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Lime compositions with polysaccharides for building walls restoration ad protection

ABSTRACT

Preserving historical heritage is a significant priority in contemporary urban planning. However, enhancing the durability of lime coatings used in the restoration of cultural heritage sites remains a challenge. This study aims to identify a method for improving the crack resistance of lime-based coatings using polysaccharide additives. The study examines water-soluble modified polysaccharides AtrenCemHV and AtrenCem LV. The maximum adsorption values of these additives on lime were determined, with AtrenCemHV at 1.83 g/g and AtrenCem LV at 1.66 g/g. Findings indicate that the adsorption of polysaccharides onto the surface of $Ca(OH)_2$ lime particles leads to the formation of a composite structure containing inter- and intercrystalline organic molecules, which enhances the crack resistance of the coatings. The research also provides insights into the carbonization process of lime coatings containing polysaccharide additives, revealing that these additives increase the thickness of the carbonized layer. Lime compositions with polysaccharides demonstrate greater cohesive strength due to a high calcite content. Additionally, the formation of a composite structure with organic molecules contributes to a reduction in the elastic modulus and hardness of the coatings. Lime coatings with the AtrenCem LV additive endured 35 cycles of freezing and thawing. Thus, the new findings on lime compositions with polysaccharide additives are crucial for restoring building walls.

Keywords: *lime; polysaccharides; monomolecular adsorption; carbonization; crack resistance; coatings*

1. INTRODUCTION AND RESEARCH AIM

The challenge of preserving historical heritage is a central concern in today's urban planning [1]. Natural stone and exterior finishes of building facades deteriorate due to physical weathering and harsh environmental conditions [2]. Restoration and rehabilitation efforts rely on specialized materials that not only restore the visual appeal of structures but also ensure their usability[3,4]. Lime-based compositions are frequently used in the restoration of historical buildings. Among the most widely used commercial lime paints for such restorations are “Holvi,” “Kalcemur,” “Silacra-lime,” “Antik 1,” and “Antik 2,” among others [5,6].

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Some of these paints maintain traditional compositions, which help protect old surfaces and make them ideal for heritage restoration, while others are enhanced with cellulose fibers to improve coverage and ease of use while retaining technical and protective qualities.

Lime compounds are known to harden slowly, complicating the finishing process [7,8]. To speed up hardening and boost the strength of lime composites, various additives are incorporated into their formulation, such as sodium aluminate, sodium fluoride, potassium carbonate, calcium chloride, amorphous aluminum oxide, lithium carbonate, calcium formate, and finely dispersed amorphous silica [9,10].

Improving the resilience of mineral binder composites has increasingly focused on the use of colloidal silicon dioxide dispersions to address technological challenges [11,12]. Silicon oxide nanoparticles, reacting with Ca(OH)2 over time to form calcium silicate hydrate, play a significant role

in reducing pore sizes (by up to 30%) as they fill the pores with sol particles and reaction products [13,14].

The use of a modified silica sol, known as “Hardness-C,” has been proposed for the formulation of dry construction mixtures [15,16]. To further accelerate the hardening of lime, adding natural zeolites to the mixture is also suggested [17,18].

Research by [19,20]shows that incorporating zeolite-containing rock particles into cement systems can result in the formation of insoluble compounds between fluorine and calcium ions, which fill the pores in the cement-zeolite paste. This reduces fluoride ion concentration, thickens the diffusion layer, lowers hydration temperature by reducing the cement content when a filler is added, enhances cement paste strength, and results in the release of free lime [21,22].

Considering the limited availability and varying properties of natural zeolites, synthetic zeolites are a promising alternative for use in mineral binder compositions [23,24,25,26]. Research by [27,28] suggests that synthesized calcium silicate hydrates can enhance the durability of lime coatings. A dry lime composition has been developed for wall finishing and restoration, containing a calcium silicate hydrate filler. This allows for the creation of mortar mixtures with water retention rates of 98-99%, a drying time of 15-20 minutes to reach degree "5," and a working time of 1-1.5 hours. These coatings have a vapor permeability coefficient of 0.05 mg/m·h·Pa, adhesive strength of 0.6-0.9 MPa, and compressive strength of 3-4 MPa. For restoring historical masonry, studies by [29,30] recommend lime-metakaolin mixtures. These mortars have been used in Genoa's white plasters, and it was found that increasing the metakaolin-to-lime ratio raises the amount of chemically bound water, reduces pore size (to 0.1 μm), and enhances compressive strength up to 9 MPa. [30] also proposed using lime compositions with organic components (such as polysaccharides, proteins, and fatty acids) in restoration projects. [31] found that adding animal glue can double the mechanical strength of the mortar, extend the carbonization front, and reduce porosity and pore size. Historically, plant extracts, glutinous rice, fruit juices, oils, and animal fats were added to slaked lime in various regions to improve the properties of lime mortars and plasters [32,33]. For instance, Vitruvius's "De Architectura" recommends adding oil (Oleo subacta) to lime for waterproofing. In the Americas, ancient Mayan masons also used plant extracts, as supported by ethnohistorical, archaeological, and analytical evidence.

