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DURABILITY PROPERTIES OF NON-HYDROPHOBIZED LIME MORTARS WITH EXPANDED AGGREGATE: SALTS AND ICE CRYSTALLIZATION RESISTANCE

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ABSTRACT

Porous pozzolanically active aggregate can be used to prepare lightweight mortars with a high ability to salt accumulation from masonry and enhanced liquid water permeability. In this study, the comparison of non-hydrophobized expanded perlite, expanded glass, and expanded clay as lightweight aggregate in air lime mortars and natural hydraulic lime mortars has been investigated with emphasis on the resistance to salts and ice crystallization. The results of frost resistance tests were expressed as frost resistance coefficient for each mortar. The salt crystallization resistance of mortars were determined using 10% Na_2SO_4, 3% NaCl and 3% NH_4NO_3 solutions. The microstructure of mortars after the testing was monitored by scanning electron microscope and amount of anions absorbed in the mortars was determined by chemical analysis. The expanded aggregate significantly improved frost resistance of the mortars. The mortars with expanded perlite and expanded clay accumulated nitrates and sulfates to a greater extent, as a result, they disintegrated earlier. The combination of the best properties showed samples with 50% sand replacement with expanded glass - they were the most frost-resistant and the most resistant to salt crystallization.

Keywords: lime mortar, expanded aggregate, frost resistance, salts crystallization, porosity, microstructure.

INTRODUCTION

The use of lightweight aggregates such as expanded perlite, expanded glass or expanded clay is quite common in hydrophobized dry pre-mixed renders. The use of hydrophobized renders on historical masonry is more and more refrained by reason of their controversial long-term benefit. Non-hydrophobized salt transporting and salt accumulating renders are more appropriate than hydrophobized salt blocking and moisture sealing systems in the case of harder moisture/salt conditions. In many examples, application of hydrophobized renovation renders for uninsulated masonry on historical buildings led to the worsening of technical state of the treated structures. Typical examples are higher capillary rise, increased moisture content due to the hygroscopicity of accumulated salts, salt induced spalling and flaking of rendering materials, etc. (Gonçalves et al. 2008; Groot et al., 2009; Pavlík et al., 2015; Petković et al., 2010).

The application of lightweight aggregates (LWA) in composition of masonry and rendering mortars has been already studied by many researchers, e.g. (Barnat-Hunek et al. 2017; Pokorný et al. 2019; Sousa et al. 2019; Contrafatto et al. 2020). By using these aggregates, such diverse properties as weight reduction, growth of open porosity and sorptivity, thermal insulation and fire resistance or workability may be improved. Thus, many lightweight materials, such as expanded perlite, expanded glass, expanded clay, hollow microspheres and expanded polystyrene were used to improve these mortar characteristics (Silva et al. 2010). Expanded perlite, expanded glass and expanded clay are some lightweight materials that, because of their composition based on silico-aluminates, are potentially able to give pozzolanic reactions. They can therefore provide additional advantages in mortars by improving their mechanical strength (Palomar et al. 2015). Porous aggregate with pozzolanic properties can improve not only the mechanical properties of mortars, but also their ability to salt accumulation from masonry, frost resistance, and liquid water transport to the mortar surface. Detailed studies on the frost resistance and salts crystallization resistance of this type of lime mortars are still lacking.

In this study, the comparison of expanded perlite (EP), expanded glass (EG) and expanded clay (EC) as lightweight aggregate in air lime mortars and natural hydraulic lime mortars has been investigated with emphasis on the resistance of salts and ice crystallization. Mortars with half and full replacement of quartz sand by expanded aggregate were first characterized by their physical and mechanical properties and then subjected to freeze-thaw cycles as well as three different salt media in order to study their resistance to aggressive environments.

