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Silane-based injection methods for moisture protection in ceramic bricks

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Abstract. Proper understanding of the mechanisms that lead to excessive moisture, as well as the development of effective methods for cutting off the flow of moisture to walls, is crucial for maintaining the durability and aesthetics of brick buildings, including historical ones. This issue is important in the context of protecting material cultural heritage and is consistent with the goals of sustainable development. The article presents the results of preliminary tests of the effectiveness of the protection against moisture of ceramic bricks, which was made using an injection method and a silane-based injection cream. Two injection methods were used: pressure and gravity, and the injections were performed in bricks with three levels of moisture: a permissible moisture content (3%), a moderate moisture content (7%), and a high moisture content (11%). The mass moisture content was determined using the gravimetric method, the spread of the injection agent in the brick was examined using the drop method and scanning electron microscopy, and the chemical composition of the bricks and injection cream was analyzed using X-ray fluorescence. The results of the tests showed that in the case of the analyzed samples, the most effective method was gravity injection applied at the permissible moisture content, while the least effective was gravity injection applied at a high moisture content of the bricks. The biggest difference in effectiveness between the injection methods was observed in high moisture content samples, where pressure injection proved to be significantly more effective. Confirmation of the results obtained requires further research.

Keywords: protection against moisture; ceramic brick; saline-based injection cream; injection methods; preliminary tests.

1. INTRODUCTION

Ceramic brick, one of the oldest and most traditional building materials, has played a key role in shaping the architectural landscape of cities for centuries [1]. The earliest fired bricks are dated to approximately 3500 BCE, with the first known brick-built structures – primarily temples and monumental buildings – appearing in around 2500–2000 BCE in Mesopotamian culture. Thanks to its durability, aesthetic qualities, and thermal mass, ceramic brick has remained a widely used construction material across diverse climates and architectural traditions [2] and continues to be valued in contemporary architecture to this day.

Ceramic brick is a porous material that can absorb water by capillary action. This mechanism is well-known and is described by Kubik [3], Hall [4], and Raimondo [5], among others. Therefore, the improper protection of the brick walls of buildings against free access to moisture results in their excessive dampness [6, 7]. As construction practice shows, the problem of excessive moisture is usually complex [8] and may be a result of many causes, including the leakage or a lack of moisture insulation, which was virtually nonexistent until the early 20th century. The effect of excessive moisture is the degradation of both the bricks and mortar that make up the wall, which leads to a decrease in its mechanical strength and thermal insulation, among others. Moreover, it contributes to the appearance of mold, fungi, and other harmful microorganisms on the wall surface [9]. These biological agents not only pose a threat to the integrity of construction materials but also to human health. Figure 1 shows examples of brick buildings located in various European cities that are affected by the problem of excessive moisture.

Proper understanding of the mechanisms that lead to excessive dampness, as well as the development of effective methods of counteracting this problem, is crucial for maintaining the durability and aesthetics of brick buildings [10]. In a situation where the source of dampness is capillary water rising from the ground, it is often necessary to make secondary damp-proof insulation in the wall. Such insulation is very often implemented in historical buildings, many of which are completely devoid of damp-proof insulation. It plays a key role in protecting these buildings from the destructive effects of moisture, while at the same time ensuring their durability and continued usage. Such protection against moisture also fits into a broader strategy of designing, building, operating, and maintaining environmentally friendly urban solutions that aim to reduce energy consumption by minimizing heat loss. This aligns with the principles of sustainable development and the long-term preservation of built heritage.

Secondary damp-proof insulation is commonly performed using the injection method. It involves the introduction of special agents into the brick wall through previously made boreholes [11]. Injection agents spread into the material through a process of penetration and diffusion, filling pores, and capillaries in the substrate structure [12]. Depending on the type of injection – pressure or gravity – the substance can be injected

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(a) (b)

(c) (d)

Fig. 1. Fragments of brick walls with visible traces of destruction caused by moisture and frost: (a) the wall surrounding Pardubice Castle (Czech Republic); (b) the external wall of a residential building in Kaunas (Lithuania); (c) a residential building in Bergen (Norway); (d) the building of Collegium Marianum in Pelplin (Poland)

actively or spread by absorption. The pressure method involves injecting the injection agent under pressure, which allows it to penetrate the wall quickly and deeply. In the gravity method, the agent penetrates the wall thanks to the action of gravity and capillary action. This process is therefore less invasive and slower. After application, a chemical reaction takes place, as a result of which the injection agent forms a permanent hydrophobic barrier. This changes the properties of the material, reducing its ability to absorb water and preventing its capillary rise.

There are many different injection agents available on the market, but in recent years those with a creamy consistency have been gaining popularity [13]. Their slow penetration into the wall structure allows for a more even distribution of the preparation, which theoretically increases the effectiveness of the protection against moisture.

Previous studies described in literature concerning the effectiveness of secondary damp-proof insulation using injection creams were most often performed in situ and focused on comparing the effectiveness of injections applied to sections of brick or brick-stone walls in historic buildings – for example, in structures located in Adelaide (Australia) [14], a former sugar factory in Ferrara (Italy) [15], and a damp church in Košice (Slovakia) [16]. However, researching existing buildings limited the possibility of obtaining detailed knowledge on the way the injection spreads in the wall structure and made it impossible to control the factors that may have an impact on it. In contrast, laboratory tests on injection creams, described in [16–18], were conducted on test walls made of ceramic brick and lime-cement mortar or cement mortar, which had high moisture content. Evaluating the effectiveness of injection agents in a single moisture condition does not provide a comprehensive understanding of their behavior under real-world conditions, where, as construction practice shows, the humidity level of a brick wall can vary significantly along its length. Hence, incorporating multiple moisture levels in the research plan allows for a more accurate assessment of the effectiveness of the applied methods and a deeper insight into the mechanisms governing their performance.

Extensive research on the effectiveness of moisture insulation by injection in ceramic bricks with different moisture levels was described in [19]. First, the tests were performed on solid ceramic bricks soaked in water at three different moisture levels: 20%, 50% and 80%. In these cases, the gravity injection method was used. Additionally, experiments were conducted on clay brick walls with lime-trass and lime mortars moistened to moisture levels of 20%, 50% and 100%. In the case of the first wall, both pressure and gravity injection techniques were used, and in the case of the second, the low-pressure injection method was used. In each of these tests, different injection agents were used, such as silicates, microemulsions of different concentrations, methyl silicates, and hydrophobic agents. However, the silane-based injection cream was not tested.

This article aims to present the results of preliminary research on the effectiveness of protection against moisture of solid ceramic bricks with various degrees of moisture, performed by the injection method using a silane-based injection cream and two injection methods: pressure and gravity. To date, such studies have not been conducted.

Therefore, the following research questions were formulated:

- Does the method of silane-based injection (pressure vs. gravity) affect the effectiveness of moisture protection?
- How does the moisture level of the brick at the time of the silane-based cream application affect the effectiveness of the moisture protection?

Knowledge in this area can contribute to the design of more effective and improved strategies for brick wall anti-moisture protection with the use of the silane-based injection method, in turn contributing to an increase in the durability of buildings and the improvement of their functionality in accordance with the principles of sustainable development.

