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Self-cleaning capacity of photocatalytic building plasters under frost attack

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ABSTRACT

Introduction. The durability of the self-cleaning capacity of photocatalytic building materials under real operating conditions is a crucial issue, as their efficiency decreases over time due to surface degradation and carbonation. The purpose of the research is to evaluate the stability of the photocatalytic activity in two types of plasters – gypsum-cement-pozzolan plaster (based on red gypsum) and cement plaster (with anatase photocatalyst) – to cyclic freezing and thawing, as well as to investigate the effect of water-reducing and pozzolan additives on maintaining their self-cleaning ability. **Materials and Methods.** Series of plaster samples were prepared with and without different combinations of additives. Photocatalytic activity was assessed using the rhodamine test. Changes in the materials were analyzed using compressive strength, density, and water absorption test methods. Scanning electron microscopy with energy-dispersive spectrometry was used to measure the titanium (photocatalyst marker) and calcium (carbonation marker) content on the surface. Destructive frost effects were simulated by the cyclic freezing and thawing of samples in a water-saturated state. **Results.** It was found that the primary mechanism causing the loss of the self-cleaning capacity was photocatalyst washout due to surface degradation. Shielding of the photocatalyst by carbonation products is also crucial for cement plasters. Water-reducing additives increased the initial self-cleaning efficiency by 45% due to structure compaction, which slowed surface degradation. Pozzolan additives reduced surface calcium content by 6–8%, suppressing carbonation and almost doubled the initial efficiency. The combined use of these additives demonstrated the best results in maintaining photocatalytic activity after freezing and thawing. **Discussion and Conclusion.** The durability of the self-cleaning capacity directly depends on the resistance of the carrier material to climatic impacts. Combined modification with water-reducing and pozzolan additives is the most effective strategy for improving the durability of self-cleaning plasters, as it simultaneously counteracts two key degradation mechanisms: physical washout of the photocatalyst and its chemical shielding by carbonates. This study provides a practical approach to developing more sustainable photocatalytic building materials.

KEYWORDS: Mortar, mixtures, photocatalytic plasters, photocatalytic additives, anatase, self-cleaning, frost resistance

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Самоочищение фотокаталитических строительных штукатурок при морозной агрессии

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АННОТАЦИЯ

Введение. Долговечность самоочищающейся способности фотокаталитических строительных материалов в реальных условиях эксплуатации является критически важной проблемой, поскольку их эффективность со временем снижается из-за деградации поверхности и карбонизации. Целью данного исследования была оценка устойчивости фотокаталитической активности двух типов штукатурок – гипсо-цементно-пуццолановой (на основе красного гипса) и цементной (с фотокатализатором анатазом) – к циклическому замораживанию-оттаиванию, а также изучение влияния водоредуцирующих и пуццолановых добавок на сохранение их самоочищающейся способности. **Материалы и методы.** Были приготовлены серии образцов штукатурок с различными комбинациями добавок и без них. Фотокаталитическая активность оценивалась с помощью родаминового теста. Для анализа изменений материалов использовались методы определения прочности на сжатие, плотности, водопоглощение, а также растровая электронная микроскопия с энергодисперсионной спектрометрией для измерения содержания титана (маркер фотокатализатора) и кальция (маркер карбонизации) на поверхности. Моделирование разрушающего действия мороза проводилось путем циклического замораживания-оттаивания образцов в водонасыщенном состоянии. **Результаты.** Установлено, что основной механизм потери самоочищающейся способности связан с вымыванием фотокатализатора вследствие деградации поверхности. Для цементных штукатурок значительную роль играет также экранирование фотокатализатора продуктами карбонизации. Водоредуцирующая добавка повышала начальную эффективность самоочищения на 45% за счет уплотнения структуры, что замедляло деградацию поверхности. Пуццолановая добавка снижала содержание поверхностного кальция на 6–8%, подавляя карбонизацию, и обеспечивала почти двукратный рост начальной эффективности. Совместное применение добавок показало наилучшие результаты по сохранению фотокаталитической активности после замораживания-оттаивания. **Обсуждение и выводы.** Долговечность самоочищающейся функции напрямую зависит от устойчивости материала-носителя к климатическим воздействиям. Комбинированное модифицирование водоредуцирующей и пуццолановой добавками является наиболее эффективной стратегией для повышения долговечности самоочищающихся штукатурок, так как одновременно противодействует двум ключевым механизмам деградации: физическому вымыванию фотокатализатора и его химическому экранированию карбонатами. Это исследование предлагает практический подход к разработке более устойчивых фотокаталитических строительных материалов.

