DOI:10.22337/2587-9618-2026-22-2-105-112

INFLUENCE OF SYNTHETIC POLYSACCHARIDES ADDITIVES ON STRUCTURE FORMATION OF LIME COMPOSITES

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Abstract: The purpose of this work is to study the effect of synthetic polysaccharides on the structure formation and properties of lime coatings. Information is provided on the influence of synthetic polysaccharides on the structure formation of lime coatings. It has been shown that due to the water-retaining effect of the additive, more favorable conditions are created for lime carbonation. Using X-ray phase and thermographic analysis, optical and infrared spectroscopy, the presence of inter- and intracrystalline organic compounds was established, which modify the (nano)structure of newly formed calcite crystals and increase the strength of lime coatings.

Keywords: lime, polysaccharides, structure, crack resistance, strength, carbonation

ВЛИЯНИЕ ДОБАВОК СИНТЕТИЧЕСКИХ ПОЛИСАХАРИДОВ НА СТРУКТУРООБРАЗОВАНИЕ ИЗВЕСТКОВЫХ КОМПОЗИТОВ

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Аннотация: Целью данной работы является изучение влияния синтетических полисахаридов на структурообразование и свойства известковых покрытий. Представлена информация о влиянии синтетических полисахаридов на структурообразование известковых покрытий. Показано, что благодаря водоудерживающему эффекту добавки создаются более благоприятные условия для карбонизации извести. С помощью рентгенофазового и термографического анализа, оптической и инфракрасной спектроскопии установлено наличие меж- и внутрикристаллических органических соединений, которые модифицируют (нано)структуру вновь образованных кристаллов кальцита и повышают прочность известковых покрытий.

Ключевые слова: известь, полисахариды, структура, трещиностойкость, прочность, карбонизация

INTRODUCTION

Of the many types of binders, along with polymer and cement, lime compositions are used in some cases. Lime-based binders are widely used for the restoration of cultural heritage sites [1]. During operation, coatings applied to brick, concrete and other walls are adversely affected by cyclical temperature and humidity factors, biocorrosion, etc. [2, 3, 4, 5]. An effective method of increasing the durability of building materials is the introduction of various additives [6, 7, 8], activation of the sealing water and other components [9, 10, 11, 12]. Given the low resistance of lime binder coatings, various modifying additives are introduced into the formulation [13, 14, 15, 16, 17, 18]. In a number of studies, various organic additives based on blood albumin, casein, natural resins, egg proteins, decoctions of grain grains, and vegetable oils were noted in the composition of plaster bases of ancient paintings [19]. The role of organic additives remained unclear for a long time, due to the difficulty of determining them in gypsum and lime binders using existing analytical methods. Until now, organic adhesives have been considered as plasticizing and retarding additives for binding materials. Only the effect of glue additives on the setting time of gypsum binders was studied.

The ancient masons of the Maya tribe used plant extracts in the manufacture of solutions [20, 21]. The works [22, 23, 24, 25] describe the properties of lime plasters with additives of natural polysaccharides. The authors have established the adsorption of organic substances on the surface of calcite crystals. It is of interest to study the possibility of using synthetic polysaccharides in the manufacture of lime formulations for the restoration of cultural heritage sites.

METHODS

Slaked lime (fluff) with a true density of 2230 kg/m^3, a bulk density of 280 kg/m^3, an activity of 83 %, and a specific surface area of 559 m^2/kg were used in the work. The water-soluble modified polysaccharides Atren Cem LV and Atren Cem HV (TU 2458-062-63121839-2014) were used as an organic additive.

The cohesive strength of the coatings was determined by the axial tensile strength on samples measuring 10x10x50 mm and calculated by the formula

$$R_p = \frac{P}{F} \qquad (1)$$

where P is the destructive force, H;

F is the cross–sectional area of the sample before the test, m^2.

The Vickers hardness was calculated using the formula

$$HV = \frac{2Psin\propto/2}{d^2} \qquad (2)$$

where P is the load on the indenter, N;

α is the angle between the opposite faces of the Vickers indenter;

d is the diagonal of the Vickers indenter print, mm.

The surface area of the print was determined by the diagonal d of the print, which was measured under a microscope.

The rate of carbonization of lime paste was studied by changing the thickness of the carbonized layer. The kinetics of carbonization of control samples and with the addition of Atren Cem LV polysaccharide were determined. The thickness of the carbonized layer was determined by applying a 1% alcohol solution of phenolphthalein to a chip of the test sample. Areas of non-carbonated lime react to phenolphthalein, turning purple. Lime carbonation took place under natural conditions at a temperature of 20 °C.

