

INTERNATIONAL JOURNAL CONSERVATION SCIENCE





DOI: 10. 36868/IJCS.2025.si.11

THE IMPROVEMENT OF THE DURABILITY AND FROST RESISTANCE OF TRADITIONAL AND HISTORICAL LIME MORTARS BY MEANS OF SURFACE IMPREGNATION WITH LIQUID POTASSIUM SILICATE AND SILICIC ACID ESTER

Dominik LOGOŃ^{1,*}, Anna KRUKOWIECKA-BRZĘCZEK²

¹ Wroclaw University of Science and Technology, Faculty of Civil Engineering, Wybrzeże Wyspiańskiego 27, 50-370 Wroclaw, Poland

²Cracow University of Technology, Faculty of Architecture, Warszawska 24, 31-155 Kraków, Poland

Abstract

The research focused on improving the durability and frost resistance of lime mortars. The tests were carried out on traditional lime mortar, but the conclusions can also be applied to historical composites based on lime binders. Surface impregnation with liquid potassium silicate and silicic acid ester was used in the tests. The test results showed a slight improvement in the bending strength of the test specimens. A significant improvement in the physical and mechanical properties (compressive strength, water absorption resistance and frost resistance) was noted. The obtained results indicate that it is possible to increase the durability of the lime mortars without changing their color. In the summary it was noted that surface impregnation of lime mortars with silicic acid ester proved to be the most effective way to improve the physical and mechanical properties of built-in lime mortars, but the higher cost of impregnation, compared to liquid potassium silicate, may be a reason for the limitation of this solution. It was pointed out that surface impregnation is often the only way to increase the durability of brittle composites with low strength.

Keywords: Historical architecture; Historical lime mortars; Liquid potassium silicate; Silicic acid ester; Lime mortar; impregnation; Frost resistance; Compressive strength; durability

Introduction

The modification of traditional and historical lime mortars to improve their durability is the subject of many scientific papers. This applies to both currently used mortars and historical mortars [1, 2]. The range of additives and admixtures introduced to modify the properties of those composites is very wide [3-5].

The aim of revitalization work concerning historical lime mortars is to select methods and materials that do not significantly interfere with the architectural properties relating to the color and structure of the reinforced materials [6-10]. One of the most important issues is the sustainable interference in the modification of the physical and mechanical properties of the reinforced materials [11-27]. Many polymer-based agents are not considered to be appropriate for restoration work on historical structures because they have too much impact on the properties of mortar.

^{*} Corresponding author: dominik.logon@pwr.edu.pl

The use of water glass (sodium silicate) to improve the properties of cement and lime mortars has been the subject of many studies [19-21]. It is important to distinguish between liquid potassium silicate and sodium silicate for impregnation purposes. In repair work for the surface reinforcement of lime mortars, the application of liquid potassium silicate is more justified than the application of liquid sodium silicate [17]. The results of the tests should not be treated as also generally valid for other materials due to the possibility of uncontrolled reactions. The application of impregnating agents on new surfaces requires in each case the confirmation of their effectiveness in increasing the durability of those composites.

Tests of silicic acid esters with respect to various historical materials are a relatively new issue and, as in the case of liquid potassium silicate (as regards application on the surface of other materials), require appropriate verification.

An important question regarding the reinforcement of brittle composites with relatively low strength is the method of application of impregnating agents to increase their durability [22, 23]. The use of the pressure injection method is not recommended due to the possibility of destroying the structure of those composites [24, 25]. The gravitational soaking method cannot always be used. It generates a large volume of impregnating agents, which results in increased repair costs. The surface impregnation method used in this work limits the reinforcing effect to surface layers but, as the obtained results indicate, can be effectively used to increase the durability of brittle composites with lime binder characterized by relatively low strength.

Experimental part

Materials

The reference mortar M0 corresponds to a traditional lime mortar based on hydrated lime CL 90-S with a compressive strength of approx. 1MPa. The weight proportion of mortar components lime to sand is 1:4.5

Water – tap water added in an amount that made it possible to obtain a constant mortar consistency (PN-B-04500 consistency 6 - Novikow cone).

The specimens were molded into 40mm×40mm×160mm beams and kept in a humidity chamber for 28 days. The specimens were left for a further 28 days under laboratory conditions at 20°C. Six specimens of the tested mortars were used for each determination of the result and the results were presented as an arithmetic mean.