2. MATERIALS AND METHODS

Twenty-one modern solid ceramic bricks measuring $250\times120\times$ 65;mm and with an average water absorption rate of approximately 15% were used for preliminary tests to assess the effectiveness of saline-based moisture protection using the injection (gravity vs. pressure) method.

To determine the chemical composition of the examined brick, X-ray fluorescence (XRF) analysis was performed on a sample taken from a randomly selected reference brick. The obtained ED-XRF spectrum revealed distinct peaks corresponding to elements such as silicon (Si), aluminum (Al), iron (Fe), calcium (Ca), potassium (K), and titanium (Ti), which confirmed their presence in the material structure. The highest intensity was recorded for the Si Kα line (~ 1.75 keV), indicating a dominant content of silica (Fig. 2).

Quantitative analysis of the elemental composition of the examined brick (Table 1) showed that the main component of the sample is silicon dioxide (SiO_2), with a concentration of 37.57% m/m, which is typical for ceramics fired from clay. Significant amounts of Al_2O_3 (7.25%) and Fe_2O_3 (3.26%) may influence the mechanical properties and coloration of the material. The presence of CaO (0.55%) and K_2O (1.22%) may affect the chemical reactivity of the brick in alkaline environments.

Elemental analysis of the silane-based injection cream, containing approximately 80% of active ingredients, was performed using inductively coupled plasma optical emission spectrometry (ICP-OES) in accordance with PN-EN ISO 11885:2009. This technique facilitates the simultaneous quantification of multiple elements with high sensitivity and precision. The samples were subjected to chemical etching and then analyzed to determine the concentrations of selected metals and metalloids, including Ca, Fe, Zn, and Al. The elemental composition of the injectable cream is presented in Table 2.

First, all the bricks were dried to a constant mass. For this purpose, they were placed in a laboratory dryer at a temperature of 105°C, with their weight being checked every 24 hours. After obtaining a constant mass, the bricks were removed from the dryer and divided into three groups (labeled as I, II, and

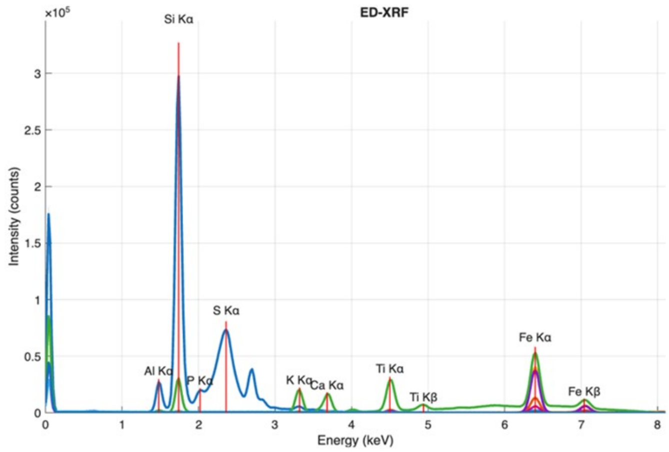


Fig. 2. ED-XRF spectrum showing elemental peaks in the brick sample

Table 1
The elemental composition of one random reference brick (X-ray fluorescence (XRF) analysis)

Compound	m/m%	StdErr%	El	m/m%	StdErr%
SiO_2	37.57	0.30	Si	17.57	0.14
Al_2O_3	7.25	0.14	Al	3.84	0.07
Fe_2O_3	3.36	0.20	Fe	2.35	0.14
P_2O_5	2.26	0.09	Px	0.988	0.041
K_2O	1.23	0.02	K	1.02	0.02
TiO_2	0.889	0.087	Ti	0.533	0.052
CaO	0.555	0.028	Ca	0.397	0.020
SO_3	0.139	0.010	Sx	0.0555	0.0042
ZrO_2	0.0273	0.0017	Zr	0.0202	0.0013
Co_3O_4	0.0237	0.0080	Co	0.0174	0.0059
V_2O_5	0.0175	0.0085	V	0.0098	0.0048
Cr_2O_3	0.0133	0.0012	Cr	0.0091	0.0008
ThO_2	0.0127	0.0035	Th	0.0112	0.0030
ZnO	0.0093	0.0004	Zn	0.0075	0.0003
CuO	0.0074	0.0008	Cu	0.0059	0.0007
NiO	0.0066	0.0004	Ni	0.0052	0.0003

Table 2
Elemental analysis of the injection cream

Element	Content [Mg/kg]
Al	11.04
B	10.53
Ba	0.9935
Ca	368.0
Cu	6.396
Fe	10.72
K	34.43
Mg	52.77
Mn	1.586
Na	74.11
Si	772.6
Zn	7.674

III) of six bricks. Three bricks were left as a reference (REF). Afterwards, the bricks were conditioned to specific moisture levels, as described below. The bricks from group I were stored in stabilized laboratory conditions at a temperature of about 21°C and an air humidity of about 49%, so that they absorbed moisture from the air. The bricks from groups II and III were placed in a building caster, which was gradually filled with tap water until it reached a level of 11/12 of the height of the bricks (Fig. 3). During this soaking period, the bricks from all groups were regularly weighed to track moisture uptake and ensure that the designed humidity gradient was consistently achieved. Mass readings were recorded using a precision laboratory balance with an accuracy of 0.01 g, ensuring a high level of measurement reliability.

Fig. 3. A building caster containing bricks flooded with water to achieve adequate moisture content

The conditioning process was continued until each group reached its target average moisture content U_m, which for group I was equal to 3%, for group II to 7%, and for group III to 11%. These values correspond to the following moisture content levels of brick walls, which can be found in the specialist literature, e.g., [20,21]: permissible moisture content (group I), medium moisture content (group II), and high moisture content (group III). The mass moisture values were calculated using (1)

$$U_m = \frac{m_w - m_s}{m_s} \cdot 100\%, \tag{1}$$

where U_m – mass moisture, m_w – wet sample weight, m_s – dry sample weight.

The inclusion of different degrees of wall moisture in the studies was based on the authors' previous experience, which indicates that the moisture of a wall varies both across its cross-section and at different heights. The analysis of the lowest moisture level was conducted to determine the borderline cases (which are important with regard to preventive conservation) and to consider local areas of limited moisture content (typically in historic buildings).

In the subsequent steps, two boreholes were made in the bases of the bricks from groups I–III using a 12 mm diameter drill. They were performed at a distance of about 12 cm from each other (according to the recommendations of the manufacturer of the injection cream) and to a depth that is 50 mm less than the thickness of the brick. The holes were cleaned with compressed air.

In nine bricks, which were characterized by three different degrees of moisture content (three bricks each from groups I, II, and III), a pressure injection was performed. It involved the implementation of the injection cream (in a continuous manner) using a dispenser under a pressure of 2 atm. In the remaining nine bricks (also three bricks each from groups I, II, and III), a gravity injection was performed. It consisted of the introduction of the injection agent, as a result of the action of gravity, into the borehole (Fig. 4). After conducting the injections, the holes were sealed with cement mortar, and the bricks were placed in plastic zip-lock bags to maintain their moisture content while the cream spread. In the reference bricks, no injections were performed.