In this context, evaluating the applicability of the theory of monomolecular adsorption is crucial. This theory posits that adsorption occurs at specific active centers on the adsorbent surface, where each center interacts with only one adsorbate molecule, forming a single molecular layer [34,35]. The process is balanced by dynamic equilibrium between adsorption and desorption. Despite significant research on improving the durability of lime composites, many aspects, especially those related to restoring cultural heritage sites, remain unresolved. A promising approach for developing lime compositions involves a biomimetic method that creates a lime composite with meso-nanostructural properties similar to calcite biominerals, thereby greatly enhancing the long-term performance of the restoration material.

This article aims to develop lime compositions enhanced with polysaccharide additives for building wall restoration. To achieve this, the study focuses on analyzing the structure formation process of lime compositions with polysaccharides, examining the surface activity of polysaccharides in water solutions, and applying the theory of monomolecular adsorption to understand the adsorption behavior of polysaccharide additives on lime.

2. MATERIALS AND METHODS

This study utilized slaked lime with a true density of 2230 kg/m³, a bulk density of 280 kg/m³, and an activity level of 83%. The organic additives used were water-soluble modified polysaccharides AtrenCemHV and AtrenCem LV, both based on cellulose ether (hydroxyethylcellulose) produced through the reaction of alkali cellulose with ethylene oxide. These additives differ primarily in their molecular weight, with AtrenCemHV having a higher molecular weight. According to standard guidelines, the additives were used in proportions ranging from 0.2% to 1.0% of the binder's weight. During the production of the lime composition, the water-to-lime (W/L) ratio was maintained at 1.0 for all formulations to avoid excessive liquidity. The AtrenCemHV and AtrenCem LV additives were introduced into the mixture during the addition of mixing water.

The true and bulk densities of the slaked lime were measured by weighing a specified volume in both compacted and loose states. The lime's activity was assessed according to the Russian standard GOST 22688-2018. For this, a 1 g lime sample was placed in a 250 ml conical flask, and 150 ml of distilled water was added along with three to five glass beads. The flask was covered with a watch glass and heated to the boiling point for 7 minutes. After cooling to 20-30°C, the flask's

walls and the watch glass were rinsed with boiled distilled water, and a few drops of a 1% phenolphthalein alcohol solution were added. The solution was then titrated with a 1 N hydrochloric acid solution while stirring continuously until complete discoloration occurred. The active calcium and magnesium oxides (A, %) in the hydrated lime were calculated using the formula:

$$A = \frac{VT_{CaO}}{W(100 - W)} 100\%$$

where:

W: moisture content of hydrated lime

V: volume of 1 N hydrochloric acid solution used for titration, in ml

T_{CaO}: titer of 1 N hydrochloric acid solution, expressed in grams of CaO

The moisture content of the hydrated lime was determined by placing a 10 g lime sample in a pre-dried weighing bottle, which was then dried in an oven at 105°C to 110°C until a constant mass was reached. The moisture content (W, %) was calculated using the formula:

$$W = \frac{m - m_1}{m} \cdot 100\%$$

where:

m: initial mass of the lime sample, in grams

m_1: mass of the lime sample after drying, in grams

The structure of the hardened lime composite was analyzed using a D8Advans powder diffractometer (Germany), utilizing CuKα radiation with sample rotation. The measurements were conducted for lime composite powder in continuous mode (1 degree per minute) and step-by-step mode (0.02° step size, 10 seconds exposure) across a 2Θ angle range of 5°–100°. X-ray diffraction-based quantitative phase analysis methods leverage the fact that each substance generates a unique set of interference lines. While the ratio of line intensities for each phase remains constant, individual line intensities are proportional to phase content and are influenced by the sample's absorption coefficient.