MATERIALS AND METHODOLOGY

A commercial hydrated lime CL90-S (Čertovy Schody, Inc., Lhoist group, Czech Republic), a natural hydraulic lime NHL 3.5 (Zement- und Kalkwerke Otterbein GmbH & Co. KG, Germany) were used as binders in prepared mortar mixes. Each mortars group consisted of reference samples made of quartz sand (Filtrační písky, Ltd., Czech Republic), and 3 types of samples with different expanded aggregate (0/2 mm), half and fully replacing quartz sand, namely, non-hydrophobized expanded perlite (EP 150 PB, fraction 0/2 mm from PERLIT PRAHA, Ltd., Czech Republic), expanded glass (LIAVER, fraction 0/2 mm from Liaver GmbH & Co. KG, Germany), and expanded clay (LIAPOR, fraction 0/2 mm from Lias Vintířov, LSM k.s., Czech Republic). The chemical composition of all raw materials is given in Table 1. The phase compositions obtained by the X-ray diffraction analysis are presented in Table 2. Some physical parameters of expanded aggregate used are shown in Table 3. Expanded aggregates contained large amount of hydraulic oxides (SiO_2, Al_2O_3, Fe_2O_3) – 86%, 73%, 93%, respectively, which, synergically with a high amorphous phase content (91%, 98%, 77%), is a very good prerequisite for aggregates` pozzolanic reactivity. The very low loose bulk densities and the thermal conductivities gave great preconditions for the production of lightweight thermal insulation mortars. The granulometry of aggregates was tested with a Mastersizer 2000 Ver. 5.60 laser analyzer. The size of expanded aggregates and quartz sand particles was very similar (Figure 1); thus, the aggregate replacement comprised the same particle sizes. Pozzolanicity of the aggregates was tested by a modified Chapelle test method according to the standard NF P 18-513 (Figure 1). The limit of consumed $Ca(OH)_2$ for the consideration of the material as pozzolanically active – 650 mg/g (Raverdy et al. 1980) was exceeded after 3 days of treatment for all aggregate used; EG was the most pozzolanically active. It was seen that the pozzolanic reaction of the aggregates evolved over time. Therefore, their effect on the properties of the mortars was expected after an adequately long period of time.

Mortar mixtures were made using the exact amount of water required to obtain a good workability of the mortars (160 ± 5 mm; measured by the flow table test). The composition of mortar mixtures considered constant binder: aggregate volume ratio of 1:1.15, which was chosen after conversion the 1:4 weight ratio in the reference lime mortar, Table 4. This weight ratio is commonly used in the preparation of lime renders in research and practice supported by the results obtained by (Lanas et al. 2003). According to the perlite producer's recommendation, non-hydrophobized EP was immersed for 24 h in water to eliminate its high water absorption capacity that could greatly worsen the workability of mortar mixtures. Fresh mortars were cast into prismatic moulds of size 40 × 40 × 160 mm. Hardened mortar specimens were demolded after 48 h and then cured in a wet chamber at temperature (T) of 22 ± 3 °C and a relative humidity (RH) of 95 ± 5% for 26 days. The samples were then stored under laboratory conditions at T = 22 ± 3 °C, RH = 50 ± 5%. During the entire ageing period, the samples were placed on plastic grids in order to make their surface as accessible as possible for carbonation.

Table 1 – Chemical composition of initial materials (wt%).

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	P_2O_5	TiO_2	SO_3	L.O.I.
Lime	0.92	0.71	0.39	68.09	1.33	0.48	0.11	0.05	0.10	0.19	27.94
NHL 3.5	12.76	4.12	1.47	59.87	2.79	1.13	0.09	0.15	0.05	0.15	15.28
Quartz sand	98.50	0.38	0.15	0.01	0.03	0.09	0.01	0.04	0.09	0.02	0.12
EP	68.02	16.04	1.91	4.54	0.41	2.50	4.62	0.14	0.10	0.02	0.33
EG	70.27	2.16	0.49	9.43	2.11	0.84	13.82	0.12	0.18	0.21	0.13
EC	55.84	23.27	13.86	5.04	0.12	0.48	1.10	0.05	0.04	0.14	0.11

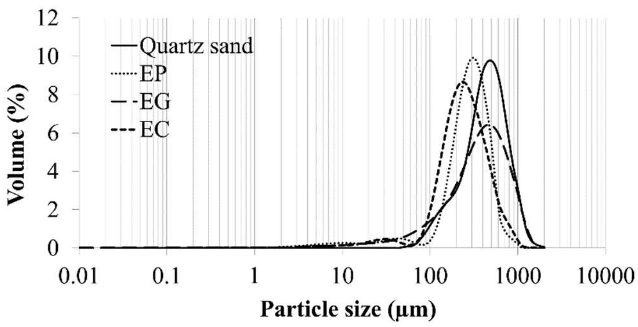
Table 2 – Mineralogical composition of initial materials (wt%).