Fig. 4. Performing gravity injection in the brick with injection cream

Figure 5 shows the research plan, which divides the test samples into groups based on the moisture content and injection method. Three moisture ranges were distinguished: permissible moisture (group I), moderate moisture (group II), and high moisture (group III); and two types of injection: pressure (P) and gravity (G). Three samples were included in each of the six groups (IP, IG, IIP, IIG, IIIP, IIIG).

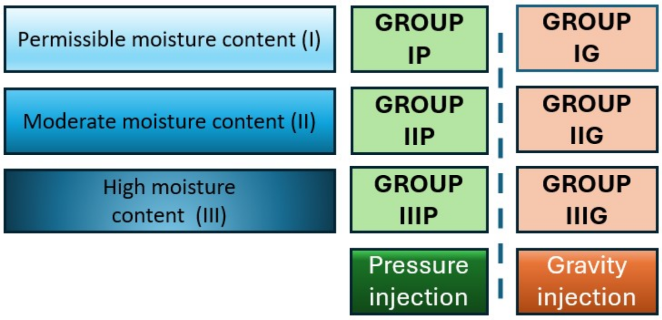
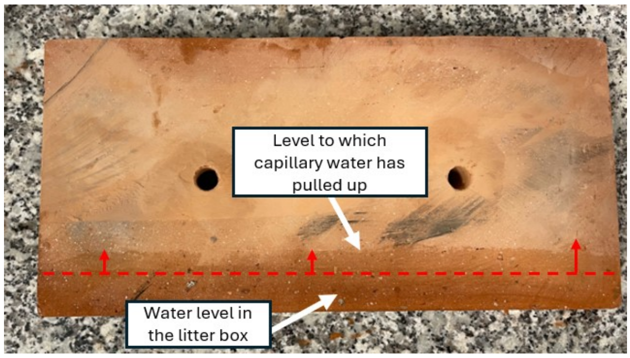


Fig. 5. Division of the test samples according to the moisture content and injection method used

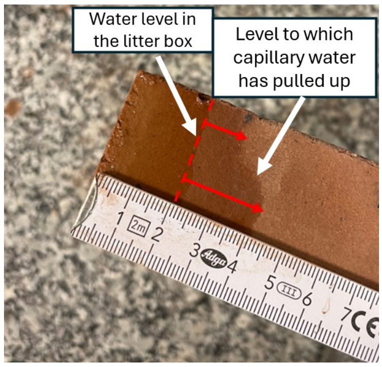
After three weeks, bricks from groups I, II, III, and REF were dried to a constant mass in a laboratory dryer at a temperature of 30°C to ensure uniform starting conditions for further research. The choice of drying temperature was preceded by tests aimed at determining the effect of different drying temperatures on the properties of the injection cream. The tests consisted of preparing three samples of injection cream weighing approximately 10 grams, which were placed in Petri dishes and then in a laboratory dryer, where they were exposed to temperatures of 30°C, 60°C, and 90°C. Afterwards, changes in the material under the influence of thermal factors were analyzed. It was observed that at 90°C the injection cream experienced significant evaporation, indicating intense moisture loss and a change in its structure. At lower temperatures, i.e., 30°C and 60°C, the material behaved similarly – it showed no weight loss, but its consistency changed, becoming more fluid. Finally, it was decided that a temperature of 30°C would be used in the research to maintain conditions more similar to the natural ones that occur in the environment surrounding brick walls.

The bricks, dried to a constant weight, were then weighed and placed in a plastic box on plastic grids. Tap water was gradually poured into the box until it reached a level of about 11/12 of the height of the bricks. For 35 days, the weight of the bricks was checked approximately every 24 hours until it stabilized. Figure 6 shows a brick from group IG in the initial phase of dampness, which was removed from the box when the water level reached approximately 1/6 of the brick height. The figure clearly shows the boundary between the water level in the box and the moisture that was drawn up by the capillary action.

Throughout the test period, the bricks were stored at a controlled temperature of 210°C (fluctuation range: 20–220°C). The relative air humidity was kept at 49%. Next, based on the collected mass measurements, the mass moisture content of all the tested bricks was determined using formula (1).



(a)



(b)

Fig. 6. View of the brick from group IG in the initial phase of water saturation: (a) view from the base side; (b) view from the head side

In the next step, an attempt was made to assess the spread of the injection agent within the brick structure. For this purpose, the injected bricks were cut in half. Since it was not possible to observe a clear trace of the injection agent on the cut surface of the brick, a water drop test was performed. For this purpose, single drops of water were placed on the surface of three selected bricks – one from group IIIP, one from group IIIG, and one reference brick. A pipette was used for this, and a uniform distance of approximately 2 cm from the base of the sample was maintained. Bricks from groups IIIP and IIIG were selected because the greatest differences in the effectiveness of the injectable agent were observed within group III. The evaluation consisted of a 2-hour observation to determine whether each water drop remained on the surface or was absorbed by the material. Droplet absorption was interpreted as an indication of insufficient distribution of the injection agent in the area to be tested. Additionally, to assess the spread of the injection agent within the brick structure, microstructural analysis using scanning electron microscopy (SEM) was performed on small samples cut from the bricks used in the water drop test. The sampling locations are shown in Fig. 7.

To complement the moisture analyses, samples taken from the bricks from groups IIIP, IIIG, and REF also underwent X-ray fluorescence (XRF) testing to identify and quantify key elements in the brick material, in turn offering insight into changes caused by the injection process. The sampling locations were identical to the sampling locations for the SEM studies (Fig. 7).

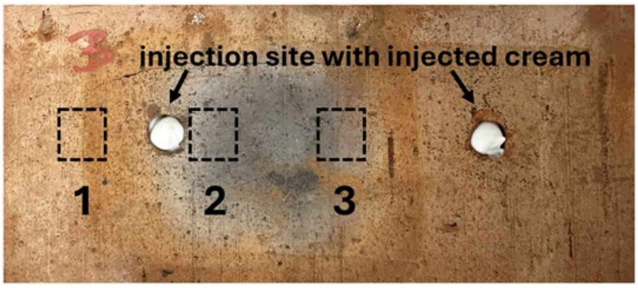


Fig. 7. Brick sampling locations located at different distances from the injection hole: 1 – approximately 3 cm from the edge of the injection hole, 2 – near the injection hole, 3 – approximately 6 cm from the edge of the injection hole

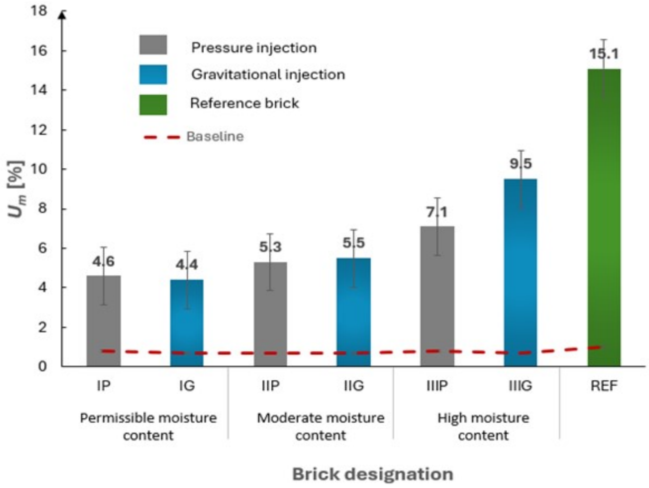


Fig. 8. Average mass moisture values (U_m) for re-moistened bricks from groups IP, IG, IIP, IIG, IIIP, III, and the reference group (REF)

3. RESEARCH RESULTS AND THEIR ANALYSIS

The following chapter presents and interprets the results of all the laboratory tests. The results obtained confirm that there is a relationship between the brick moisture content at the time of injection and the used injection method (which influences the effectiveness of moisture protection).