RESULTS AND DISCUSSION

Formulations with the addition of polysaccharides are characterized by a slightly higher water retention capacity of 98 %. The research results indicate that lime formulations with the addition of polysaccharide in an amount of 1 % by weight of lime are characterized by delayed drying times. For example, the drying time to degree 3 on a concrete substrate is 15–20 minutes, while the control composition (without additives) is 7 minutes. An increase in the frost resistance grade from F25 (control) to F35 (with the addition of polysaccharide) has been established.

Table 1. Properties of the finishing composition with the addition of polysaccharides

Name of indicators	Value
Water retention capacity,%	98
Drying time to degree 3 on a concrete base, minutes	15-20
Tensile strength at the age of 3 months, MPa (Atren Cem LV additive in the amount of 1% by weight of lime)	0.547
Relative deformations, mm/mm	0.392
Frost resistance, brand	F35
Adhesion strength to concrete, MPa	1.1-1.3
Modulus of elasticity, MPa	0.925

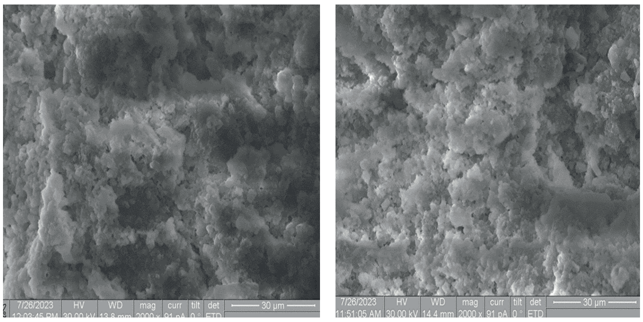
An X-ray phase analysis of lime samples was performed on a D8 Advance powder diffractometer (Germany). It was found that the calcite content increases in samples with the use of polysaccharide additives. Obviously, due to the water-retaining effect of the additive, more favorable conditions are created for the carbonization of lime. The amount of calcite in the control samples is 87.4 %, and in the samples prepared with the additive - 88.87 %. The diffractograms show a slight increase in the width of $CaCO_3$ peaks, which indicates the possible incorporation of organic molecules into calcite (Table 2).

Table 2. Crystal Lattice Parameters

Composition	Crystal Lattice Parameters		
	$CaCO_3$	$Ca(OH)_2$	SiO_2
control			
a	4.98700	3.58440	4.90000
c	17.05800	4.89620	5.40000
Slaked in the presence of an additive			
a	4.99100	3.58620	4.91580
c	17.06200	4.88010	5.40910
With an addition			
a	4.98700	3.58620	4.91000
c	17.05800	4.88010	5.40000

Fig. 1 shows micrographs of the hardened lime binder. Chains of rounded $Ca(OH)_2$-crystals are found (Fig. 1, a), as well as elongated polyhedral plates of calcium carbonate. The analysis of the images shows low-contrast nanometer-sized regions in individual calcite crystals with inhomogeneities corresponding to the amorphous (organic) phase (Fig. 1, b), similar to those observed in several $CaCO_3$ biominerals with occluded organics.

The inclusion (adsorption) of polysaccharide additives in/onto calcite is confirmed by the data of polysaccharide adsorption on calcite. The adsorption value was determined by the change in the surface tension of the Atren Cem LV additive solution of different concentrations. It was found that the adsorption value of the Atren Cem LV additive on calcite is 0.000457 g/cm^2

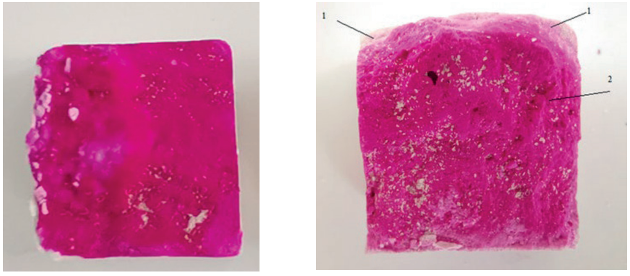


a *b*

Figure 1. The structure of calcareous hardened stone: a – control composition; b - with the addition of polysaccharide Atren Cem HV

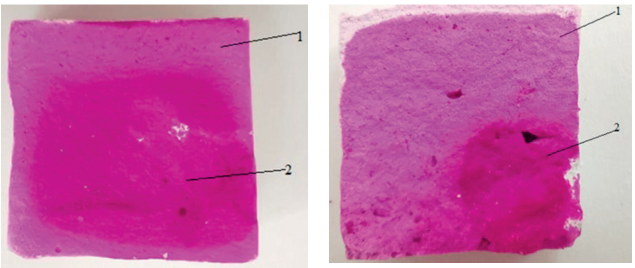
Taken together, these results confirm the presence of both inter- and intracrystalline organic compounds that modify (nano)the structure of newly formed calcite crystals at various scales reproduces the multilevel hierarchical mesocrystalline features observed in many $CaCO_3$ biominerals and their biomimetics, which include inter- and intracrystalline (bio)macromolecules.

