24	16.31	0.50
Composition 2. Temperature of 60 °C, heat treatment time 2 h.	61.7	2.86	0,843	67.55	52.09	15.46	0.53
Composition 2. Temperature 80 °C, heat treatment time 30 min.	63.6	2.6	0.844	67.51	53.61	13.90	0.68
Composition 2. Temperature 80 °C, heat treatment time 1 h.	58.8	2.51	0.881	66.10	51.80	14.30	0.68
Composition 2. Temperature 80 °C, heat treatment time 2 h	61.3	2.77	0.794	69.45	48.65	20.80	0.72
The contents of the additive of 5%							
Composition 1. Temperature 60 °C, heat treatment time 30 min.	66.04	1.71	0.82	68.41	54.23	14.18	0.65
Composition 1. Temperature 60 °C, heat treatment time 1 h.	69.71	2.13	0.75	70.88	52.78	18.10	0.41
Composition 1. Temperature 60 °C, heat treatment time 2 h.	66.45	2.20	0.78	69.83	52.10	17.73	0.61
Composition 2 Temperature 60 °C, heat treatment time 30 min.	66.06	1.81	0.80	67.12	56.92	10.20	0.66
Composition 2 Temperature 60 °C, heat treatment time 1 h.	67.06	2.16	0.79	68.47	54.96	13.51	0.49
Composition 2. Temperature of 60 °C, heat treatment time 2 h.	67.05	2.36	0.82	67.87	56.01	11.86	0.52
Composition 2. Temperature 80 °C, heat treatment time 30 min.	65.87	2.1	0.84	67.83	55.07	12.76	0.67
Composition 2. Temperature 80 °C, heat treatment time 1 h.	64.60	2.01	0.84	67.72	54.21	13.51	0.67
Composition 2. Temperature 80 °C, heat treatment time 2 h	61.19	2.27	0.85	67.19	52.21	14.98	0.71

Thermal effect at the temperature range of 65.9–134.3 °C is 3.846 J. That is significantly larger when compared to the samples containing the additive.

We have proposed lime composite binder (LCB) which contains 5–10% of its mass as synthesized additive. The following are the results of the research of the properties of the composites with LCB. We used powder lime made from lime with activity of 84%. The ratio of water to lime (W/L) was 1.0. The samples hardened in air-dry conditions at temperatures of 18–20 °C and relative humidity of 60–70%.

The data analysis testifies that the samples with LCB have higher porosity in comparison with the composites based on lime binder. For example, porosity of the control samples makes 53.81%, while porosity the samples based on LCB is 67.18–69.84%.

Despite the porosity increase, the samples are characterized by high durability which makes 1.71–2.32 MPa (with 5% of the additive) and 2.21–2.86 (with 10% of the additive).

When evaluating the structure formation kinetics of the composites, it was found that at the initial stage of hardening a rapid growth of durability is observed. So, the plastic strength of the sample with 10% of the additive by weight of lime after 8 h of dry curing is 0.019135 MPa, while plastic strength of the control sample (without additives) is 0.001004 MPa

As the additive content increases, the plastic strength increases too.

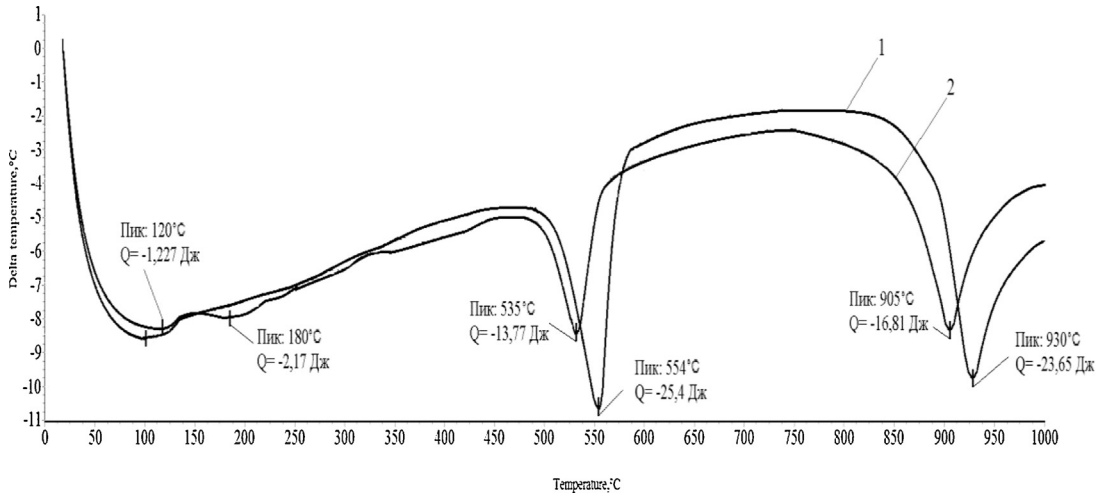

Fig. 3. The curves of the sample thermal analysis: 1 – control sample; 2 – sample with the synthesized additive.

3. Conclusions

The efficiency of use of the additive based on amorphous aluminosilicates in dry lime mixes is proved. The chemical interaction between the additive and lime is revealed. The increase in strength with the addition of lime composites based on amorphous aluminosilicate is showed. This LCB is recommended to use in the manufacture of heat-insulating dry building mixes.

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