Mineral	Lime	NHL 3.5	Quartz sand	EP	EG	EC
Albite	–	–	–	1.9	–	2.6
Aluminate	–	2.7	–	–	–	–
Anorthite	–	–	–	3.2	–	2.3
Biotite	–	–	–	2.8	–	1.1
Brownmillerite	–	1.4	–	–	–	–
Brucite	0.5	–	–	–	–	–
Calcite	1.8	6.2	–	–	–	–
Cristobalite	–	–	–	–	–	0.9
Mullite	–	–	–	–	–	7.5
Larnite	–	22.5	–	–	–	–
Portlandite	97.1	41.3	–	–	–	–
Quartz	–	–	98.3	0.4	2.2	8.4
Sanidine	–	–	–	0.6	–	–
Staurolite	–	–	1.5	–	–	–
Amorphous phases	–	25.1	–	90.8	97.7	77.1

Table 3 – Selected properties of expanded aggregates imparted by producers.

Property	EP	EG	EC
Loose bulk density [kg m^{-3}]	180	310	575
Water absorption [l m^{-3}]	350	25	35
Thermal conductivity [W m^{-1}K^{-1}]	0.04	0.07	0.12
Thermostability [°C]	900	750	1000
Water vapor diffusion resistance factor, μ-value [–]	3	5	–
Capillary evaporation [g h^{-1})	0.37	–	–
Compressive strength [MPa]	0.3	3	7
pH [–]	7	7	7

After 90 days of aging, the mortars were characterized by their physical and mechanical properties, water absorption of mortars was measured according to EN 13755:2008, and total porosity of the specimens was assessed using a mercury intrusion porosimetry (MIP). Frost resistance tests were carried out according to modified Czech standard ČSN 722452. The total tests required 15 freeze-thaw cycles. One cycle consisted of 6 h freezing of water saturated samples at −20 °C and 12 h thawing in a desiccator at constant relative humidity of 98 % and temperature of 20 °C. The frost resistance coefficient D_f was determined as the ratio of flexural strength of specimens subjected to 15 freeze-thaw cycles to the flexural strength of reference specimens that did not undergo the frost resistance test. The salt crystallization resistance of mortars was determined using following solutions: 10% Na_2SO_4, 3% NaCl, and 3% NH_4NO_3. The dried samples were immersed into the solutions for 7 h and then dried for 16.5 h at 60 °C. The process was performed in the number of 10 cycles or till partial disintegration of the samples. The procedure was performed according to the relevant European standard (EN 12370:1999). The state of the test specimens was monitored photographically, and detailed microstructure images were taken using scanning electron microscope (SEM) equipped with an EDX probe. The content of anions in aqueous leaches of the samples was determined by routine chemical analyses (10 g of mortar sample, 500 mL of deionized H_2O). Sulfates were set gravimetrically according to ISO 9280:1990, chlorides by mercurimetric method (ISO 5790:1979), and nitrates using Nitratax sc optical probe allowing the determination of nitrates directly in the medium.



Pozzolanic reaction time [days]	Pozzolanic activity [mg $Ca(OH)_2$/g]		
	EP	EG	EC
1	259	676	611
2	468	972	823
3	997	1077	879
4	1072	1172	959
5	1137	1234	1038

Fig. 1 – Particle size distribution and pozzolanic activity of expanded aggregates.

Table 4 – Proportioning of mortar mixtures.