A TESCAN Vega 3 scanning electron microscope (SEM) was used to examine pre-polished samples (50×25×8 mm). The adsorption behavior of AtrenCemHV and AtrenCem LV additives on lime was studied during the structure formation process of the lime composite. The adsorption was quantified by observing changes in the surface tension (σσσ) of the polysaccharide solution due to a reduction in adsorbent concentration upon adding lime. The stalagmometric (drop counting) method was used for surface tension measurements. To determine the amount of adsorption, lime was mixed into the additive solution, stirred, and allowed to settle until adsorption equilibrium was reached. Adsorption measurements were taken at intervals of 15 minutes and 48 hours after mixing. Data processing followed the equation:

$$a = \frac{a_\infty b C_p}{1 + b C_p}$$

where:

a: adsorption value, kg/m²

a_∞: maximum adsorption at complete mineral surface coverage, kg/m² or g/g

b: constant representing the adsorption capacity of additives, 1/%

C_p : equilibrium concentration in the solution, %

Graphical methods were used to determine the maximum adsorption (a_∞) and the constant *b* according to the Langmuir equation, with plots of $\frac{C_p}{a} - C_p$coordinates.

The elastic modulus of the lime coatings was evaluated using tensile testing, while Vickers hardness was assessed by pressing a tetrahedral diamond pyramid into a 4×4×16 cm sample, with a 136° angle between opposing faces (Figure 1). The surface area of the indentation was calculated using the diagonal (*d*) measured under a microscope.

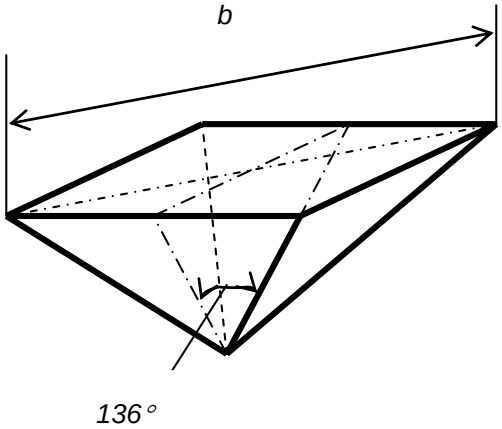

Figure 1. Tetrahedral diamond pyramid for hardness determination by the Vickers method

$$HV = \frac{2P \cdot \sin\frac{\alpha}{2}}{d^2}$$

where:

P: load on the indenter, N

α: angle between opposite faces of the Vickers indenter

d: diagonal of the indentation, mm

The tensile (cohesive) strength of the samples was measured using an IR 5057-50 tensile testing machine (Russia) according to the Russian standard GOST 18299-72*. Tests were conducted at 20°C and 60% relative humidity. The samples (10×10×50 mm) were clamped in the machine, with force applied evenly along their axis. A deformation rate of 0.06 m/hr was used, and only samples that fractured within the designated working section were considered. The tensile strength (R_{cog}), in Pa, was calculated using:

$$\boldsymbol{R_{cog} = \frac{F_{Pi}}{S_{Oi}}}$$

where:

F_{Pi} : tensile load at rupture, N

S_{Oi}: initial cross-sectional area, m²

The elastic modulus (E) was derived from the stress-strain curve as the slope of the tangent to the initial linear section:

$$\boldsymbol{E = \frac{R'_{cogi}}{\varepsilon'_i} \cdot 100}$$

where:

R'_{cogi}: tensile strength at the point where the tangent detaches from the curve, Pa

$\boldsymbol{\varepsilon'_i}$: relative elongation at this point, m/m

Cohesive strength was calculated based on the axial tensile strength using samples with a cross-sectional area of 10×10×50 mm:

$$R = \frac{P}{F}$$

where:

P: failure force, N

F: initial cross-sectional area, m²

Crack formation in polymer coatings was assessed using the correlation between crack length, the Vickers indentation size, and fracture toughness. The critical stress intensity factor (K_{1c}) was calculated as follows:

$$K_{1c} = 0{,}028 HV a^{0{,}5} (E/HV)^{0{,}5} (C/a)^{-1{,}5}$$

where:

HV: Vickers hardness

C: half-length of radial cracks

a: half-length of the indentation diagonal

Frost resistance, a key durability measure for lime coatings in various climates, was also tested. For each analysis, six samples were prepared.

3. RESULTS AND DISCUSSION

The cohesive strength of the newly developed lime coatings was measured, and the results are presented in Table 1.

