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Salt crystallization resistance of nano-modified repair lime mortars

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Abstract

This paper describes the results of the application of nano-binders (SiO2 and TiO2) in repair lime mortars, focusing on the durability of the composites against salt crystallization. A total of 12 new mixtures have been designed, produced and investigated in the laboratory, in an effort to produce durable, environmentally friendly, energy-efficient, sustainable composites, primarily for conservation purposes but also for contemporary applications. The effect of different aggregates and binders on the end-product was also investigated using a combination of physico-mechanical tests and analytical experimental techniques (e.g. MIP, capillary absorption, flexural and compressive strength tests). The results show that the use of nano-binders generally enhances the mechanical properties of lime mortars, without significantly affecting their pore structure. They also suggest that nanosilica enhances the durability of aerial lime mortars, while nanotitania significantly improves the resistance of hydraulic lime mortars subjected to salt weathering by full immersion. Nevertheless, attention should be drawn to the development of a standardized salt crystallization test methodology, which would be more appropriate for relatively weak masonry materials, such as lime mortars.

Keywords: lime, mortars, nanosilica, nanotitania, salt crystallization

1 Introduction

In the field of tangible heritage conservation, the use of compatible, yet durable, restoration materials is of upmost importance, in order to prevent further irreversible damage on monuments and art objects that have survived over the centuries [e.g. 1-2]. Despite the fact that traditional cementless mortars (e.g. lime-based mortars) are generally weaker in mechanical performance, when compared to most of the modern cement-based composites, their physical properties, such as their relatively high permeability and flexibility, can significantly benefit the durability of the masonry [3]. Nevertheless, since deterioration primarily affects the weakest materials in a structure, mortars are usually much more prone to weathering damage than other masonry materials; therefore relevant durability studies should be thoroughly carried out before any application of repair mortars whatsoever. With salt crystallization being widely considered as one of the most important weathering factors in the field of porous materials [4], the resistance of repair mortars to salt damage is vital. This paper presents ongoing research on the effects of selected nano-materials (SiO_2 and TiO_2) in the performance of lime-based composites. At the same time, it investigates the influence of two different aggregates and lime binders on the nano-modified end-products and it focuses on their durability against salt weathering.

1.1 The use of nanosilica and nanotitania in construction materials

The application of nanotechnology in the field of construction materials has resulted in significant improvements in recent years. Existing relevant literature mainly focuses on cementitious composites. The use of nanosilica, in particular, in concrete, cement mortars and pastes, has been widely investigated [e.g. 5-8]. Well-dispersed silica nanoparticles have been found to accelerate the hydration process and enhance pozzolanic activity. Moreover, nanosilica improves the materials microstructure by providing a more homogeneous, denser and compact structure. Hence, it may lead to materials with higher compressive strength and lower porosity. Despite the relatively broad research on the use of nanosilica in cement-based composites, this additive has only recently been tested in lime-based materials [9-10]. In these cases, the addition of nanosilica dramatically changed the mesopores distribution, causing a decrease of pores in the range of 20-100 nm and a rise of gel pores (<10 nm). The nano-sized particles also provided a large reactive total surface that led to the formation of Calcium Silicate Hydrate (C-S-H), thus accelerating the hydration process and the “filling” effect [11]. At the same time, the reduction of the mean pore size and porosity improved the mechanical properties of the composites. Nanotitania, on the other hand, has recently been used as an additive in cementitious mixtures, mostly due to its photocatalytic properties. It is considered an efficient and environmentally friendly photoactive material due to its non-toxic nature and ability to oxidize organic pollutants, producing harmless end-products [12]. Therefore, it has been used in the production of self-cleaning materials, which reduce structural maintenance costs [13]. Although significant research regarding the effects of nanotitania in cement-based composites has been published already, only very limited relative scientific data exists in the field of lime binders [14-15]. Yet, the results are promising and show an increase in the compressive and flexural strengths of the materials, while they also prove the enhancement of their elastic modulus over time. Acceleration of carbonation and hydration has also been noticed due to the addition of nanotitania in lime-based mixtures. Experimental data regarding the durability of optimized composites, after the addition of nanosilica and nanotitania, against different weathering phenomena, and in particular salt attack, are rather limited, even in the case of cement-based materials. However the results published so far regarding artificial weathering laboratory tests indicate the high potential of such additives. Frost resistance of concrete mixtures has been considerably improved by the addition of nano-SiO_2, which behaved not only as a promoter of the pozzolanic reaction but also as a filler, thus improving the pore structure and densifying the microstructure of the material [16]. A study on High Performance Concrete (HPC) mixtures, modified with different amounts of nanosilica, showed an increase in their freeze-thaw resistance [17-18], while improvement of the dynamic elastic modulus of frost-weathered HPC samples has also been reported [18]. Similar tests on air lime mortars have recently shown that the addition of nanosilica gives rise to more resistant composites [19]. Nanosilica has also been used as a potential additive for enhancing the properties of concrete in terms of salt resistance. The formation of a denser structure limited the transport of sodium sulphate ions and their subsequent reaction with $Ca(OH)_2$ [20]. Other studies showed that, when cement paste with added nanosilica was kept in sulphate solution for 180 days, less reduction in its compressive strength was observed, compared to the reference mixture [21]. Regarding the salt resistance of lime-based composites, very limited published data exists; this is, however, encouraging and shows that the presence of large amounts of nanosilica improves the durability of air lime mortars by delaying the decay evolution of magnesium sulphate solution