КЛЮЧЕВЫЕ СЛОВА: Растворы, смеси, фотокаталитические штукатурки, фотокаталитические добавки, анатаз, способность к самоочищению, морозостойкость

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INTRODUCTION

Self-cleaning photocatalytically active building materials both help to keep surfaces clean and improve the aesthetics of the urban environment, as well as reduce the amount of harmful organic compounds. This has a positive impact on the environment and human health [1–4]. The widespread practical application of these materials has become possible due to the development of science in this field, in particular, the production of more effective and cost-efficient multicomponent photocatalysts based on titanium oxide [5–12], as well as the use of natural minerals and industrial waste as a source of photocatalysts [13, 14]. Examples of such implementations include the Dives in Misericordia Church in Rome (Italy, 2003), the Cité de la Musique et des Beaux Arts music and art town hall in Chambéry (France, 2001), the Residentie Commodore residential complex in Ostend (Belgium, 2007), the Pavilions of Expo 2015 in Milan (Italy, 2015) [15–18]. Pilot projects using self-cleaning paving tiles, such as the installation of 10,000m^2 of photocatalytic paving blocks in Antwerp, Belgium, and the use of a photocatalytic wall solution in Guerville, France, have confirmed the effectiveness of the technology, demonstrating a significant reduction in the concentration of polluting oxides [19, 20]. Data accumulated over more than a decade of operating these facilities allow evaluating their long-term effectiveness and identifying problems. For example, the investigation of the Dives on Misericordia Church in Rome reveals that façades made of a material based on photocatalytic concrete with titanium oxide do not exhibit high self-cleaning efficiency after 16 years. The façade blocks exhibited the following defects: small and large cracks, stuck contaminants, surface degradation, and a large number of microorganisms destructing the material. According to researchers, this resulted from a combination of design factors (the complex configuration of double-curved concrete blocks and an ill-conceived drainage system), as well as the impact of operating conditions (a large amounts of pozzolan sand within the location of the structure and frequent heavy rainfall). Although the photocatalytic activity of the added titanium oxide was expectedly maintained over time, the self-cleaning efficiency was significantly affected by structural changes under the influence of external climatic and natural factors [21]. Similar changes—discoloration and cracking—were also observed on the facades of the building in Ostend [22]. The studies confirm the difference between testing self-cleaning materials in laboratory and real-world conditions, as well as the need to investigate the durability of the self-cleaning capacity under the influence of weather factors, which are aggressive for photocatalytic surfaces.

The efficiency of photocatalysts in building materials depends on three groups of factors: the physicochemical properties of the photocatalyst (band gap, pH) [23], process parameters (surface area, uniformity of distribution and resistance to photocatalyst agglomeration) [1, 3, 22], and the composition and structure of the carrier material (porosity, roughness, presence of impurities) [3, 6, 24]. During operation, factors related to the material structure become the key causes of deactivation. In cement-based systems, the photocatalyst is 'shielded' by non-degradable inorganic compounds, such as calcium carbonate [25–27]. Climatic conditions also contribute negatively to changes in the self-cleaning ability of materials. Cyclic freezing and thawing, especially in a humid environment, leads to increased surface roughness, changes in porosity, the formation of microcracks and, consequently, to the degradation of the material structure. This weakens the bond between the photocatalyst and the matrix, leading to its washout from the surface [28–32]. Thus, the main reasons for the decrease in the self-cleaning capacity of photocatalytic concretes during operation are the physical washout of the photocatalyst and its shielding by material degradation products or non-degradable deposits.