3.1. The results of the moisture tests

The results of the preliminary moisture tests, conducted with the use of the gravimetric method, are presented below in the form of tables and figures.

The maximum values of the mass moisture content (U_m) of re-moistened bricks from groups IP, IG, IIP, IIG, IIIP, IIIG, and REF are shown in Fig. 8.

When analyzing the graph shown in Fig. 8, it can be seen that the average mass moisture content U_m for the bricks from the IG group (with an initial permissible moisture content, subjected to gravity injection) was characterized by having the lowest moisture content ($U_m = 4.4\%$). In contrast, the average mass moisture content (U_m) for the bricks from group IIIG (with a high moisture content at the time of injection, subjected to gravity injection) had the highest value ($U_m = 9.5\%$). The mass moisture content values of all the brick groups in which injections were performed are two to three times lower than the average moisture content of the reference brick group, which amounted to 15.1%. This demonstrates a clear reduction in water absorption capacity as a result of the applied protection.

Table 3 and the graph in Fig. 9 show the values of mass moisture U_m of the tested bricks at different stages of their re-moistening. The test was conducted for 35 days, during which systematic humidity measurements were taken at specified intervals. The presented results (measurements 1–7) refer to the average mass moisture content of the soaked groups of bricks on the 5th, 10th, 15th, 20th, 25th, 30th, and 35th day of testing, respectively.

Based on the data presented in Table 3 and Fig. 9 on the increase in mass moisture content U_m in percentage points, it can be concluded that the largest moisture content rises of 8.8 pp and 6.3 pp were observed in brick groups IIIG and IIIP, while the smallest moisture content increase was observed in brick group IG, and amounted to 3.7 pp. Converting the recorded values into percentages, it should be noted that in the case of brick group IIIG, its mass moisture content increased by 1257%

Table 3

Average values of mass moisture content U_m, and changes in this moisture content for individual groups of bricks at various stages of the 35-day test

Degree of moisture content	Brick group number	Type of injection	U_m (average of three samples) of bricks [%]								Increase in the moisture content in percentage points
			Measurement								
			Baseline	1	2	3	4	5	6	7	
Permissible moisture content (I)	IP	pressure	0.8	1.6	2.3	2.9	3.5	4.1	4.4	4.6	3.8
	IG	gravitational	0.7	1.4	3.0	3.5	3.7	3.9	4.3	4.4	3.7
Moderate moisture content (II)	IIP	pressure	0.7	2.7	3.7	4.5	4.8	5.0	5.2	5.3	4.6
	IIG	gravitational	0.7	3.1	4.6	5.0	5.3	5.4	5.5	5.5	4.8
High moisture content (III)	IIIP	pressure	0.8	3.3	4.9	5.5	6.2	6.7	6.9	7.1	6.3
	IIIG	gravitational	0.7	7.1	8.7	9.1	9.2	9.3	9.4	9.5	8.8
Reference brick	REF	none	1	13.6	14.1	14.4	14.6	14.8	15.0	15.1	14.1

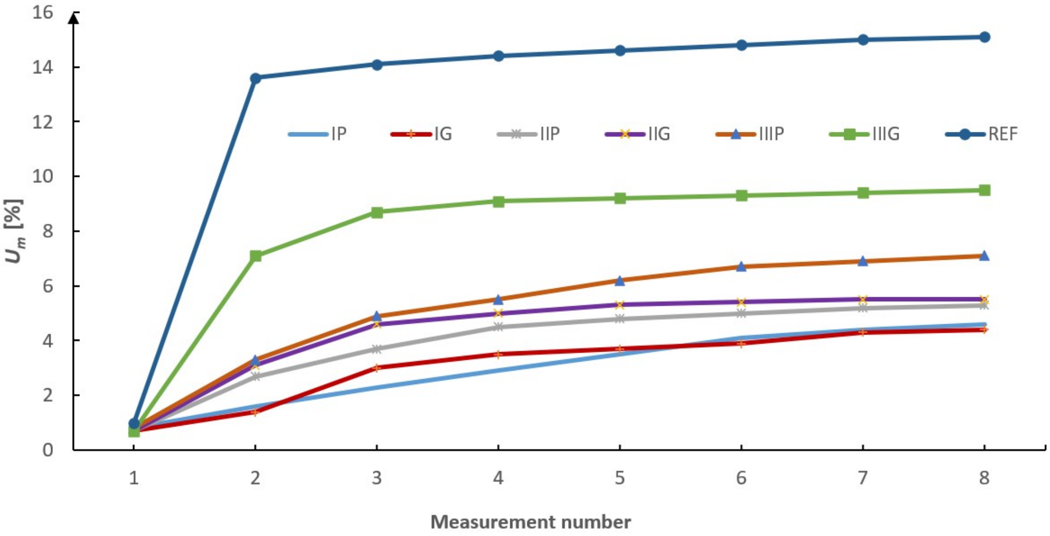
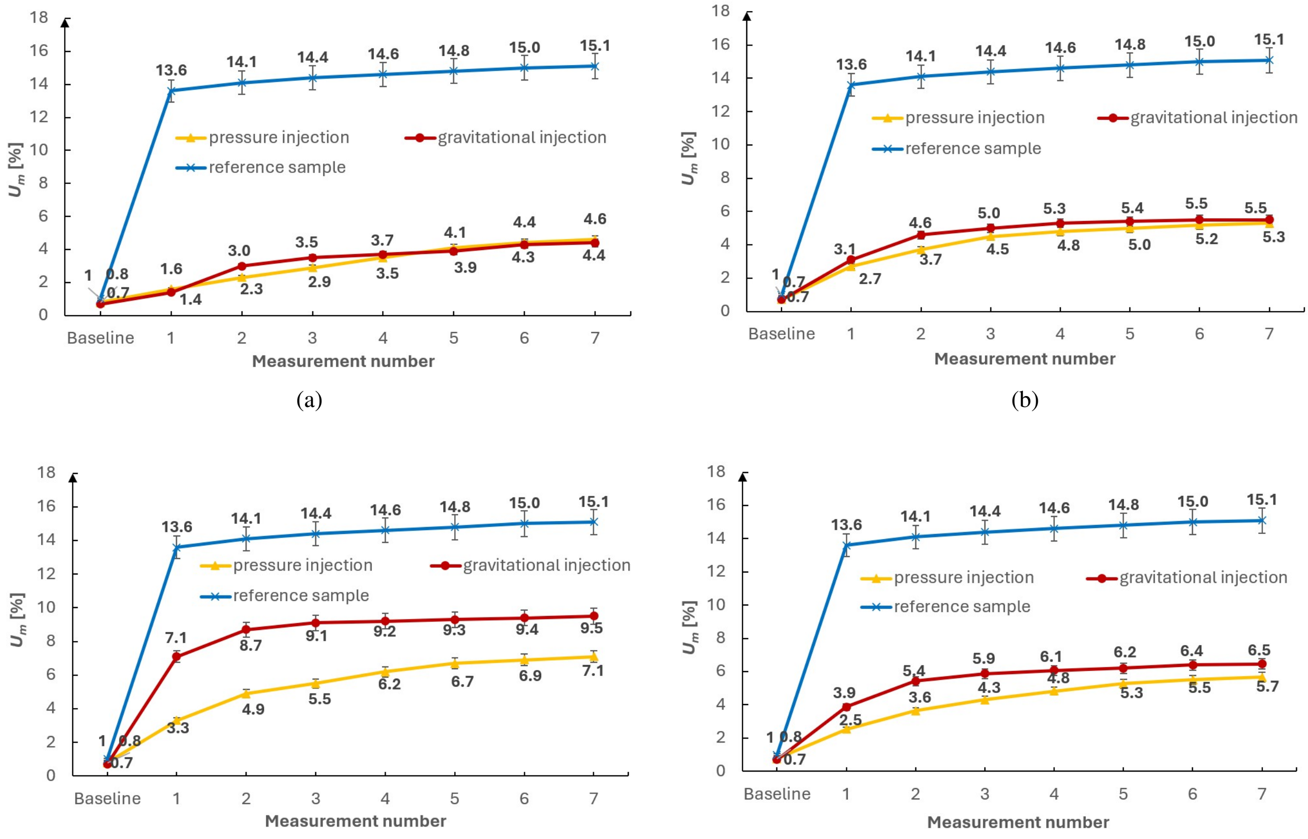