[19]. To the best of the authors´ knowledge, no relevant results have been published regarding the effect of nanotitania.

The present study aims at expanding current knowledge on the effects of nanosilica and nanotitania on the salt crystallization resistance of lime-based mortars, thus triggering further research on this subject. Due to the lack of a suitable standardized methodology to study the salt weathering phenomenon on mortars under laboratory conditions, this attempt adopted the concept of full immersion in sodium sulphate solution and subsequent drying. It should be noted that this test is considered to be excessively aggressive and inconsistent with what occurs in the natural built environment, let alone the fact that it has been performed on relatively weak materials such as lime mortars. An alternative to the full immersion test could be considered the “Continuous Partial Immersion Test” that was firstly introduced by Lewin in 1982 [22]. This procedure lets the solution to be absorbed by capillarity forces and allows the water to evaporate naturally, leaving the salt ions behind [23]. Many scientists used this method [22-24], since they consider it beneficial over other methodologies, due to the fact that the results for different specimens and salt combinations can easily be compared. However, no standardized procedure exists for this method either.

2 Materials and experimental procedure

2.1 Design of mortars

Two different types of binder were used for the production of the nano-modified mortars: aerial lime (A: CL80 by Hellenic Mining Public Co.) and natural hydraulic lime (H: NHL 3.5 by Lafarge). 3% of the lime binder was occasionally replaced by either nanosilica (nSi: Submicron Silica 995, 993 by Elkem) or nanotitania (nTi: Aeroxide® TiO_2 P25 by Evonik Industries). Crushed local calcareous sands from Mitsero (M), derived from an extensively recrystallized reef limestone, and Potamia-Latouros (L), originating from a clastic limestone consisting of grains cemented with a calcite matrix, were also used in the mixtures [25]. The binder to aggregates ratio (B:A) was kept constant at 1:3, since this has been found to be the prevalent ratio in ancient lime mortars both in Cyprus and in other areas of the world [26-27]. The quantity of water required for every mixture was estimated based on a constant workability of 165±5 mm; the water to binder ratios (W:B) for the 12 different mortar mixtures produced in the laboratory are given in Table 1, together with the rest of mix design details. All quantities given in this table were measured by mass.