This article examines changes in the self-cleaning capacity of photocatalytic plasters under frost attack.

METHODS AND MATERIALS

Changes in self-cleaning performance under frost attack were studied on two types of self-cleaning plasters: gypsum-cement-pozzolan and cement.

Impurity compounds naturally present in red gypsum, such as anatase, rutile, and iron oxide, were used as the photocatalytic component in the gypsum-cement-pozzolan plaster [14, 33–35]. Red gypsum has a normal density of 60%, compressive strength of 3 MPa, and grinding fineness of 1–2% with a 0.2 mm screening section. In addition to red gypsum (22%), the composition also included CEM I 42.5 N cement (manufactured by Lafarge Cement OJSC, Russia) in an amount of 13.2% and MKU-85 microsilica (manufactured by Kuznetsk Ferroalloys JSC, Russia) in an amount of 4.8% by weight, as well as fine sand, in an amount of 60%. The structure of the gypsum-cement-pozzolan plaster was modified using Melflux 5581F polycarboxylate water-reducing additive (BASF Construction Additives, Germany) to compact the structure of the material. Citric acid monohydrate E-338 (Foodchem International Corporation, China) was also used in all plasters at a dosage of 0.1% above the gypsum mass, to slow the setting time. The characteristics of gypsum-cement-pozzolan plaster are described in detail in [14].

The photocatalyst used in the cement plaster is an anatase-modified titanium oxide additive (particle size 7–10 nm, specific surface area 248 m^2/g) (Anhui Fitech Material Co., China). The photocatalyst was added at a dosage of 2% by weight of cement. This dosage was chosen during the preliminary experiment as the lowest and most effective. To modify the cement plaster, we used MKU-85 microsilica pozzolan additive (Kuznetsk Ferroalloys, Russia), which binds free calcium hydroxide in the hardened cement paste and reduces the degree of surface carbonation. We also used SP-1 water-reducing naphthalene sulfoformaldehyde additive (Polyplast, Russia) to compact the material structure and surface. The plaster consisted of 45% Portland cement and 55% fine sand. The types and amounts of modifying additives in plaster formulations are listed in Table 1.

This study examined the compressive strength of plasters determined through destructive testing, as well as their self-cleaning ability using the rhodamine test and density and water absorption measurements. The surface structure was examined using electron microscopy with a JSM-7001F scanning electron microscope (JEOL, Ja-

Table 1. Modifying additives in plaster formulations

Short name of sample series	Type of plaster	Type of modifying additive	Name of additive	Dosage, % of binder weight	True water-to-binder ratio
GCP	Gypsum-cement-pozzolan	N/a	–	–	0.60
GCP+W		Water-reducing	Melflux 5581F	0.8	0.30
C	Cement	N/a	–	–	0.45
C+W		Water-reducing	SP-1	0.8	0.32
C+P		Pozzolan	MKU-85	8	0.46
C+W+P		Water-reducing	SP-1	0.8	0.35
		Pozzolan	MKU-85	8	

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Нанотехнологии в строительстве
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5–14

pan). The elemental composition of the sample surfaces, specifically the titanium (anatase marker) and calcium (surface carbonation marker) content, was analyzed using an X-max 80 energy-dispersive spectrometer (Oxford Instruments, UK) mounted on a JSM-7001F scanning electron microscope (JEOL, Japan).

The process of freezing in a water-saturated state and thawing simulates the destructive effects of frost.

Gypsum-cement-pozzolan plaster samples, which demonstrated frost resistance of 10 and 50 cycles based on property testing, were subjected to 50 freeze-thaw cycles in a water-saturated state. The surface properties and characteristics of the samples were determined after 5, 10 cycles, and then every 10 cycles. Cement plaster samples, depending on their composition, had frost resistance of 50 to 100 cycles. Their properties and surface characteristics were determined after 50, 75, 100, and 150 freeze-thaw cycles.