Table 1. Cohesive strength (tensile strength) of the lime samples

Composition	Cohesive Strength (MPa)	Standard Deviation (%)
Curing Age: 28 days		
Control (lime + water)	0.22	<1
AtrenCem LV (0.5% by weight of lime)	0.23	<1
AtrenCemHV (0.5% by weight of lime)	0.24	<1
AtrenCem LV (1% by weight of lime)	0.24	<1
AtrenCemHV (1% by weight of lime)	0.25	<1
Curing Age: 3 months		
Control (lime + water)	0.26	<1
AtrenCem LV (0.5% by weight of lime)	0.38	<1
AtrenCemHV (0.5% by weight of lime)	0.40	<1
AtrenCem LV (1% by weight of lime)	0.47	<1
AtrenCemHV (1% by weight of lime)	0.51	<1

These results indicate that the addition of Atren Cem LV and Atren Cem HV improves the tensile strength of lime-based coatings, likely due to structural changes in the lime matrix. The incorporation of these additives promotes the formation of inter- and intercrystalline organic macromolecules, enhancing the durability of the coatings. This structural improvement was confirmed through analysis using SEM images (Fig. 2).

The microstructural analysis revealed low-contrast, nanoscale areas within the calcite crystals, corresponding to the amorphous organic phase (Fig. 2b). The integration of polysaccharides into the calcite matrix was further confirmed through adsorption studies, which measured changes in the surface tension of the mortar with varying concentrations of AtrenCem LV.

As shown in Figure 3, increasing the concentration of AtrenCem LV reduces the surface tension of the solution until it stabilizes at around 1% concentration, achieving a surface tension of $56.62 \cdot 10^{-3}$ J/m². This stabilization indicates the saturation of adsorption sites. Similar behavior was observed for AtrenCemHV, which stabilized at $54.5 \cdot 10^{-3}$ J/m².

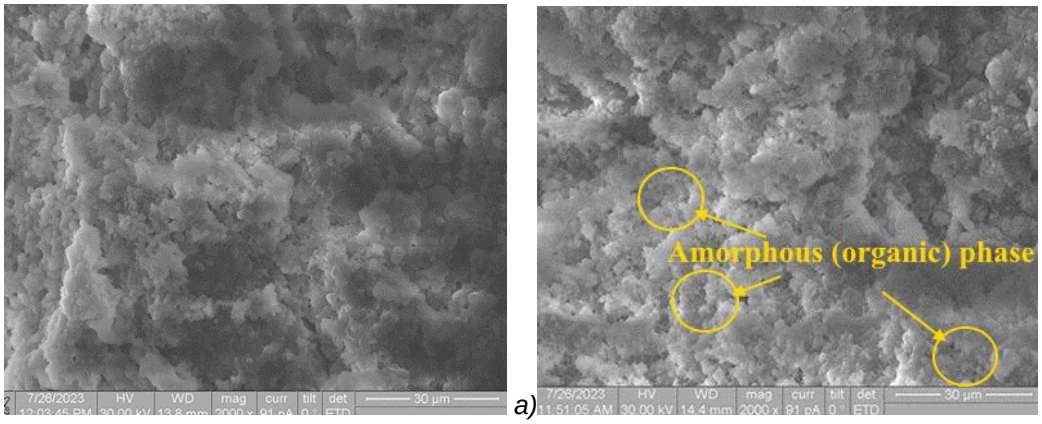

Figure 2. Structure of Lime Hardened Paste:(a)Control composition (lime + water); (b)Composition with AtrenCemHV (0.5%)

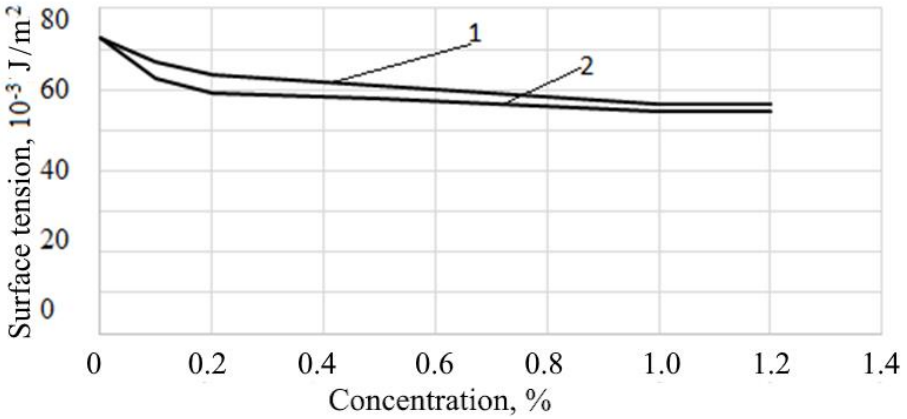

Figure 3. Change in Surface Tension of Polysaccharide Solution AtrenCem LV (1) AtrenCemHV (2)