The reference mortar M0 was impregnated twice with the use of a brush-liquid potassium silicate and silicic acid ester were applied separately (Table 1).

Table 1. Sample lime mortars used in the research

Lime mortar specimen	
M0	reference mortar
MS	ref. mortar impregnated with liquid potassium silicate (S)
MH	ref. mortar impregnated with silicic acid ester (H)

Methods

Compressive strength and three-point bending test

The three-point bending test ftb performed according to the guidelines for standard cement mortar (Fig. 1b). The compressive strength test f_c was performed on the beam halves obtained from the f_{tb} test (Fig. 1a).



Fig. 1. Mortar specimens: a) compressive test, b) bending test

Water absorption test

The water absorption test was carried out by placing the lime mortar beams $(40 \text{mm} \times 40 \text{mm} \times 160 \text{mm})$ in a vertical position in a cuvette filled with water to a height of 10 mm. The specimens were left for 1 hour. The table presents the water absorbed by the specimens (percent by mass).

Frost resistance

The frost resistance test was conducted by carrying out the cycles of freezing and thawing of the lime mortar beam halves obtained after the 3-point bending test. Before the frost resistance test, the fracture surfaces of the specimens were impregnated.

One cycle consisted of flooding the specimens with water and storing them in the water at 20°C for 4 hours, after which the wet specimens were frozen in a freezer at -20°C for 4 hours. One hour after the start of the next cycle (flooding with water), the specimens were removed, visually assessed, weighed and documented photographically. Next, they were placed in the water and the freeze-thaw cycle was continued.

The specimens that were damaged, showed cracks, or had a weight loss of more than 5% were considered to present a negative frost resistance result.

Results

Table 2 shows the results of the tested physical and mechanical properties. The results indicate the amount of water in [g] that was absorbed by the specimens and the amount of water in [%] calculated with reference to the dry mass of the specimen. In addition, the table shows the number of freeze-thaw cycles after which the first cracks were observed. It also presents the destructive forces and the corresponding values of strength in the 3-point bending strength test f_{tb} and compressive strength test f_c .

Specimen	Water abs.	Water abs.	Frost resistance	Bending force	Compressive force	Bending strength	Compressive strength
~	[g]	[%]	[cycle]	[N]	[N]	f _{tb} [MPa]	f _c [MPa]
M0	55.18	14.46	1	101	1402	0.24	0.9
MS	39.64	10.73	3	133	3201	0.31	2.0
MH	25.06	7.06	7	150	3950	0.35	2.5

Table 2. The results of tests for water absorption, 3-point bending strength f_{tb} and compressive strength f_c

Figure 2a and Table 2 show the results for the amount of water absorbed by the tested specimens and figure 2b for frost resistance (number of cycles until the first signs of destruction are visible).



Fig. 2. Tested mortars: a) water absorption, b) frost resistance - number of cycles

The results for 3-point bending tensile strength are shown in Figures 3a and 3b presents the results for compressive strength.



Fig. 3. Strength: a) 3-point bending tensile strength f_{tb} , b) compressive strength f_c

Figure 4 presents specimen MH during the frost resistance test. Figure 4a shows the detached hardened layer. The impregnated layer is approx. 3mm thick. The hardened layer can be visually distinguished from the remaining specimen structure. Figure 4b shows air bubbles appearing during the thawing (soaking) of the specimens with water after the freeze cycle. The appearing bubbles indicate that the specimen exhibited diffusion properties. Similar phenomena were also observed in specimens M0 and MH.

Figure 5 shows the frost resistance results for reference mortar M0 and mortars MS and MH. The photographs of beam halves before the frost resistance test (0 cycles) and after 1, 2, 7 and 9 freeze cycles are presented. Figure 5 shows examples of specimens with the visible destruction process at different cycles of the frost resistance test. Red markers were placed on the photographs to indicate the first cracks occurring during the freeze-thaw process. Some of the cracks are only clearly visible when the photographs are zoomed in. In the reference specimen M0, the first cracks occurred after the first freeze-thaw cycle (Fig. 5b). Specimens MS showed the first destruction processes after 2 freeze-thaw cycles (Fig. 5c). Specimens MH presented the best frost resistance, exhibiting the first destruction processes after 7 frost cycles (Fig. 5d).