RESULTS

Figure 1 presents graphs of changes in plaster properties and the elemental composition of their surfaces under frost attack.

The analysis of the mechanical test results showed fundamental differences in the behavior of the modified and unmodified gypsum-cement-pozzolan plasters during cyclic freezing and thawing. While the control plaster samples exhibit a progressive decrease in strength, with its almost complete loss by 20 cycles (Fig. 1a, blue curve), the addition of a water-reducing additive ensures that the strength remains above the initial level throughout the entire testing period, with only a downward trend after 20 cycles (Fig. 1a, orange curve).

The initial self-cleaning ability is almost independent of the presence of a water-reducing additive. However, there is a noticeable progressive decrease in this ability during cyclic freezing and thawing (Fig. 1c). The dynamics of this decrease correlates closely with changes in the titanium content on the sample surface (Fig. 1e). The calcium concentration, however, remains almost unchanged (Fig. 1g). Micrographs of the surface reveal that the unmodified samples show noticeable destruction of the surface microstructure after only five freeze-thaw cycles (Fig. 2b). In contrast, a similar degree of destruction is observed in the water-reduced samples only after 30 freeze-thaw cycles (Fig. 2d).

The introduction of modifying additives to cement-based plasters had a significant impact even on the initial self-cleaning efficiency. The introduction of a water-reducing additive significantly altered the material properties, leading to a 45% increase in the initial self-cleaning efficiency (Fig. 1d, 0 cycles, orange dot). This increase corresponds to an observed 2% increase in sample density and a 30% decrease in water absorption. The pozzolan additive resulted in a nearly twofold increase in self-cleaning efficiency (Fig. 1d, 0 cycles, gray dot) compared to the control sample (Fig. 1d, 0 cycles, blue dot) and contributed to a 6–8% reduction in the surface calcium content compared to the unmodified formulation (Fig. 1h, 0 cycles, gray and blue dots).

The cement plaster samples without modifying additives exhibit a rather sharp decline in self-cleaning efficiency from the first freeze-thaw cycles (Fig. 1d, blue line). This regression is accompanied by a decrease in the titanium concentration on the surface (Fig. 1f, blue line) and an increase in the calcium concentration (Fig. 1h, blue line). After 50 freeze-thaw cycles, micrographs of the surface show significant surface degradation and the presence of numerous carbonate deposits measuring between 12 and 10μm in size (Figs. 3a, 3b).

Water reduction in cement plasters and the introduction of a pozzolan additive have different effects on the surface degradation process and the nature of the decline in self-cleaning efficiency. The use of a water-reducing additive results in a more gradual decline in self-cleaning efficiency (Fig. 1d, orange line) compared to the samples with a pozzolan additive (Fig. 1d, gray and yellow lines). With water reduction, the decrease in the titanium content on the surface is also more gradual and slower (Fig. 1f, orange curve), compared to the samples without water reduction (Fig. 1f, blue and gray lines). The calcium concentration curve shows a steady increase (Fig. 1h, orange line). After 100 freeze-thaw cycles, micrographs show large calcite formations on the surface (Figs. 3c, 3d).

The cement plaster samples containing a pozzolan additive exhibit the most distinctive pattern of surface changes. Self-cleaning efficiency drops sharply after the first 50 freeze-thaw cycles (Fig. 1d, gray and yellow lines), as does the surface titanium concentration (Fig. 1f, gray line). After that, both indicators gradually decrease, and the surface calcium concentration even decreases slightly during frost attack (Fig. 1h, gray and yellow lines). After 100 freeze-thaw cycles, micrographs of the samples with a pozzolan additive show isolated, rare calcium carbonate formations with significant surface degradation (Figs. 3e, 3h).