Adsorption measurements demonstrated that the amount of AtrenCem LV adsorbed on lime after 15 minutes and 48 hours was similar, at approximately 0.909 g/g for a 1% solution. In comparison, AtrenCemHV showed a higher adsorption capacity of 1.1 g/g. The maximum adsorption values were determined to be 1.83 g/g for AtrenCemHV and 1.66 g/g for AtrenCem LV, as shown in Figure 4.

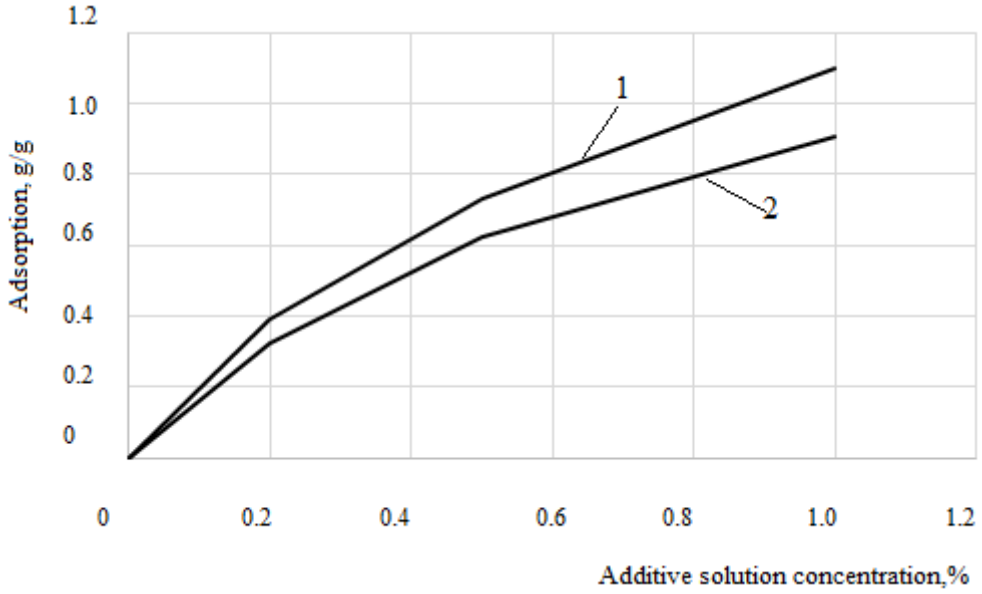

Figure 4. Adsorption of Polysaccharides on Lime Depending on Concentration (Atren Cem HV (1), Atren Cem LV (2))

These results suggest that the theory of monomolecular adsorption can be applied to describe the adsorption of Atren Cem LV and Atren Cem HV on lime surfaces.

The adsorption isotherm (Fig. 5) plotted in coordinates $\frac{1}{a_\infty b}$. C confirmed this relationship, allowing the calculation of the adsorption equilibrium constant b. For Atren Cem LV, b was calculated as $1.18^{1}/_{\%}$ and for Atren Cem HV, $1.30\ ^{1}/_{\%}$.