Table 1: Mix design, expressed in mass, of laboratory produced mortars. M: Mitsero sand, L: Potamia-Latouros sand, A: Aerial lime, H: Hydraulic lime, nSi: nanosilica, nTi: nanotitania

Mixture	Aggregates		Binder				Workability (mm)	B:A	W:B
			Lime		Additive				
	M	L	A	H	nSi	nTi			
MA	3		1				167.81	1:3	0.99
LA		3	1				163.41	1:3	0.91
MAnSi	3		0.97		0.03		167.73	1:3	1.04
LAnSi		3	0.97		0.03		166.05	1:3	0.92
MAnTi	3		0.97			0.03	160.85	1:3	1.00
LAnTi		3	0.97			0.03	162.67	1:3	0.92
MH	3			1			163.80	1:3	0.94
LH		3		1			160.30	1:3	0.84
MHnSi	3			0.97	0.03		162.16	1:3	0.96
LHnSi		3		0.97	0.03		166.15	1:3	0.86
MHnTi	3			0.97		0.03	161.93	1:3	0.96
LHnTi		3		0.97		0.03	160.68	1:3	0.84

2.2 Testing methodology

2.2.1 Physical and mechanical properties

In order to characterize both the physical and the mechanical behaviour of the mortars, several conventional laboratory tests were carried out and various analytical techniques were used, at different time intervals after the day of casting. In this paper, we present results recorded 180 days after the production day of each mixture. Open porosity (p_o) and apparent density (p_a) were measured on prismatic specimens (40x40x160 mm) by vacuum saturation, using water as the wetting liquid. Water capillary absorption (s) was also measured on the same specimens. Additionally, porosimetric analyses (e.g. open porosity, apparent density, average pore size, pore size distribution) were carried out on bulk samples using Mercury Intrusion Porosimetry (MIP). Prismatic specimens (40x40x160 mm) were also tested under flexural load (three point bending); the materials mean flexural strength (FS) was estimated using the results from three specimens. The mean uniaxial compressive strength (UCS) of each mixture was also measured based on the results of six cubic test specimens (40x40x40 mm).

2.2.2 Salt crystallization resistance

In order to evaluate the durability of each mortar mixture against salt crystallization, artificial weathering was performed under controlled laboratory conditions. For the purposes of this test, cubic specimens (40x40x40 mm) were subjected to a maximum of 15 wetting and drying cycles using a 10% w/w aqueous solution of Na_2SO_4. The test for each mixture began 180 days after casting. During the wetting cycle, the specimens were fully immersed in the salt solution at 20 °C for two hours. After that, they were put in an oven at 105 ± 5 °C for at least 16 hours. In order to estimate the durability of each specimen after each crystallization cycle, two parameters were considered: a) qualitative evaluation based on visual inspection and b) quantitative evaluation based on percentage loss in mass. For the purposes of the qualitative evaluation, the specimens were macroscopically investigated and their condition was "scored", following a five-level scale which is described in Table 2.

Table 2: Scale for visual inspection of the materials resistance to salt weathering.

Score	*Description*
Perfect	*Specimen intact*
Good	*Very minor damage or minor cracks*
Moderate	*Rounding of corners and several cracks or detachment of small fragments*
Bad	*Specimen with several major cracks or broken*
Fail	*Specimen in pieces or disintegrated*

3 Results and discussion

The results of the physico-mechanical characterization of the 12 laboratory mixtures carried out 180 days after the production day are summarized in Table 3.

Table 3: Physico-mechanical properties of the laboratory mortars. Values in brackets represent standard deviations of the calculated mean values. p_o: open porosity, p_a: apparent density, s: water capillary absorption, FS: flexural strength, UCS: uniaxial compressive strength. See Table 1 for sample abbreviations.

Sample	p_o		p_a		Av. pore size	s	FS	UCS
	(%)		(kg/m^3)		(nm)	(mm/min$^{1/2}$)	(MPa)	
	Under vacuum	MIP	Under vacuum	MIP	MIP			
MA	32.3	30.4	1766	1579	288.4	0.28	0.91 (0.05)	3.5 (0.1)
LA	30.9	27.0	1802	1669	279.1	0.88	0.95 (0.02)	3.5 (0.1)
MAnSi	30.3	29.1	1758	1687	199.0	1.68	1.21 (0.07)	5.3 (0.1)
LAnSi	30.8	25.5	1777	1740	197.5	1.34	1.37 (0.11)	6.1 (0.2)
MAnTi	32.2	28.6	1749	1775	327.0	0.64	0.75 (0.04)	3.4 (0.1)
LAnTi	30.5	27.2	1776	1706	264.4	1.08	1.00 (0.07)	3.5 (0.1)
MH	35.1	29.0	1687	1790	32.7	0.88	1.66 (0.06)	7.3 (0.1)
LH	30.7	29.0	1704	1870	34.5	0.10	1.88 (0.08)	7.7 (0.1)
MHnSi	35.7	30.7	1679	2024	33.7	0.07	2.31 (0.09)	8.6 (0.4)
LHnSi	34.4	31.4	1701	2020	34.2	0.17	2.50 (0.14)	9.9 (0.2)
MHnTi	35.7	32.8	1671	1792	36.6	0.57	1.70 (0.06)	6.5 (0.1)
LHnTi	33.6	31.6	1717	1892	34.1	0.38	1.96 (0.07)	8.0 (0.1)