Maximum final self-cleaning efficiency (Fig. 1d, yellow line), as well as a higher surface titanium content (Fig. 1f, yellow line) and a low calcium content (Fig. 1h, yellow line) are observed when both water-reducing and pozzolan additives are used. After 100 freeze-thaw cycles, the surface structure of the samples appears to be more preserved, with individual calcium carbonate formations measuring 1–5 μm (Fig. 2i, 2j).

DISCUSSION

The slight increase in the strength of water-reduced gypsum-cement-pozzolan plaster after 5–20 freeze-thaw cycles is likely explained by the fact that some unhydrated

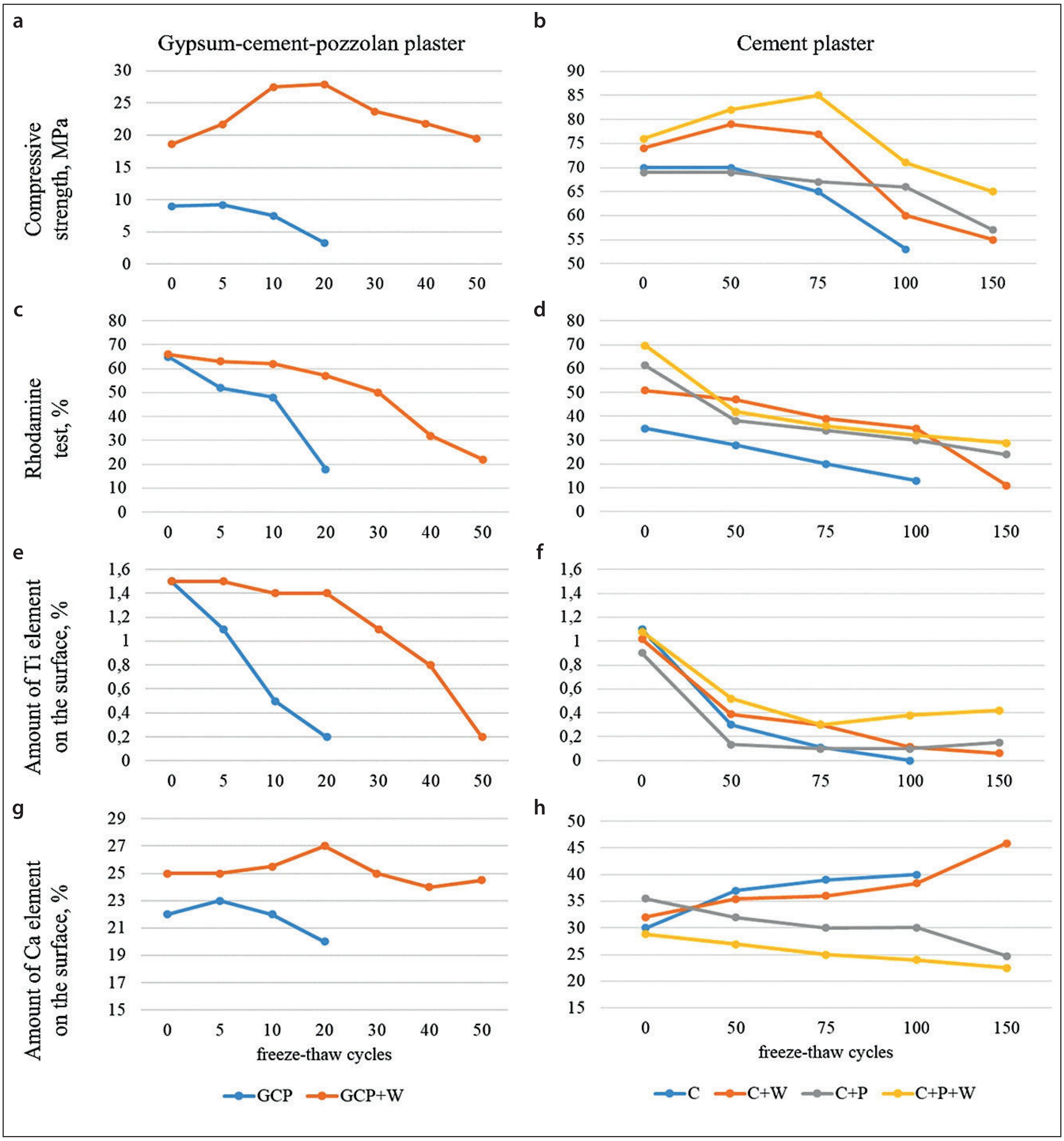

Fig. 1. Changes in the properties of plasters and the elemental composition of their surfaces under frost attack