	Lime [g]	NHL 3.5 [g]	Quartz sand [g]	EP [g]	EG [g]	EC [g]	H_2O [mL]
LQ	100	–	400	–	–	–	120
LEP50	100	–	200	71	–	–	73
LEP100	100	–	–	142	–	–	35
LEG50	100	–	200	–	37	–	125
LEG100	100	–	–	–	74	–	125
LEC50	100	–	200	–	–	70	125
LEC100	100	–	–	–	–	140	143
NHLQ	–	100	340	–	–	–	75
NHLEP50	–	100	170	60	–	–	34
NHLEP100	–	100	–	120	–	–	5
NHLEG50	–	100	170	–	31	–	73
NHLEG100	–	100	–	–	62	–	75
NHLEC50	–	100	170	–	–	59	90
NHLEC100	–	100	–	–	–	118	109

RESULTS AND DISCUSSION

Mechanical Parameters

The bulk density and strength properties of the investigated mortars are introduced in Figure 2. The lightening effect of the expanded aggregates used was quite apparent. The most significant reduction in bulk density was achieved by mortars lightened with expanded perlite, which corresponds to its lowest loose bulk density and also results in the lowest observed mortar strengths. The bulk density of mortars with incorporated lightweight aggregates was below the criterion of WTA directive 2-9-04/D (1400 kg m^{-3}). The drop in bulk density followed the increase in the porosity (Figure 3), whereas these two parameters were results of two combined effects: (i) low loose bulk density of expanded aggregates, and (ii) structural changes in the mortars due to higher amount of mixing water. In general, application of expanded aggregates gave less dense mortars meeting the demands for repair mortars. In respect to the presumed application of the developed mortars in salt and moisture laden masonry, their high porosity will enable safe salt accumulation and evaporation of stored water.

The highest strengths were recorded for the reference NHL mortar, however, 50% replacement of quartz sand with expanded aggregates did not lead to a significant drop in strengths and, in the case of EC, even 100% replacement of sand did not negatively affect the observed strengths of NHL mortars. Significant decreases in strength were observed for air lime mortars. According to the EN 998-1, all air lime mortars are ranked in category CS I and all NHL mortars belong to category CS III. Although, both the European standard EN 998-1 and WTA directive 2-9-04/D prescribe for repair mortars strength class CS II, there are many examples based on analysis of historical masonry where much lower compressive strength values are recommended for the repair of traditional lime rendering and plastering mortars. For example, (Nogueira et al. 2018; Veiga et al. 2001) recommended the repair mortars with a 90-day compressive strength in the range of 0.4–2.5 MPa. In this regard, the developed air lime mortars can be considered efficient for restoration purposes, especially in desalination of lime-based mortar constructed buildings.