Fig. 2–3 show photos of the kinetics of carbonation of lime samples.



a *b*

Figure 2. Change in the thickness of the carbonized layer of lime samples at the age of 4 days: a - control composition; b - with the addition of polysaccharides; 1 – carbonized layer; 2 – non-carbonated layer



a *b*

Figure 3. Change in the thickness of the carbonized layer of lime samples at the age of 10 days: a - control composition; b - with the addition of polysaccharides; 1 – carbonized layer; 2 – non-carbonated layer

It is evident from Fig. 2 and 3 that the carbonization process begins in the surface layer of the material (the carbonized areas are colored lighter purple), gradually moving inside the sample. The highest degree of carbonization is observed in samples with the addition of Atren Cem LV at a humidity of 21 % (Fig. 3, b). Only a small area in the center of the sample is colored dark purple. The humidity of the control samples was 14 %, the thickness of the carbonization layer is significantly less.

The results of the assessment of the degree of carbonization of lime composites are also confirmed by the data of X-ray phase analysis. The mineralogical composition of the lime composite was determined using a powder diffractometer D8Advans (Germany). It was found that the amount of calcite in the control samples is 87.4 %, in the samples prepared on lime slaked in the presence of the additive Atren Cem HV – 92.266 % and in the samples prepared with the additive Atren Cem HV – 88.87 %.

Polysaccharides cause a hardening effect, contributing to plastic deformation and, consequently, preventing catastrophic destruction caused by external factors.

The obtained results indicate that lime compositions with the addition of Atren Cem LV polysaccharides are characterized by higher cohesive strength. As the hardening age increases, the cohesive strength of the samples with the addition of the synthetic polysaccharide Atren Cem LV increases to a greater extent. Thus, at the age of 28 days of hardening, the cohesive strength of the samples based on the control composition is R 0.22 MPa, and with the addition of polysaccharide – 0.24 MPa. At the age of 3 months of hardening, differences in the values of cohesive strength are more pronounced. The cohesive strength of the control samples is 0.264 MPa, and samples with the addition of Atren Cem LV in the amount of 1% by weight of lime – 0.47 MPa, with the addition of Atren Cem LV in the amount of 0.5% by weight of lime – 0.379 MPa.

Taken together, these results indicate that organic substances induce a hardening effect by promoting plastic deformation and therefore inhibiting failure. The presence of crystalline organic molecules between and inside the lime coating gives higher deformative properties. This is evidenced by the data of the modulus of elasticity and hardness of the coatings. It was found that the modulus of elasticity of a sample of lime coating with the addition of Atren Cem LV in an amount of 1 % by weight of lime is 0.925 MPa, and the control is 0.987 MPa. This is also evidenced by data on the hardness of lime coatings. It has been established that the Vickers hardness of samples based on the control composition is HV = 1.649 MPa, and the hardness of coatings based on the composition with the addition of Atren Cem LV is 1.144 MPa.

It has been established that coatings based on compositions with the addition of polysaccharides are characterized by reduced shrinkage deformations. After 85 days of hardening, shrinkage deformations of coatings based on the control composition amount to 6.56 %, and on the basis of the composition with the addition of Atren Cem LV – 2.82 %.

CONCLUSION

It was found that diffractograms of calcareous samples with additives of Atren Cem HV show a slight increase in the width of $CaCO_3$ peaks, It has been established that the introduction of polysaccharide additives into the formulation of lime compounds causes the formation of inter- and intracrystalline organic compounds that modify the (nano) structure of newly formed calcite crystals at various scales, which helps to increase the strength of the composite. The results of the conducted studies confirm that the introduction of additives based on polysaccharides Atren Cem LV and Atren Cem HV into the formulation of lime compositions contributes to an increase in the carbonization front.

FUNDING

The research was funded by the National Research Moscow State University of Civil Engineering.

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