cement minerals remain in the hardened matrix because of a slight water deficit (as confirmed by XRD). The gypsum component provides the necessary porous structure, allowing water to penetrate into the material and initiate hydration processes in cement minerals [36]. The combination of increased strength during cyclic freezing and a decrease in photocatalytic activity with visually observable surface degradation suggests the following frost damage mechanism. The primary damage is surface destruction caused by the dissolution and washout of less stable gypsum compounds. Since photocatalyst particles are not strongly bonded to the matrix, this partial surface destruction leads to their mechanical removal. Thus, the process of photocatalyst 'washout' is the main factor leading to the reduction in the self-cleaning ability of gypsum-cement-pozzolan plasters.

A similar process of photocatalyst washout with surface texture degradation was also observed in the cement

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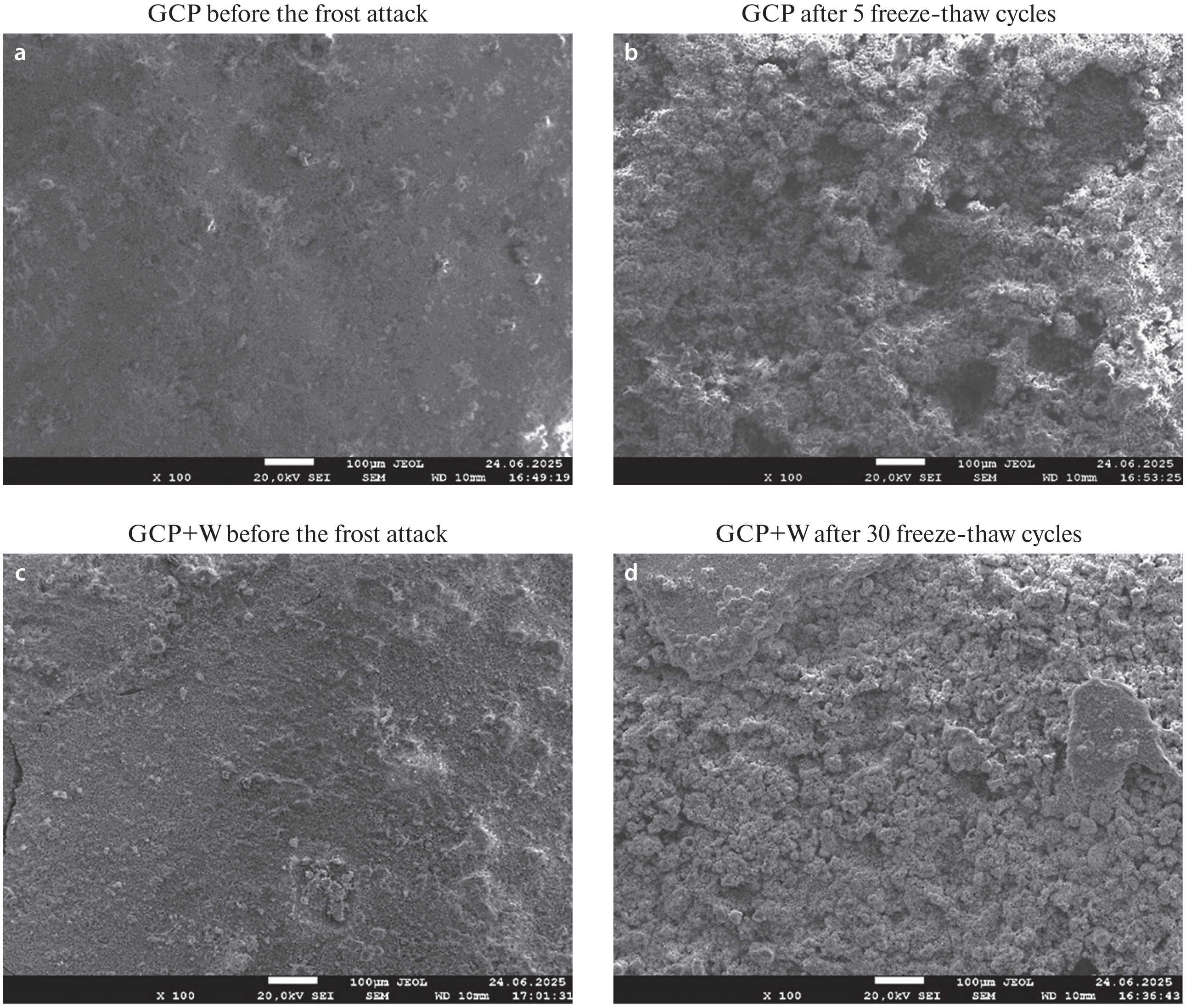