Fig. 9. Changes in the mass moisture content of brick groups IP, IG, IIP, IIG, IIIP, IIIG, and REF during the 35-day test

(initial $U_m = 0.7\%$ vs. final $U_m = 9.5\%$), and in the case of brick group IG by 529% (initial $U_m = 0.7\%$, final $U_m = 4.4\%$). In general, the fastest increase in moisture content was observed in the initial phase of brick soaking.

The effectiveness of damp-proofing the bricks was also compared according to the injection method used. Pairwise comparisons (pressure vs. gravity injection) were made for groups IP, IG, IIP, IIG, IIIP, IIIG, and REF, as shown in Figs. 10a–10c. In



(a) (b) (c) (d)

Fig. 10. Comparison of the effectiveness of the protection against moisture in the bricks subjected to pressure and gravity injection, considering different degrees of brick moisture content at the time of injection: (a) permissible moisture content (IP vs. IG); (b) moderate moisture content (IIP vs. IIG); (c) high moisture content (IIIP vs. IIIG); (d) average values

addition, Fig. 10d shows the average moisture values obtained for the pressure and gravity methods, excluding the degree of brick moisture at the time of injection.

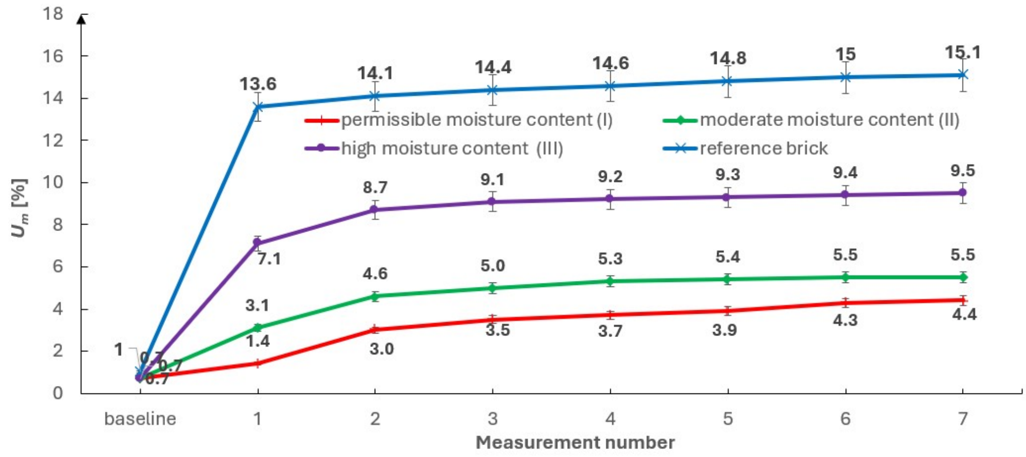
The conducted tests showed slight differences in the effectiveness of gravity and pressure injections in conditions of the permissible moisture (Fig. 10a) and moderate moisture content (Fig. 10b) at the time of injection. When analyzing subsequent measurements, in most cases, gravity injections give higher U_m values than pressure injections, but the differences are not significant. However, under high humidity conditions (Fig. 10c), the differences between the methods are clearly noticeable, reaching a maximum of approximately 3.8 pp in measurements no. 1 (7.1% vs. 3.3%) and no. 2 (8.7% vs. 4.9%). In subsequent measurements, this difference gradually decreased, stabilizing at around 2.4 pp in measurements no. 6 and 7. In relation to the average difference between the mass moisture values obtained for the pressure and gravity injection methods, its values were: 0.1% in group I, 0.4% in group II, and 2.7% in group III.

Considering the maximum achieved humidity values of the re-moistened bricks (measurement No. 7), the results suggest a potential advantage of the gravity injection method under conditions of acceptable material moisture content. Conversely, under medium to high moisture levels, the pressure injection method demonstrated greater effectiveness.

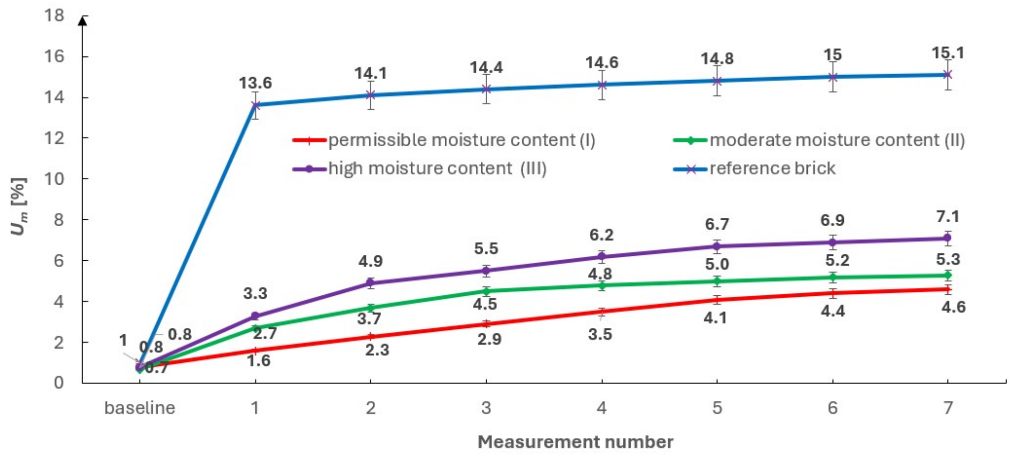
However, given the relatively minor differences in performance between the two techniques, based on the results obtained to date, it is not possible to definitively determine which method is generally more optimal. For this reason, further research is necessary, including a larger number of samples and a broader range of variables, including comparisons of different injection creams.