Fig. 4. Specimen MH during the frost resistance test: a) visible thickness of the hardened layer, b) air bubbles during the flooding of the specimen with water



Fig. 5. Frost resistance test—general appearance of the lime mortars M0, MS and MH: a) before the test, b) 1 cycle, c) 2 cycles, d) 7 cycles, e) 9 cycles

Specimens M0 and MS, with lower frost resistance, also showed a faster process of complete destruction after the first cracks occurred, compared to specimen MH, in which this process was much slower.

The photographs were taken after the thawing in water, immediately before the freeze cycle. Slight differences in the colors of the surface of presented specimens were caused by the fact that the photographs were taken under natural and artificial lighting (day and night).

Discussion

The conducted testing aimed at increasing the durability of traditional and historical lime mortars. It was proposed to modify the durability using liquid potassium silicate and silicic acid ester.

As is shown in Table 1 and Figure 2a, the impregnation of mortar M0 with potassium silicate limits the absorption of water from 14.46% to 10.73% (specimen MS). When silicic acid ester was used for impregnation, the amount of absorbed water was limited more effectively, from 14.46% to 7.06% (specimen MH).

The improvement of frost resistance (Table 2, Figs. 2b and 5) correlates with the limitation of water absorption. Impregnation with agent S slightly increased frost resistance from 1 cycle to 2 cycles (compare M0 and MS). The use of agent H increased frost resistance seven times (compare M0 and MH), indicating greater effectiveness.

Impregnations S and H slightly increase the bending strength of reference mortar from 0.24MPa to 0.31MPa (impregnation S) and 0.35MPa (impregnation H). The obtained results indicate that the proposed impregnation is not sufficiently effective to increase bending tensile strength.

The improvement of the compressive strength of the impregnated mortars is significant (Table 2 and Fig. 3b), from 0.9MPa for reference mortar to 2.0MPa (impregnation S) and 2.5MPa (impregnation H). When assessing the strength, it should be noted that the reinforcement of the structure only occurred on the surface, to a depth of approx. 3mm (Fig. 4a). Inside, the structure of the specimens did not change. A more than twofold increase in the strength with surface reinforcement of the specimens can be considered significant. The most effective impregnation occurs with the use of silicic acid ester (specimens MH).

No direct tests relating to the determination of the diffusivity coefficient were carried out (outside the scope of the study). As can be seen from Table 2, the amount of water absorbed after impregnation was reduced. Despite the reduction in the amount of water absorbed, the specimens diffusion properties are preserved after impregnation. This is evidenced by the water absorption capacity (Table 2, Fig. 2a) and the appearance of air bubbles when the specimens were thawed in water (Fig. 4b).

Summarizing the obtained results, it can be said that the proposed surface impregnation with liquid potassium silicate and silicic acid ester significantly increases the durability of traditional lime mortars. At the same time, the obtained results can also be applied to historical lime mortars. The improvement of durability by means of the proposed impregnation is the result of reduced water absorption, increased compressive strength and improved frost resistance.

The applied impregnation, either with liquid potassium silicate or silicic acid ester, does not cause a change in the color of the specimen surface. The slight differences in surface colors visible in the photograph result from the fact that the photographs were taken after freeze-thaw cycles in daylight and at night under artificial lighting.

It appears that the test results indicate the validity of using impregnation S and H to increase durability in relation to other materials characterized by relatively low strength (<5MPa) and significant water absorption.

The obtained test results show that silicic acid ester is a more effective way of improving the physical and mechanical properties of lime mortars compared to liquid potassium silicate, but the approx. three times higher cost of this type of impregnation may be a reason for the limitation of this solution.

Due to the brittleness of lime mortars, pressure impregnation cannot be used. The gravitational application modifies the physical and mechanical properties more effectively than surface impregnation but increases the volume of materials, which generates higher costs.

Conclusions

Significant improvement was noticed in the physical and mechanical properties of lime mortars with surface impregnation (liquid potassium silicate and silicic acid ester). A reduction in water absorption was achieved, with an increase in bending and compressive strength. The results indicate the possibility of increasing the durability of traditional and historical lime mortars with the use of the proposed method.

Impregnation with silicic acid ester results in the best improvement of the physical and mechanical properties of lime mortars. The effects of impregnation included a significant reduction in water absorption, an increase in compressive strength and a sevenfold increase in frost resistance, while diffusion capacity was maintained.