Fig. 2. Micrographs of the surface (×100) of gypsum-cement-pozzolanic plaster samples before and after frost attack

plaster samples without modifying additives. An additional negative factor in this case is active surface carbonation. The formation of carbonates over a large area shields the photocatalyst from UV radiation and 'clogs' the surface, thereby reducing the self-cleaning ability. The introduction of a water-reducing additive compacts the cement plaster surface layer, which slows the photocatalyst washout rate and ensures a smoother decline in the self-cleaning ability. However, the compacted surface does not appear to impede carbonation, which becomes the dominant negative factor in this case, shielding the photocatalyst and preventing the achievement of high absolute values of self-cleaning ability. In contrast, pozzolan additives do not significantly affect the kinetics of anatase washout, but they effectively limit surface carbonation due to free calcium hydroxide binding during the curing process. This gives materials containing a pozzolan component an advantage in long-term retention of their photocatalytic properties compared to formulations without the additive.

The combined use of water-reducing and pozzolan additives ensures maximum final self-cleaning efficiency with most gradual decline in dynamics. This result is achieved through a combination of independent mechanisms: the compacting and strengthening effect of water-reducing additives, which slows physical surface degradation and photocatalyst washout; and the limited surface carbonation due to pozzolan additives, which maintains the accessibility of the photocatalyst to activating UV radiation.

CONCLUSION

The cyclic freezing and thawing method has proven to be suitable for modeling the aging of self-cleaning cement-based plasters. It identified two main mechanisms