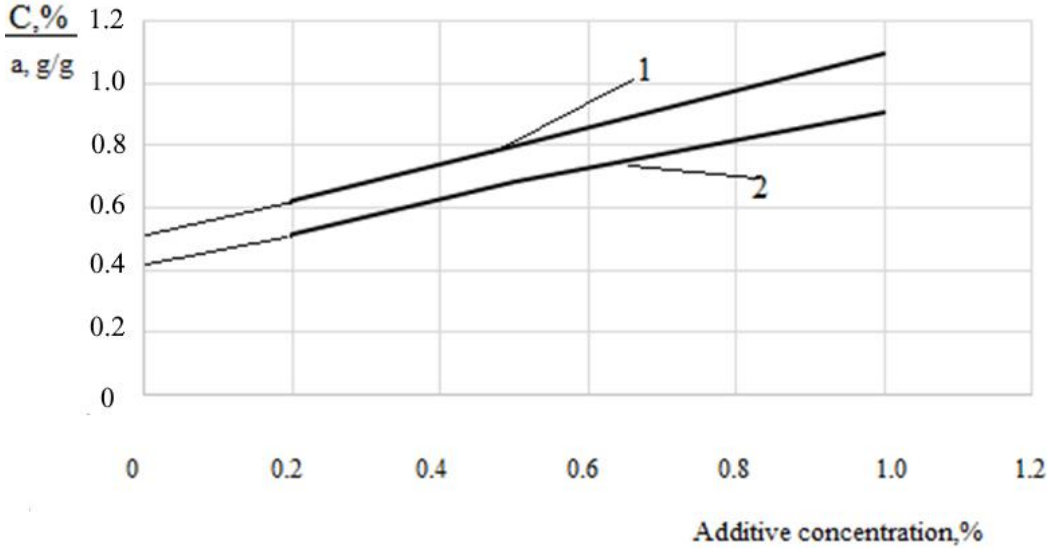

Figure 5. Adsorption Isotherm of Additives in Coordinates $1/a = f(C)$: AtrenCem LV (1); AtrenCemHV (2)

The study further showed that the presence of polysaccharides in the lime composite aids in the carbonization process, leading to a thicker carbonized layer. Figure 6 illustrates this carbonization, which starts from the surface and gradually progresses inward. Samples with AtrenCem LV showed better carbonization, indicated by the lighter coloration and a higher carbonized thickness at 21% humidity.

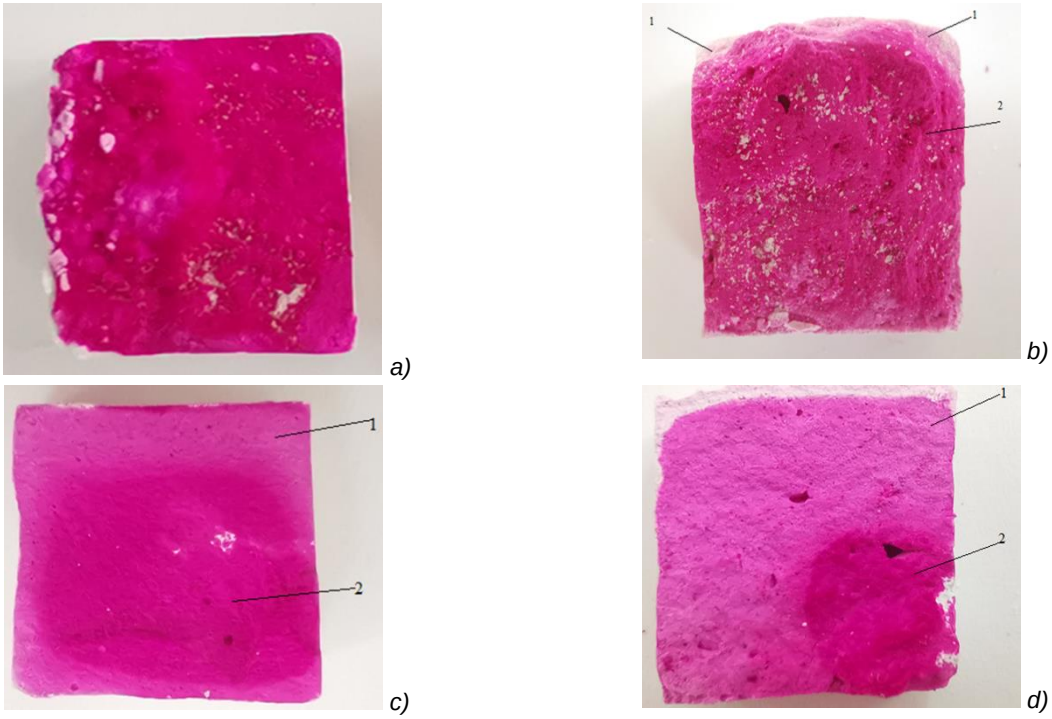

Figure 6. Carbonized Layer Changes in Lime Samples at 4 (a, b) and 10 (c, d) Days: (a, c) Control composition; (b, d) Composition with 1 wt. % AtrenCem LV;1 - the carbonated part is indicated in light color; 2 - the low-carbonated part is indicated in dark color

X-ray diffraction analysis confirmed the increased calcite content in modified samples, with 92.02% calcite in control samples and 96.21% in those with 1% AtrenCemHV (Fig. 7). This increase in calcite content suggests enhanced durability of the modified coatings.