The applied method of impregnation, either with liquid potassium silicate or silicic acid ester, does not result in a change in the color of the surface.

The obtained results indicate that the achieved effects can also be valid for other building materials, particularly brittle absorbent materials.

Acknowledgments

The authors are grateful to KEIM Poland for supplying the impregnating agents—liquid potassium silicate and silicic acid ester.

References

- J.J. Hughes, *RILEM TC 203-RHM Repair mortars for historic masonry, The role of mortar in masonry an introduction to requirements for the design of repair mortars,* Materials and Structures, 45(9), 2012, pp. 1287-1294, DOI: 10.1617/s11527-012-9847-9.
- [2] E.A. Laycock, D. Pirrie, F. Clegg, A. Martin, T. Bell, P. Bidwell, An investigation to establish the source of the Roman lime mortars used in Wallsend, UK, Construction and Building Materials, 196, 2019, pp. 611-625, DOI: 10.1016/j.conbuildmat.2018.11.108.
- [3] E. Navrátilová, P. Rovnaníková, Pozzolanic properties of brick powders and their effect on the properties of modified lime mortars, Construction and Building Materials, 120, 2016, pp. 530-539, DOI: 10.1016/j.conbuildmat.2016.05.062.
- [4] D. Logoń, J. Kobaka, J. Domski, Modifying water-frost resistance and mechanical properties of lime mortar using siliceous and fluidised bed fly combusted ashes activated with cement. Materials, 16(8), 2023, art. 3013, pp. 1-16, DOI: 10.3390/ma16083013.
- [5] J. Jasieńko, D. Logoń, W.J. Misztal. *Trass-lime reinforced mortars in strengthening and reconstruction of historical masonry walls*, Construction and Building Materials, 102, 2016, pt. 1, pp.884-892, DOI: 10.1016/j.conbuildmat.2015.08.098.
- [6] A.M. Forster, Building conservation philosophy for masonry repair part 2 'principles', Structural Survey, 28 (3), 2010, pp. 165-188, DOI: 10.1108/02630801011058906.
- [7] J.J. Hughes, C. Groot, K. Van Balen, J. Elsen, RILEM TC 203-RHM: *Repair mortars for historic masonry, The role of mortar in masonry: an introduction to requirements for the design of repair mortars*, Materials and Structures, 45, 2012, pp.1287-1294, DOI 10.1617/s11527-012-9847-9.
- [8] M. Carosell, S.A. Ruffolo, F. Piqué, Mortars and plasters how to manage mortars and plasters conservation, Archaeological and Anthropological Sciences, 13, 2021, Article Number: 188, pp. 1-20, DOI: 10.1007/s12520-021-01409-x.
- S. Kang, S. Lee, S. Hong, Y. Kwon, *Historical and Scientific Investigations into the Use of Hydraulic Lime in Korea and Preventive Conservation of Historic Masonry Structures*, Sustainability, 11(9), 2019, Article Number: 5169, DOI: 10.3390/su11195169.
- [10] J.J. Hughes, K. Callebaut, *Practical Sampling of Historic Mortars*, RILEM International Workshop, Historic Mortars Characteristics, and Tests (Eds: P. Bartos, C. Groot, J.J. Hughes), Paisley, Scotland, 1999, pp. 17-26.
- [11] M. Arandigoyen, J.I. Alvarez, Pore structure and mechanical properties of cement-lime mortars, Cement and Concrete Research, 37(5), 2007, pp.767-775, DOI: 10.1016/j.cemconres.2007.02.023.
- [12] L. Schueremans, Ö. Cizer, E. Janssens, G. Serré, K. Van Balen, *Characterization of repair* mortars for the assessment of their compatibility in restoration projects: Research and

practice, **Construction and Building Materials**, **25**(12), 2011, pp. 4338-4350, DOI: 10.1016/j.conbuildmat.2011.01.008.