Higher flexural and mechanical strengths are attributed to the use of Latouros sand and hydraulic lime. Significant strength increases were also noticed in all mixtures where nanosilica was added, irrespective of the binder and aggregate type. Less significant improvements were observed with the addition of nanotitania to mixtures with Latouros sand, while decreases in strength were recorded in some cases where nanotitania was added to mixtures cast using Mitsero sand. It is worth noting that Latouros sand is much finer (0-2 mm) than the recrystallized Mitsero sand (0-4 mm). The latter also has a much different mineralogical composition, as it only consists of calcite and dolomite. The systematically lower porosity values given by MIP may be attributed either to the samples´ highly tortuous pore network, which does not allow mercury to fully intrude them [28], or to the fact that the size of some pores may be outside the range (360-0.003 mm) which can be detected by the MIP system [29-31]. Generally, lower porosity values were observed when Latouros sand was used. The addition of nanosilica or nanotitania to aerial lime mortars also led to a decrease in open porosity and average pore size; the opposite trend was noticed in the case of hydraulic mortars. Lower porosity was surprisingly observed in most of the aerial lime mixtures, compared to the hydraulic ones. The latter could be considered rather misleading and can possibly be explained by carbonation effects on the external surfaces of the samples. With regards to the capillary absorption results, the addition of nanotitania seems to have led to significantly reduced (over 160%) sorptivity values in the case of aerial mortars (compared to the addition of nanosilica); the opposite applies to hydraulic mixtures. It is also worth mentioning that, despite the changes in the physical property values observed before and after the addition of nano-binders, the mode of pore size distributions and the intensity of peaks recorded by MIP for the relevant mixtures do not show any significant changes (see Figure 1). The positive effect of the nano-binders is also apparent in the qualitative and quantitative evaluation of the samples subjected to salt crystallization tests (Figures 2 and 3). For example, even though the reference mixture LH has survived the test, the addition of nanotitania significantly improved the quality of the specimens, which in this case scored “Perfect” (intact material) even at the end of the 15th cycle. The better performance of Latouros mixtures is obvious when comparing the qualitative salt crystallization test results of all five surviving mixtures. The latter was also clear in the case of reference mixtures (MA & LA, MH & LH).