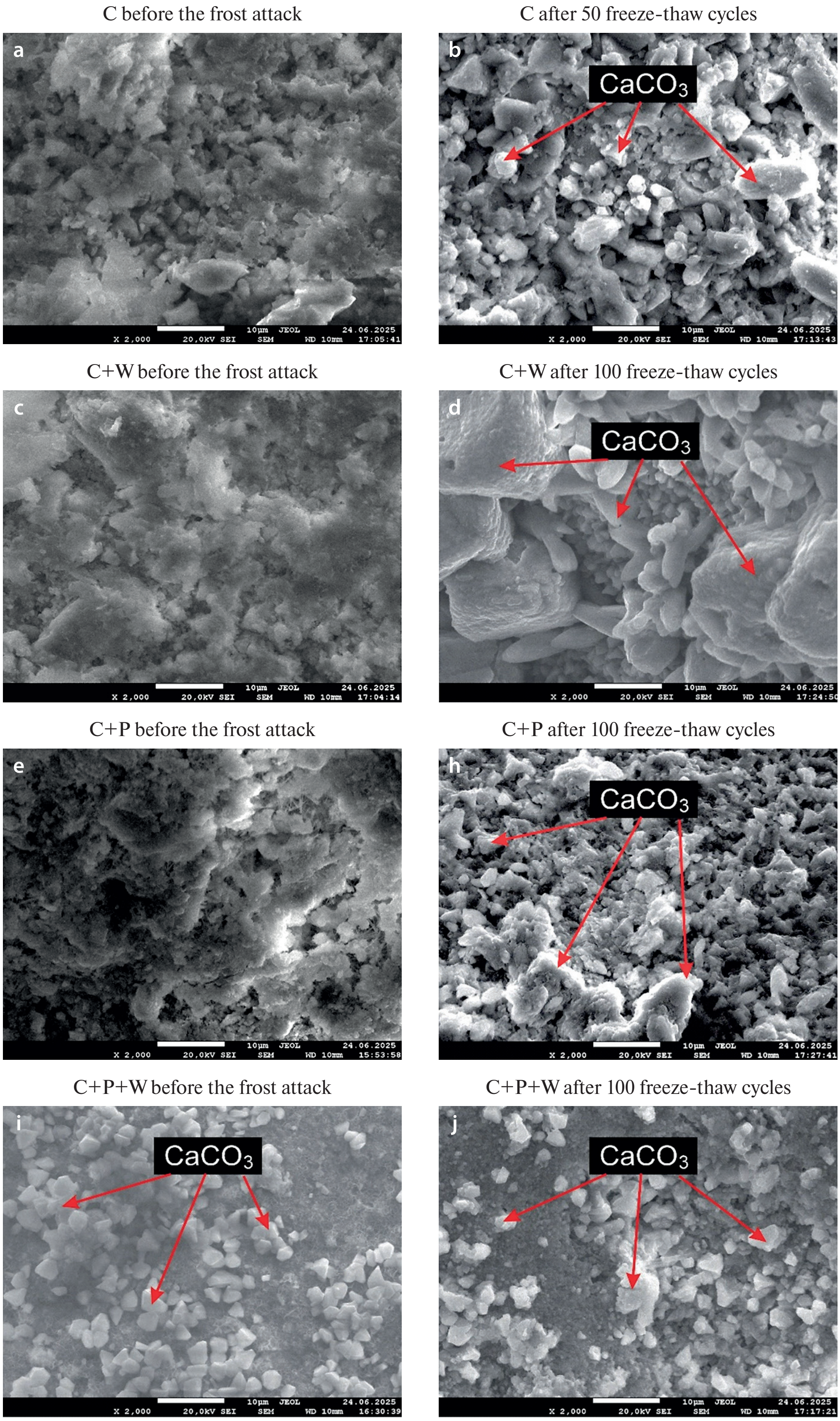

Fig. 3. Micrographs of the surface (×100) of cement plaster samples before and after frost attack

that contribute to the decline in the self-cleaning ability: photocatalyst washout due to surface degradation and its shielding due to carbonation. The studies demonstrate that the durability of the self-cleaning performance depends significantly on the stability of the carrier material and its microstructural characteristics.

The combined use of modifying additives demonstrated the maximum effectiveness in maintaining the self-cleaning ability. Water-reducing additives help to compact the surface structure, slow surface erosion, and prevent photocatalyst washout. Pozzolanic additives, in turn, effectively suppress carbonation by binding free calcium hydroxide, thereby improving the durability of the plaster. The combined effect of these additives ensures maximum initial self-cleaning efficiency and retention after frost exposure.

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ADDITIONAL INFORMATION

The authors declare that neither generative artificial intelligence technologies nor artificial intelligence-based technologies were not used in the preparation of this article.

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Polina I. Kiiko – methodology development; drafting; participation in curriculum development and implementation.

Tamara N. Chernykh – scientific management; research concept; final conclusions.

Mikhail V. Kriushin – participation in curriculum development and implementation.

Jiao Wang – participation in curriculum development and implementation.

Aleksandr A. Orlov – participation in curriculum development and implementation; follow-on revision of the text.

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