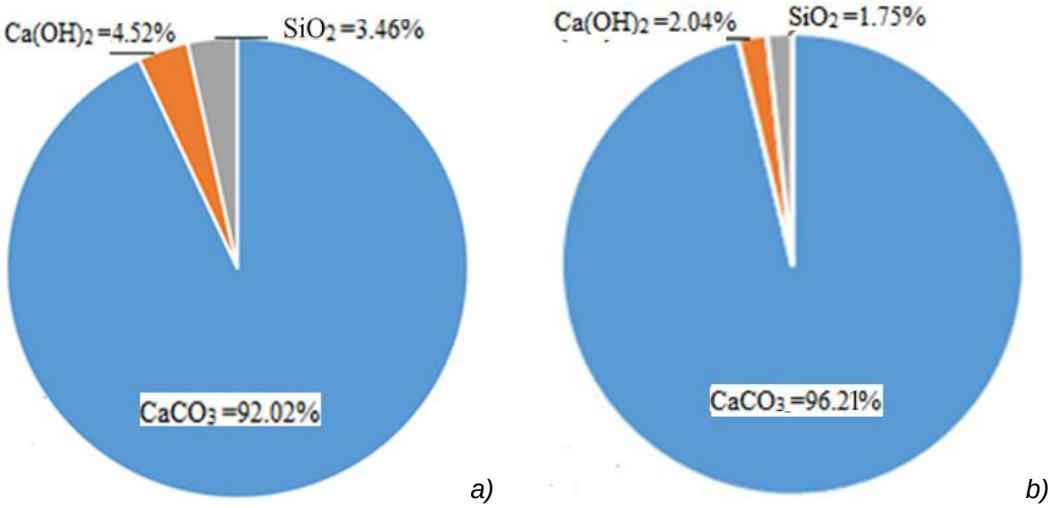

Figure 7. Mineral Content: (a) Control sample (lime) (b) Lime + 1 wt. % AtrenCemHV

The inclusion of organic molecules improves the deformation properties of the coatings. This is evident from the reduced elastic modulus of 0.925 MPa in samples with 1% AtrenCem LV, compared to 0.987 MPa in control samples (Fig. 8).

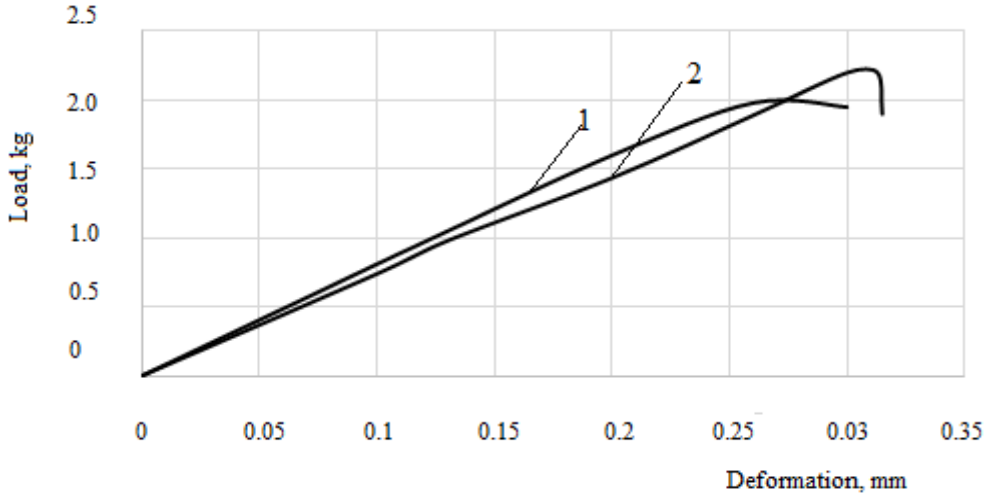

Figure 8.Tensile Deformations of Lime Coatings: (a) Control sample (lime) (b) Lime + 1 wt. % Atren Cem HV

Vickers hardness tests showed a reduction in hardness from 1.890 kgf/mm² in the control composition to 1.448 kgf/mm² with AtrenCem LV, leading to improved crack resistance. These modified lime coatings endured 35 freeze-thaw cycles without notable changes in their appearance.

In terms of resistance to freeze-thaw cycles, Table 2 provides the stress intensity factor (K_1C) and microhardness values after 25 cycles.