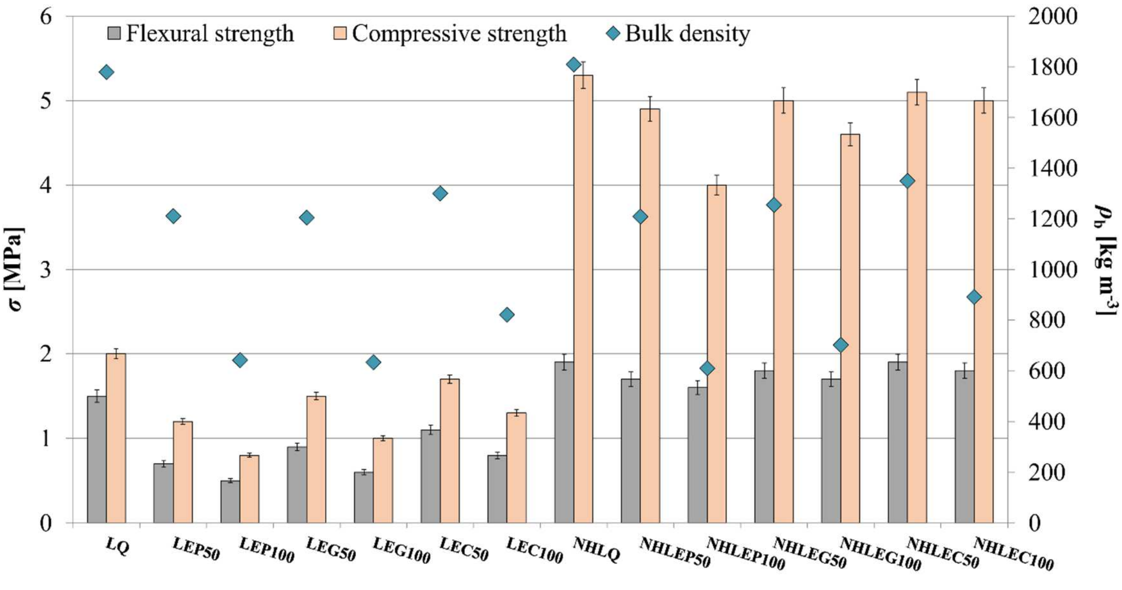


Fig. 2 – Flexural and compressive strength and bulk density of mortars at 90 days.

Total Porosity and Water Absorption

Since the resistance and durability of lime mortars is very dependent on their pore structure, the total porosity of the mortars (obtained by MIP) and the water absorption has been determined before the resistance tests (Figure 3). The increasing total porosity of the mortars is caused by two reasons, as already reported: (i) low loose bulk density of expanded aggregates, and (ii) high dosages of mixing water required to achieve the desired consistency of these mortars (Table 4), therefore the mortars with the lightest aggregate and the highest amount of mixing water showed the highest total porosity, which can be potentially beneficial for salt and ice crystallization resistance of these mortars. The type of lightweight aggregate had a great influence on the water absorption of mortars as well. Above all, the open porous structure of the aggregate and its water-binding capacity plays an important role in water absorption, which is especially evident when using expanded perlite. The measured values of total porosity and water absorption are more than 50% higher compared to the values of similar mortars with natural zeolite, lava sand or pumice (Vyšvařil et al. 2020). This is mainly due to the low bulk density of the expanded aggregates used in the present study.

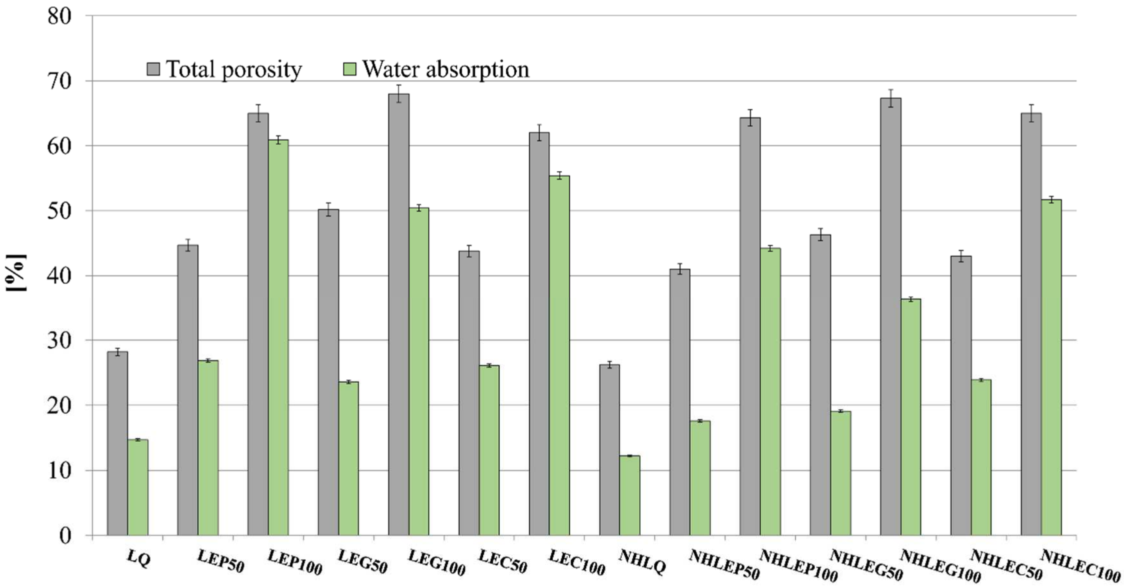


Fig. 3 – Total porosity and water absorption of mortars at 90 days.