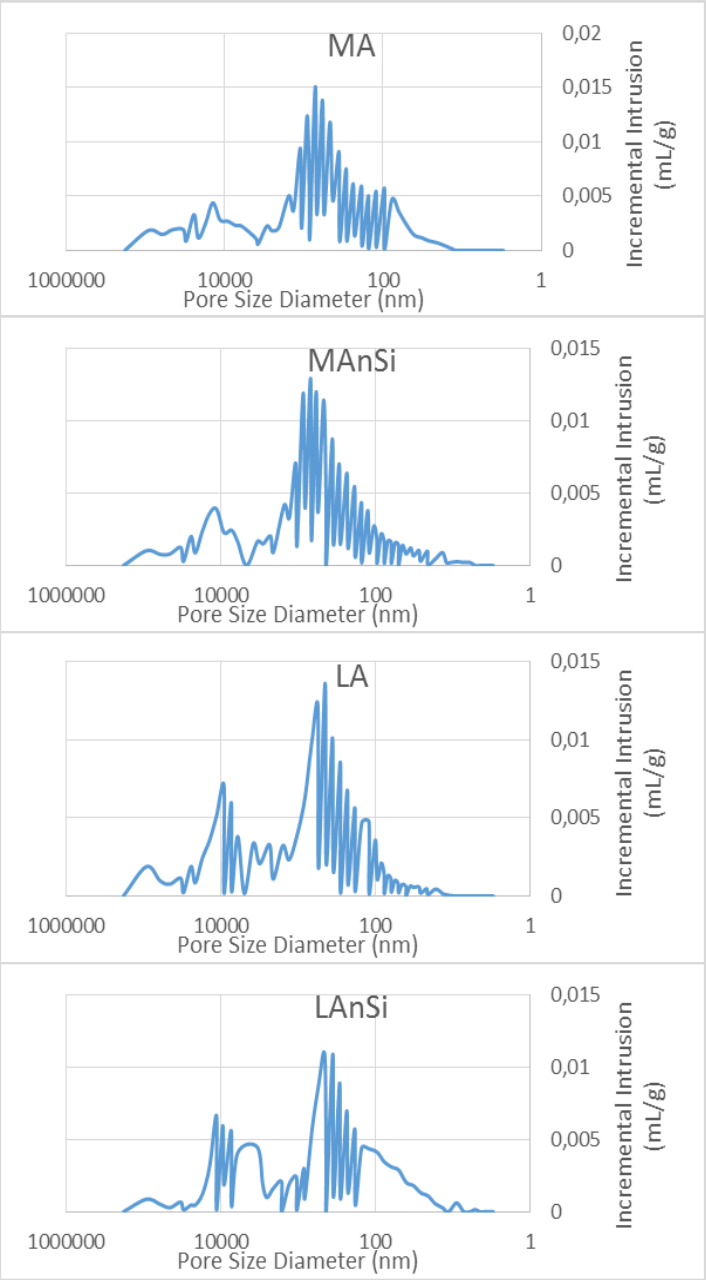

Figure 1: Indicative pore size distributions (see Table 1 for sample abbreviations).

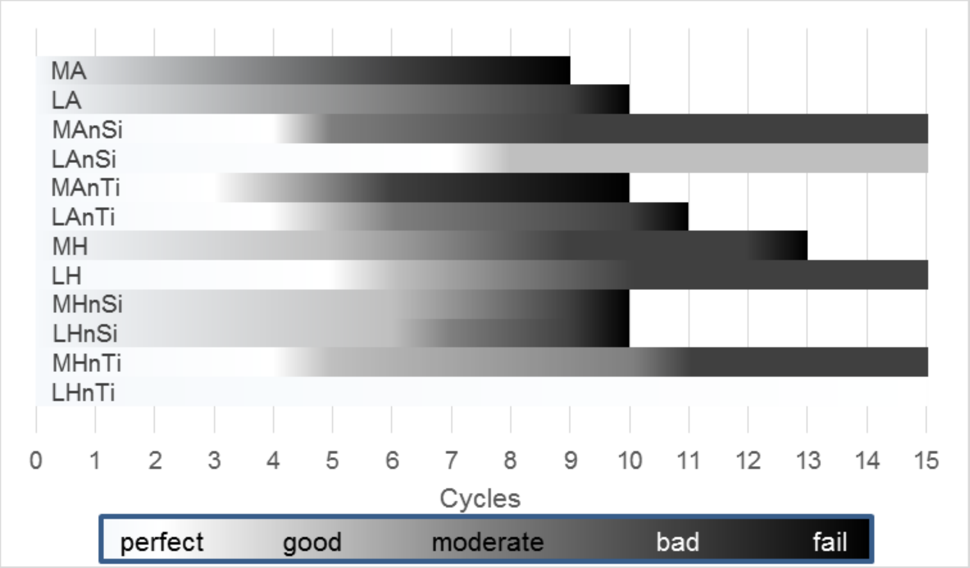

Figure 2: Qualitative evaluation of salt resistance by visual inspection of mortar mixtures. For explanation on the scale used see Table 2, while for sample abbreviations see Table 1.