The effectiveness of the moisture protection was also analyzed concerning the degree of moisture content of the bricks at the time of their injection. The comparison was made in two groups, separately for pressure and gravity injections. This is shown in Figs. 11a and 11b.

Regardless of the type of injection, the lowest moisture content values were observed for bricks with a permissible moisture



(a)



(b)

Fig. 11. Comparison of the effectiveness of the protection against moisture with regard to the degree of the moisture content in the bricks at the time of the injection: (a) moisture values of the bricks in which gravity injection was performed; (b) moisture values of the bricks in which pressure injection was performed

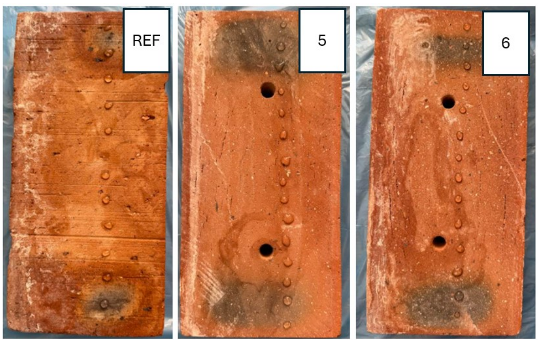
content (group IP and IG); the increase in the moisture content of these bricks was remarkably uniform. On the other hand, the highest moisture values were observed for bricks with high moisture (group IIIP and IIIG), which absorbed noticeably more water than the other groups under identical exposure conditions. In their case, too, the initial moisture (U_m) growth was the most intense, with the growth recorded between the initial and first measurements amounting to 3.3% for the pressure method, and 7.1% for the gravity method.

The findings of the study appear to contradict the manufacturer's guidelines for the injection cream, which suggest that creams exhibit optimal spreading properties in environments with elevated humidity levels, and therefore theoretically form the tightest hydrophobic barrier in walls with a high degree of moisture. Consequently, further research encompassing a broader spectrum of material and environmental conditions is required to enable a more thorough evaluation of the tested effectiveness of injection methods and their practical applicability in construction engineering. This range should consider the impact of soluble salts and freeze-thaw cycles (frost resistance), among other things.

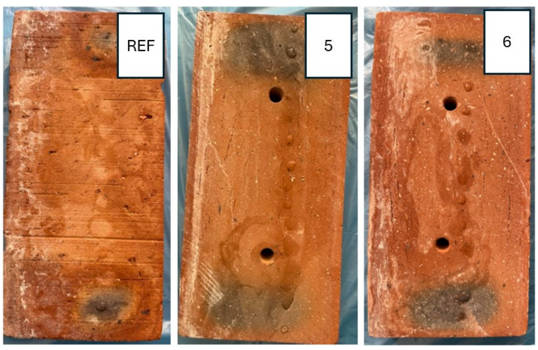
3.2. Test results concerning the spread of the injection cream

The results of tests assessing the distribution of the injectable cream within the brick structure, conducted with the use of the water drop test and SEM, are presented below.

Figure 12 shows the reference brick (REF) and two injected bricks (brick no. 5 from group IIIP and brick no. 6 from group IIIG) at the initial (Fig. 12a) and final (Fig. 12b) stages of the water drop test.



(a)



(b)

Fig. 12. Water drop tests: (a) bricks at the beginning of the test; (b) bricks after 2 hours of testing

As shown in Fig. 12b, two hours after applying water droplets, clear differences in the behavior of brick surfaces are visible. On the reference brick (REF), the droplets were completely absorbed, indicating the absence of a hydrophobic barrier. In the case of the brick from group IIIP, some of the drops remain on the surface, especially near the injection holes, while in more distant areas, most of them were absorbed. On the brick from group IIIG, individual drops are still visible, mainly in the areas closest to the injection holes, but their number is smaller than on the brick from group IIIP. This indicates a less effective action of the injection cream in this sample, which is consistent with the results of the mass moisture analysis (average $U_m = 7.1\%$ in group IIIP vs. average $U_m = 9.5\%$ in group IIIG).

In turn, the results of the SEM are presented in Figs. 13 and 14. The image in Fig. 13 reveals the complex mineral structure of the brick sample from the REF group, with numerous sharp edges and irregular grains, which is typical of materials fired at high temperatures. The visible porosity, manifested in the form of voids and microholes, plays a key role in moisture transport and interaction with chemicals present in the building environment. The variation in pore size and shape confirms the heterogeneous nature of the mineral matrix, which may affect water absorption and susceptibility to degradation. This microstructure determines both the mechanical properties of the brick and its ability to interact with the injection preparations used in hydrophobization treatments.

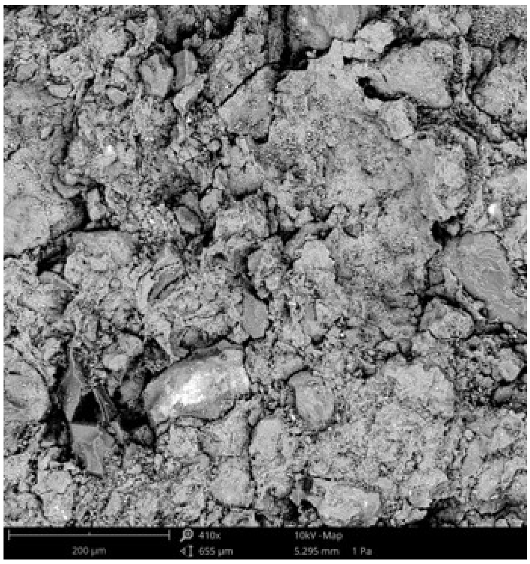


Fig. 13. SEM image of the structure of the brick from the reference group

Figure 14 compares the microstructures of three samples taken from brick no. 5, group IIIP (Figs. 14a–14c), and three samples taken from brick no. 6, group IIIG (Figs. 14d–14f). In each case, the samples were taken at different distances from the injection hole (as shown in Fig. 7), which allows for the migration of the preparation within the brick to be tracked.