Frost Resistance

Determination of frost resistance of mortars according to ČSN 72 2452 is a test of alternating freezing and thawing of water-saturated mortar beams in the number of 15 cycles. Due to the saturation of the samples with water, air lime mortars often break up in this test before the completed 15 cycles. In this study, all tested mixtures withstood 15 freeze-thaw cycles and it was possible to determine their flexural strengths and subsequently evaluate the frost resistance coefficients, D_f (Figure 4). The frost resistance of mortars increased with increasing porosity of the samples with EP and EC. Mortars with 100% sand replacement with expanded glass showed half the D_f value compared to mortars with 50% aggregate replacement. The negative effect of 100% sand replacement of EG on the frost resistance of foamed concrete has been observed by (Namsone et al. 2017) as well. In a series of air lime mortars, the highest values of D_f were achieved by LEG50 and LEC100 mixtures, but they still did not meet the criterion of frost resistance ($D_f = 0.75$). NHLEG50 and NHLEC100 mixtures can be designated as frost-resistant, as they have exceeded the limit of D_f necessary for the fulfilment of frost resistance. From the point of view of frost resistance, 50% expanded glass replacement and 100% expanded clay replacement appears to be the best of the studied quartz sand substitutes.

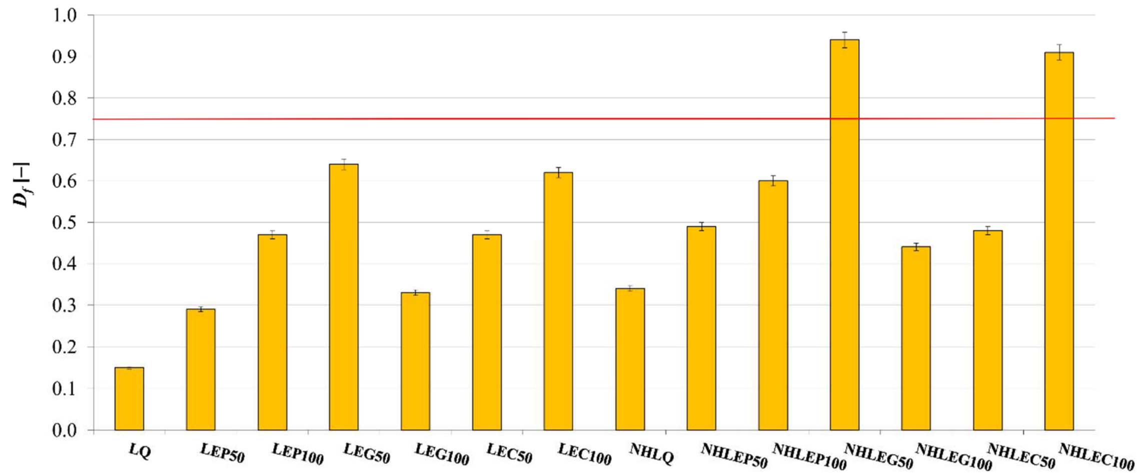


Fig. 4 – Frost resistance coefficient of mortars (red line – standard frost resistance criterion).

Salt Crystallization Resistance

The content of anions in aqueous leaches of the samples before and after salt crystallization resistance test is presented in Table 5 together with the sequence of the decay cycle. The results show that the concentration of the monitored anions in the samples after treatment with saline solutions increased more than 100 times, mostly in perlite mortars. Thus, it has been confirmed that increased porosity is a good prerequisite for higher ability to salt accumulation and mortar resistance to salt crystallization. Table 5 shows that all mortars studied are not resistant to crystallization of sodium sulfate, where gypsum with high molar volume are formed (Figure 5a). It is also evident from the values in the table that mortars accumulating larger amounts of nitrates (LEP, LEC, NHLEC) did not withstand all 10 cycles of crystallization tests. The relatively low ability of mortars to accumulate chlorides was also reflected in their integrity after 10 salt crystallization cycles (Figure 6).