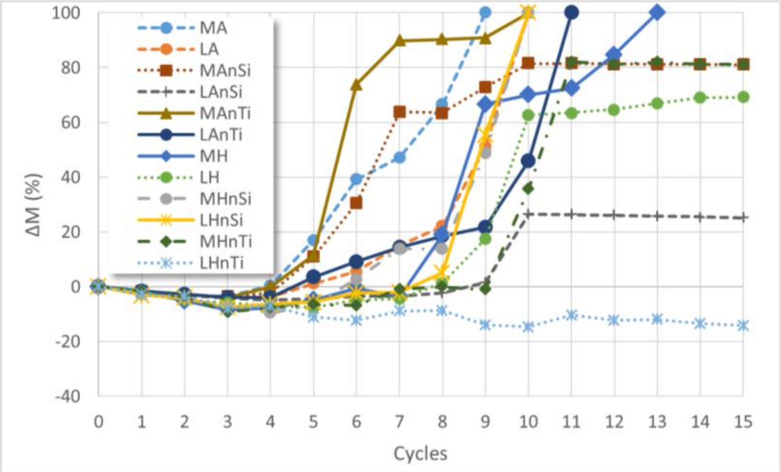

Figure 3: Weight loss (ΔM) of specimens after salt crystallization cycles (see Table 1 for sample abbreviations).

Quantitatively, the better durability results also corresponded to the mixtures with Latouros sand. This was found to be in agreement with the materials´ relevant strengths and it can be attributed to the aggregate´s finer particles, as well as to its mineralogical composition and in particular to the presence of significant amounts of hard minerals, such as silicates [25]. Only five mixtures survived the 15 cycles of artificial salt weathering test (MAnSi, LAnSi, LH, MHnTi and LHnTi). Irrespective of the aggregate type, nanosilica addition highly improved the performance of aerial lime mixtures, while nanotitania significantly enhanced the resistance of the two nano-modified hydraulic mixtures. The considerably positive effect of nanotitania on the durability of hydraulic lime mortars could be attributed to its known potential in enhancing the formation of hydraulic phases in the mixtures, which is in turn the result of its highly hydrophilic nature [15]. Complimentary thermal analyses of the same mixtures (not shown here) support this conclusion, while ongoing FTIR measurements aim to confirm the results achieved. Despite the fact that the addition of nanotitania in aerial lime mortars did not contribute to the successful accomplishment of the salt crystallization test, improvement of durability was nevertheless noticed, compared to the reference mixtures. Last but not least, lower amount of mass loss was generally noted in the case of hydraulic mortars (MHnSi & LHnTi), while the negative value of LHnTi could be attributed to insignificant loss of material and simultaneous accumulation of salt crystals in the pore structure of the specimens.

4 Conclusions

Generally higher strengths and better resistance to salt weathering were observed in the cases of mortars with Latouros sand. This can be attributed to the finer particle size distribution of this aggregate and to its mineralogical composition. Significant improvements of the materials´ strengths were also noted when nanosilica was added in the mixtures. Less noticeable changes in strength, at the time interval of 180 days after the day of production, corresponded to the addition of nanotitania. Further testing at longer time intervals is needed to investigate this finding. The modifications of several physical property values due to the addition of nanomaterials did not lead to any noticeable change in the pore size distribution of the mixtures. The most durable aerial lime mortar samples in terms of salt resistance were found to be the mixtures with nanosilica additive; the use of nanotitania, on the other hand, proved to be efficient in the enhancement of the durability of hydraulic lime mortars. These findings are considered encouraging for meeting the goals of this ongoing project, while further research is needed in the case of nanosilica addition to hydraulic lime binder. Despite the positive outcomes of this research, the selected methodology which was followed in order to test the durability of laboratory lime mortars against salt attack is considered highly aggressive; for this reason, most of the mixtures could not reach the end of the test. Visual inspection highly contributed to a better evaluation of the materials performance. Nevertheless, attention should be urgently drawn to the need for a standardized experimental approach, which would be more appropriate in the case of relatively weaker masonry materials, such as lime mortars, and could enable the comparison of results at an international level.

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