Measuring the hardness of coatings using the Vickers method showed that the Vickers hardness of coatings based on the control composition (without polysaccharide additives) is HV = 1.890kgf/mm², and that of coatings based on the composition with the addition of AtrenCem LV is 1.448 kgf/mm². This ensures higher crack resistance of coatings during operation. Lime coatings containing the AtrenCem LV additive in the formulation withstood 35 cycles of alternating freezing and thawing without significant changes in the quality of appearance.

When assessing the hardness of the coatings and the stress intensity factor after 25 freeze-thaw cycles, it was found that cracks appeared in the control lime coating already at a load of 2 kgf (Table 2).

Table 2. Stress Intensity Factor $K_{1C}$and Microhardness After 25 Freeze-Thaw Cycles

Load, kgf	Stress intensity factorK1C, MPa·m		Average diagonal length of imprint, mm		Average crack length, mm	
	Control sample without additives	Sample with additive Atren cem LV	Control sample without additives	Sample with additive Atren cem LV	Control sample without additives	Sample with additive Atren cem LV
0.5	0.00226	0.0020	0.7	0.8	0	0
1.0	0.002678	0.0027	1.0	0.9	0	0
1.5	0.00299	0.00303	1.2	1.1	0	0
2.0	0.010*	0.00321	1.5	1.3	0.7	0
2.5	-	0.00346	-	1.4	-	0
3.0	-	0.0266*	-	1.5	-	0.4

*Critical value of stress intensity factor K_{1c}.

In coatings with Atren Cem LV, cracks appeared only at a load of 3 kgf, while control samples cracked at 2 kgf. The critical stress intensity factor (K_{1C}) for control samples was 0.010 MPa·m^{½}, compared to 0.0266 MPa·m^{½} for samples with Atren Cem LV, demonstrating significantly improved crack resistance with the additive.

4. CONCLUSIONS

The development of lime compositions with polysaccharide additives for restoring building walls has led to the following key conclusions:

Structural Formation: The inclusion of polysaccharide additives Atren Cem HV and Atren Cem LV in lime composites facilitates the formation of a structure containing inter- and intercrystalline organic molecules. This is due to the adsorption of these additives on the lime surface, which enhances the elastic modulus and hardness of the lime coatings.

Surface Activity: The study confirmed the surface activity of Atren Cem LV and Atren Cem HV polysaccharides in aqueous solutions.

Adsorption Theory Application: It was demonstrated that the theory of monomolecular adsorption is applicable for describing the adsorption behavior of Atren Cem LV and Atren Cem HV on lime. The specific adsorption parameters for these polysaccharide additives were identified.

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Data Availability Statement

All data, models, and code generated or utilized during this study are included in the submitted article.

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IZVOD

KOMPOZICIJE KREČA SA POLISAHARIDIMA ZA RESTAURACIJU I ZAŠTITU ZIDOVA ZGRADA

Očuvanje istorijskog nasleđa je značajan prioritet u savremenom urbanističkom planiranju. Međutim, poboljšanje trajnosti krečnih premaza koji se koriste u restauraciji lokaliteta kulturne baštine ostaje izazov. Cilj ove studije je da se identifikuje metod za poboljšanje otpornosti na pucanje premaza na bazi kreča korišćenjem polisaharidnih aditiva. Studija ispituje modifikovane polisaharide rastvorljive u vodi AtrenCemHV i AtrenCem LV. Određene su maksimalne vrednosti adsorpcije ovih aditiva na kreču, sa AtrenCemHV na 1,83 g/g i AtrenCem LV na 1,66 g/g. Rezultati ukazuju da adsorpcija polisaharida na površinu čestica kreča Ca(OH)2 dovodi do formiranja kompozitne strukture koja sadrži inter- i interkristalne organske molekule, što poboljšava otpornost premaza na pucanje. Istraživanje takođe pruža uvid u proces karbonizacije krečnih premaza koji sadrže polisaharidne aditive, otkrivajući da ovi aditivi povećavaju debljinu karbonizovanog sloja. Krečne kompozicije sa polisaharidima pokazuju veću kohezionu čvrstoću zbog visokog sadržaja kalcita. Pored toga, formiranje kompozitne strukture sa organskim molekulima doprinosi smanjenju modula elastičnosti i tvrdoće premaza. Krečni premazi sa aditivom AtrenCem LV izdržali su 35 ciklusa smrzavanja i odmrzavanja. Stoga su nova saznanja o sastavima kreča sa aditivima polisaharida ključna za restauraciju zidova zgrada.

Ključne reči: *kreč, polisaharidi; monomolekularna adsorpcija, karbonizacija; otpornost na pucanje, premazi*

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