The microstructure of mortar samples was determined before and after the salt crystallization resistance test. All monitored mortar samples with expanded aggregate showed similar crystalline and amorphous neoplasms after salts crystallization tests. Microstructure images of NHLEG50 mortar samples were selected for presentation as the most promising mixture (Figure 5). The images show that gypsum has the biggest rate in the decomposition of samples treated with Na_2SO_4. Its formation was enormous, and the structure became more and more porous until it disintegrated. The resistance of the studied mortars to NaCl crystallization was due to the low reactivity of NaCl with Ca^{2+} cations resulting in less leaching of the binder from the mortars, which is evidenced by the denser structure of the mortars with filled pores by amorphous NaCl (Figure 5b). Treatment with NH_4NO_3 solution resulted in the formation of nitrocalcite ($CaNO_3 \cdot 4H_2O$) covered most structural units with an amorphous coating (Figure 5c). The repeated recrystallization of the nitrocalcite during the cycling of the samples in the NH_4NO_3 solution led to the failure of some samples by wide cracks (Figure 6c), but there was no massive destruction of the samples as in the case of Na_2SO_4 treatment. The integrity and way of mortar decomposition was characteristic of individual saline solutions – in Na_2SO_4, the samples disintegrated suddenly and in the whole volume of the sample, in NaCl, the samples remained intact, and in NH_4NO_3, cracks and even breakage of larger pieces of samples occurred (Figure 6).

Table 5 – Concentrations of monitored anions in aqueous leaches of mortar samples before (c_{g0}) and after (c_g) salt crystallization resistance test, and number of test cycles to sample disintegration (10 = intact sample).

Mixture	c_{g0} SO_4^{2-} [g kg^{-1}]	c_{g0} Cl^- [g kg^{-1}]	c_{g0} NO_3^- [g kg^{-1}]	c_g SO_4^{2-} [g kg^{-1}]	c_g Cl^- [g kg^{-1}]	c_g NO_3^- [g kg^{-1}]	Cycle count in Na_2SO_4	Cycle count in NaCl	Cycle count in NH_4NO_3
LQ	**0.103**	**0.055**	**0.487**	**21.04**	**7.73**	**19.85**	**3**	**10**	**10**
LEP50	0.180	0.123	0.298	41.78	16.98	31.14	2	10	8
LEP100	0.213	0.139	0.185	45.12	17.33	36.47	2	10	8
LEG50	0.159	0.081	0.239	35.74	6.51	16.88	3	10	10
LEG100	0.163	0.098	0.245	33.62	7.06	17.27	3	10	10
LEC50	0.147	0.067	0.500	37.85	11.43	29.54	3	10	7
LEC100	0.236	0.099	0.542	38.96	12.14	33.41	3	10	7
NHLQ	**0.087**	**0.049**	**0.408**	**36.44**	**9.85**	**10.43**	**4**	**10**	**10**
NHLEP50	0.180	0.139	0.251	44.12	15.70	19.90	3	10	10
NHLEP100	0.188	0.141	0.134	47.86	17.32	20.20	3	10	10
NHLEG50	0.149	0.083	0.299	36.52	7.55	7.81	4	10	10
NHLEG100	0.151	0.159	0.264	38.11	5.51	10.83	3	10	10
NHLEC50	0.160	0.099	0.285	40.07	10.47	25.41	3	10	9
NHLEC100	0.164	0.165	0.398	46.33	12.77	31.42	3	10	9