- [13] G. Chiari, G. Torraca, M.L. Santarelli, *Recommendations for systematic instrumental analysis of ancient mortars: the Italian experience*, Standards for preservation and rehabilitation (Editor: S.J. Kelley), ASTM STP 1258, American Society for Testing and Materials, West Conshohocken, 1996, pp. 275-284, DOI: 10.1520/STP15442S.
- [14] I. Sandu, Modern Aspects Regarding the Conservation of Cultural Heritage Artifacts, International Journal of Conservation Science, 13(4), 2022, pp. 1187-1208.
- [15] M. Weaver, F.G. Matero, Conserving Buildings-Guide to Techniques and Materials, New York: John Wiley & Sons, 1997, pp. 133-137.
- [16] Y. Ivashko, I. Buzin, I.G. Sandu, D. Kuśnierz-Krupa, J. Kobylarczyk, A. Dmytrenko, Ł.J. Bednarz, *State-of-the-art technologies of imitation of mural painting from the kyivan rus and baroque periods in the reconstructed St. Michael Golden-Domed Cathedral in Kyiv*, International Journal of Conservation Science, 13(1), 2022, pp. 147-162.
- [17] W. Kurdowski, Cement and Concrete Chemistry, Cement Producers Association, PWN, Warsaw, 2010 [in Polish,].
- [18] V. Caprai, A. Lazaro, H.J.H. Brouwers, Waterglass impregnation of municipal solid waste incineration bottom ash applied as sand replacement in mortars, Waste Management, 86, 2019, pp. 87-96, DOI: 10.1016/j.wasman.2019.01.025.
- [19] J.J. Chen, D. Zampini, A. Walliser, *High-pressure epoxy-impregnated cementitious materials for microstructure characterization*, Cement and Concrete Research, 32(1), 2002, pp. 1-7, DOI: 10.1016/S0008-8846(01)00620-2.
- [20] R. Zawawi, P.F.G. Banfill, Effect of burning conditions on properties of artificial hydraulic lime mortar obtained from limestone and siliceous waste materials, Advances in Applied Ceramics, 105(4), 2006, pp. 175-178, DOI: 10.1179/174367606X121295.
- [21] V.S. Samchenko, A.V. Korshunov, Formation of Cellular Concrete Structures Based on Waste Glass and Liquid Glass, Buildings, 14(1), 17, 2024, pp.1-19, DOI: 10.3390/buildings14010017.
- [22] A. Padovnik, F. Piqué, A. Jornet, V. Bokan-Bosiljkov, Injection Grouts for the Re-Attachment of Architectural Surfaces with Historic Value—Measures to Improve the Properties of Hydrated Lime Grouts in Slovenia, International Journal of Architectural Heritage, 10(8), 2016, pp. 993-1007, DOI: 10.1080/15583058.2016.1177747.
- [23] C. Pasian, M. Secco, F. Pique, G. Artioli, S. Rickerby, S. Cather, *Lime-based injection grouts with reduced water content: An assessment of the effects of the water-reducing agents ovalbumin and ethanol on the mineralogical evolution and properties of grouts*, Journal of Cultural Heritage, 30, 2018, pp. 70-80, DOI: 10.1016/j.culher.2017.10.003.
- [24] B. Rousset, S. Gentile, J. James, B. Pozzi, *Injection grouts for molasse sandstones: Preliminary assessments*. In Proceedings of the **RILEM Workshop Repair Mortars for Historic Masonry**, Delft, The Netherlands, 2005, pp. 26-28.
- [25] A. Padovnik, V. Bokan-Bosiljkov, Effect of Ultralight Filler on the Properties of Hydrated Lime Injection Grout for the Consolidation of Detached Historic Decorative Plasters, Materials, 13(15), 2020, Article number: 3360, pp. 1-19, DOI: 10.3390/ma13153360.

- [26] S. Wang, I. Sandu, Y. Ivashko, M. Krupa, A. Krukowiecka-Brzęczek, T. Yevdokimova, S. Stavroyany, O. Kravchuk, A.V. Sandu, *Methods for the preservation and restoration of Dunhuang wall paintings: foreign experience*, International Journal of Conservation Science, 15(1), 2024, pp. 731-748, DOI: 10.36868/IJCS.2024.01.24.
- [27] O. Molodid, O. Kovalchuk, Y. Ivashko, D. Kusnierz-Krupa, P. Tisliar, A. Dmytrenko, Ł. Bednarz, Research for Banksy mural 'Judoki' in Borodyanka, International Journal of Conservation Science, 14(4), 2023, pp. 1381-1390, DOI: 10.36868/IJCS.2023.04.08.

Received: November 22, 2024 Accepted: March 24, 2025