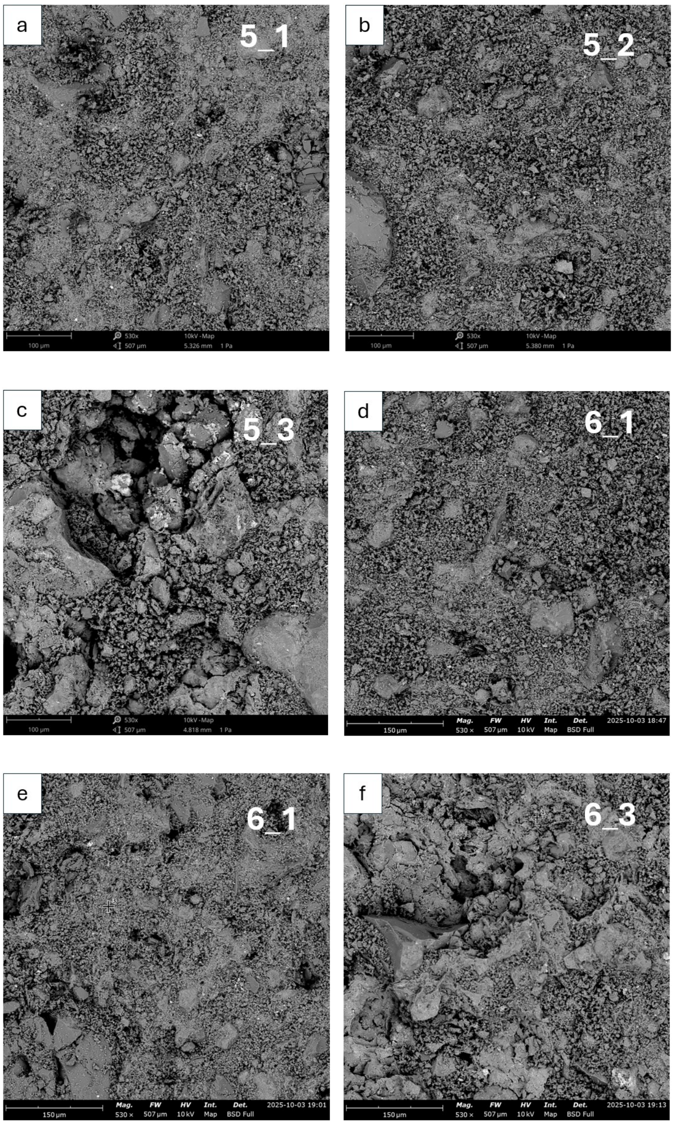


Fig. 14. SEM images of the structure of the samples taken from brick no. 5 from the IIIP group (a–c), and brick no. 6 from the IIIG group (d–f): 1 – near the injection hole, 2 – in the intermediate zone, 3 – in the distant area

In the case of the brick from group IIIP, when comparing it with the REF brick, a clear sealing of the micropores near the injection hole and a local reduction in porosity are visible in samples no. 1 and no. 2 (Figs. 14a, 14b), indicating the effective penetration of the agent into the material structure. Sample no. 3, taken further from the hole (Fig. 14c), exhibits a structure with distinct grain edges and pores, with slightly fewer pores than in the reference brick, suggesting limited migration of the agent to more distant areas.

In the case of the Group IIIG brick, a more pronounced presence of open pores can be observed than in the Group IIIP brick, particularly in samples no. 2 and no. 3. This may indicate a lower penetration of the injection cream and a limited effect in these locations.

Both the microscopic observations (SEM) and the water drop test confirm the results of the mass moisture analysis (U_m) obtained using the gravimetric method, indicating clear differences in the effectiveness of the injection agent between the analyzed samples. The better result obtained for brick no. 5 from group IIIP can be explained using pressure injection, which allows for deeper and more even penetration of the preparation into the structure of the material. In contrast, the gravity injection in brick no. 6 from group IIIG, at higher humidity, may have encountered greater capillary resistance, in turn limiting the penetration of the agent in a highly humid environment.

It is worth noting that the spread of the injection cream took place under laboratory conditions, which provided a controlled environment for observing migration. It should also be noted, however, that in this study, the time allowed for the injection cream to spread within the brick was only three weeks. Therefore, it would be necessary to verify whether the results would be the same if a longer time were allowed for the cream to spread within the brick. It would also be necessary to verify whether there are differences in the SEM images for the bricks injected under the other humidity conditions (permissible and moderate moisture content) considered in the study.

3.3. Results of the chemical composition test

To complement the moisture analyses, samples taken from the bricks from groups IIIP, IIIG, and REF underwent X-ray fluorescence (XRF) testing to identify and quantify key elements in the brick material, which offered insight into changes caused by the injection process. The sampling locations were identical to the sampling locations for the SEM studies (Fig. 7).

Figure 15 shows, based on XRF analysis, a graph illustrating the percentage share of individual elements in the samples. The elemental distribution in the injected bricks shows high consistency, with similar values regardless of the sample location. It is worth noting that the silicon (Si) content in the reference brick is about 15% higher than in the samples injected with injection cream.

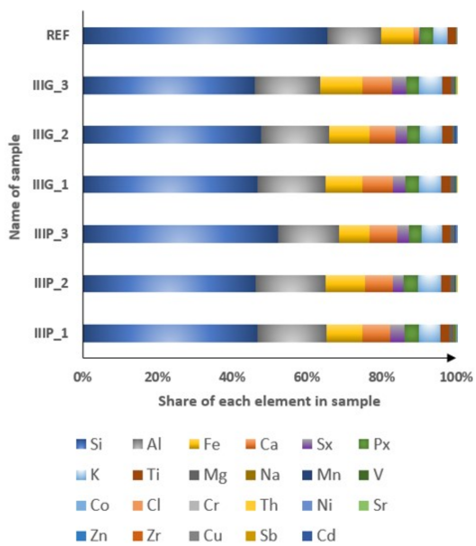


Fig. 15. Percentage share of individual elements in the samples from groups IIIP, IIIG, and REF, determined by X-ray fluorescence analysis

Table 4 shows the mass percentage of the elements present in the injected bricks, the reference brick, and the injection cream. The "REF" column refers to the control brick, which was not injected, and which serves as a reference point for comparing changes in the chemical composition after the application of the injection cream. The "Injection cream" column presents the chemical composition of the preparation itself, i.e., the agent injected into the brick structure. The columns marked IIIP_1–IIIG_3 contain data on the bricks into which the injection cream was introduced and then subjected to chemical composition analysis.