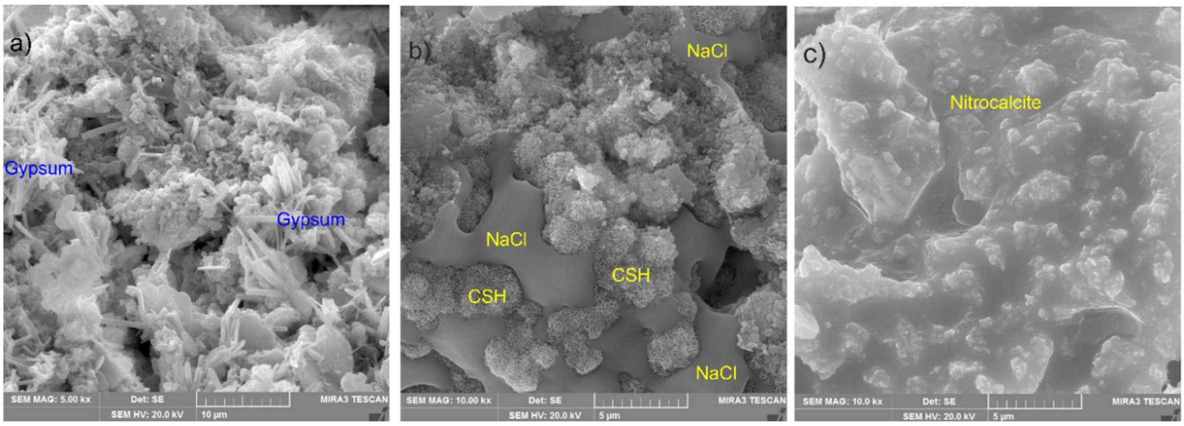


Fig. 5 – SEM microstructure images of NHLEG50 samples after salt crystallization resistance tests; (a) after 4 cycles in Na_2SO_4, (b) after 10 cycles in NaCl, (c) after 10 cycles in NH_4NO_3.

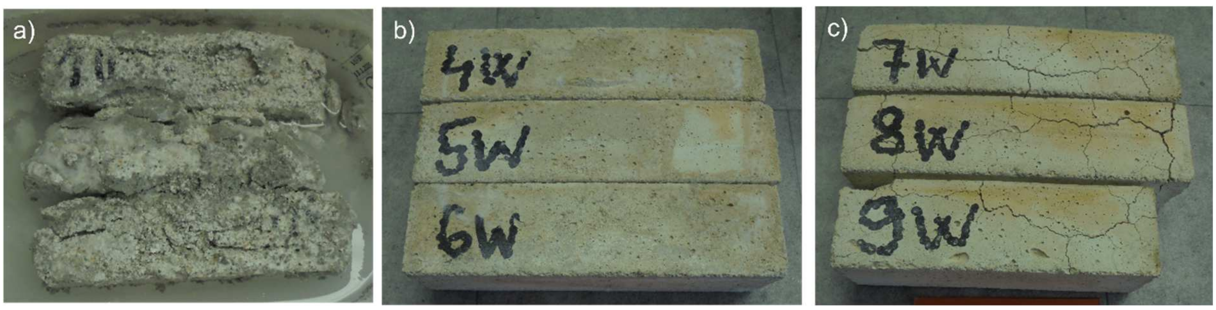


Fig. 6 – Images of NHLEG50 samples after salt crystallization resistance tests; (a) after 4 cycles in Na_2SO_4, (b) after 10 cycles in NaCl, (c) after 10 cycles in NH_4NO_3.

CONCLUSIONS

All studied expanded aggregates significantly reduce the bulk density of mortars by increasing the total porosity of mortars in accordance with their decreasing loose bulk density. For air lime mortars, the lightening results in a significant decrease in strength; for NHL mortars, the strengths decreases only slightly, but in both cases the mortars are usable for restoration purposes, for desalination and desiccation of masonry, which is also helped by their increased water absorption.

Replacement of quartz sand with expanded aggregate leads to a considerable improvement in the frost resistance of mortars; especially when using expanded glass or expanded clay. The concentration of the monitored anions in the samples after treatment with saline solutions considerably increases, mostly in perlite mortars. Thus, it has been confirmed that increased porosity is a good prerequisite for higher ability to salt accumulation and mortar resistance to salt crystallization. Expanded glass has proven to be an advantageous alternative aggregate in mortars for their lightening, for improving the frost resistance and salts crystallization resistance of mortars, but it does not contribute to salts accumulation in mortars, so it is not very suitable for desalination purposes. Conversely, expanded perlite in mortars fair increases their ability to accumulate salts.

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