Table 4

Mass percentage of the elements present in the selected samples

	IIIP_1	IIIP_2	IIIP_3	IIIG_1	IIIG_2	IIIG_3	REF	Cream
Si	14.020	13.510	16.080	14.010	15.150	14.710	17.570	
Al	5.5700	5.4200	4.9800	5.4200	5.8100	5.6100	3.8400	0.0011
Fe	2.8600	3.1300	2.5600	2.9600	3.3900	3.6600	2.3500	0.0011
Ca	2.2200	2.1300	2.2700	2.4100	2.1900	2.5300	0.3970	0.0368
Sx	1.2000	0.8370	0.9790	1.0100	1.0200	1.2400	0.0555	
Px	1.1400	1.1200	0.9780	1.1100	1.0700	1.0700	0.9880	
K	1.7400	1.8000	1.7500	1.7800	1.9100	1.9700	1.0200	0.0034
Ti	0.6890	0.7340	0.6750	0.7080	0.8110	0.8120	0.5330	
Mg	0.3410	0.3570	0.3520	0.3670	0.2480	0.2730	–	0.0053
Na	0.0890	–	–	–	–	–	–	0.0074
Mn	0.0247	0.0253	0.0218	0.0259	0.0287	0.0313	–	0.0002
V	0.0163	0.0137	0.0127	0.0155	0.0151	0.0203	0.0098	–
Co	–	0.0160	0.0151	–	–	–	0.0174	–
Cl	0.0245	0.0127	0.0194	0.0202	0.0108	0.0218	–	–
Cr	0.0131	0.0103	0.0096	0.0148	0.0120	0.0136	0.0091	–
Th	0.0121	–	0.0104	0.0098	0.0114	0.0115	0.0112	–
Ni	0.0078	0.0061	0.0054	0.0069	0.0062	0.0101	0.0052	–
Sr	0.0069	0.0062	–	0.0064	0.0052	0.0072	–	–
Zn	0.0059	0.0091	–	0.0122	0.0081	0.0068	0.0075	0.0008
Zr	–	–	0.0099	–	–	–	0.0202	–
Cu	0.0058	0.0065	0.0051	0.0109	0.0067	0.0105	0.0059	0.0006
Sb	0.0049	0.0043	–	0.0046	0.0058	0.0044	–	0.0011
Cd	0.0052	–	0.0050	–	0.0062	–	–	0.0011
B	–	–	–	–	–	–	–	0.0011
Si	–	–	–	–	–	–	–	0.0773

Based on the distribution of elements, it can be seen that elements such as magnesium (Mg) and manganese (Mn), which are not present in the reference brick, appeared in the injected bricks. Importantly, their distribution within individual samples (labeled 1–3) is relatively even and does not depend on the distance from the injection hole into which the cream was injected. In addition, a decrease in the silicon (Si) content was observed in the samples after the application of the agent. This phenomenon may be due to several factors: dilution of the sample by the injection cream, which does not contain Si in this form; inhibition of silicate migration from the environment; and the blockage of pores, which limits the exchange of silicon compounds in the brick structure.

4. DISCUSSION

The results of the research on the use of silane cream to protect ceramic bricks from moisture indicate that the best protection against capillary water was achieved for the bricks with an acceptable moisture content at the time of injection (group I). In these bricks, the cream freely permeated the capillaries, creating a continuous barrier. In the samples with a medium moisture content (group II), the effectiveness of the moisture protection was only slightly worse. However, in the cases of a high brick moisture content (group III), the injection effectiveness was significantly lower, which may be related to the cream dilution and impaired bonding with the material. This is due to the physicochemical properties of silane, which require the presence of water to cure; however, both excess and deficiency of water can disrupt this process.

The research results also showed, depending on the moisture content of the material at the time of the injection, that different injection methods (gravity vs. pressure) proved more effective. In the case of bricks with an acceptable moisture content (group I), the gravity method proved to be the most effective, resulting in the lowest U_m moisture value among all the samples. The gradual penetration of the cream promoted even distribution of the preparation, in turn reducing the risk of leaching and structural damage. On the other hand, at medium and high moisture levels (groups II and III), better results were achieved with pressure injection, which allowed for deeper saturation of the material and the more effective creation of a hydrophobic barrier.

The presented results refer to a specific injection preparation used under controlled laboratory conditions. Paper [22] emphasizes the importance of the moisture content as a key factor in the performance of injection methods, but focuses mainly on general technological challenges, without analyzing the impact of specific moisture thresholds under laboratory conditions, as was attempted in this study. Moreover, while Franzoni emphasizes in his research [22] the variability of treatment outcomes across masonry types, the present research contributes a direct experimental comparison of gravity and pressure injection techniques under fixed laboratory conditions, suggesting that gravity-based application may result in more favorable performance in moderately dry ceramic brickwork.

The results of this study are consistent with those presented in paper [23] by Francke, Piekarczuk, and Dobrev, which confirmed the overall effectiveness of gravity and low-pressure injection methods. However, while those large-scale wall tests incorporated various product types and emphasized factors such as formulation density and injection layout, the present study offers a novel contribution by isolating the influence of specific moisture thresholds under controlled laboratory conditions and identifying medium moisture as a potentially limiting factor due to pore saturation effects.

It should be emphasized that the tests were conducted using one injection cream and two application methods under controlled laboratory conditions. This limits the possibility of fully generalizing the results to other preparations and actual construction conditions. Due to the diversity of available products, further comparative studies are recommended to develop practical recommendations for designers and contractors.

Future experiments by the authors will include an assessment of the effectiveness of various injection creams in different materials and environmental conditions, including exposure to salts and in relation to historical and modern bricks. An analysis of the impact of freeze-thaw cycles, variable injection pressure, and alternative application techniques, such as vacuum and hybrid methods, is also planned.

5. CONCLUSIONS

The article presents the results of preliminary laboratory tests, which aimed to compare the effectiveness of the protection against moisture made using the injection method and a silane-based injection cream. The injection was performed in solid ceramic bricks using pressure and gravity methods. The effect of different degrees of moisture content of bricks (at the time of their injection) on the effectiveness of the obtained protection was evaluated.

Based on the conducted research results, it can be concluded that:

- The lowest average mass moisture content ($U_m = 4.4\%$) was reached by re-moistened bricks with an initial permissible moisture content at the time of injection that were subjected to gravitational injection, while the highest average mass moisture content ($U_m = 9.5\%$) was reached by re-moistened bricks with a high initial moisture content that were also subjected to gravitational injection.
- Gravity injection was most effective under permissible moisture conditions at the time of injection, leading to the lowest mass moisture U_m values after re-moistening of the bricks.
- Pressure injection showed greater effectiveness in the case of a medium and high moisture content at the time of injection, which allowed for more effective saturation of the brick with the injection cream.
- With the increase of moisture content at the time of injection, the spreading of the cream inside the brick deteriorated, which was quantitatively confirmed in laboratory conditions.
- Microscopic observations (SEM) showed that the content of the injection cream decreased with the distance from the application site, which confirms its limited ability to migrate within the brick structure.
- Some characteristic elements of the injection cream, e.g., Mg and Na, appear locally in the bricks where the injection was made, confirming its migration after application. Their distribution within the brick samples was relatively even and did not depend on the distance from the place of injection.

Based on the obtained results, approximate humidity ranges at which the tested silane-based injection cream shows the greatest effectiveness can be initially proposed. Under conditions of acceptable humidity (approx. 3%), the gravity method was the most effective, while at medium (approx. 7%) and high (approx. 11%) humidity levels, better results were obtained with the pressure method. These ranges are preliminary, but they can serve as a starting point for practical recommendations on the selection of injection techniques in relation to the moisture content of the substrate.

The studies provided important insights, but due to the limited number of samples and the early stage of the work, the results should be considered exploratory and require further validation.

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