LIME MORTARS WITH LINSEED OIL: ENGINEERING PROPERTIES AND DURABILITY

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Linseed oil has been used as mortars admixture in times before the modern synthetic admixtures were introduced. The present study deals with influence of linseed oil on the engineering properties of air lime mortar. The composition of mortar was inspired by a mortar used for fixing of 1890´ mosaic in Krásná Lípa (Czech Republic). The knowledge of complex influence of the oil on mortar's performance is important for the preparation of renovation mortars but also for possible application of the oil as sustainable mortar admixture, replacing the nowadays synthetic ones. The oil is reducing the yield stress of the fresh mortar (i.e. acts as plasticizer) and is increasing its air entrapment. The water-repealing effect is the most important impact of the oil admixing on the harden mortar; it was evaluated by measurement of water absorption coefficient. The higher air entrainment and water-repellence are resulting to the improved frost resistance of the mortar containing the oil.

Keywords: *mosaic, mortar, linseed oil, durability*

1. Introduction

The application of materials, which are copying the original ones, is frequently preferred in restoration and repair of historic buildings and works of art [1], because – among other reasons – materials incompatibility may cause further materials degradation [2]. Mortars and plasters prepared on lime basis have been used since ancient times in many world regions [3, 4] and, despite the Portland cement introduction in the late 19th century, the lime remains very important binder especially for mortars and plasters production until nowadays. In its basic form, lime mortar is composed from air or hydraulic lime, fine aggregates (sand, crushed stone – e.g. marble) and water. The (slaked) air lime is composed from $Ca(OH)_2$ (eventually – as dolomitic lime – it contains some $Mg(OH)_2$); hydraulic limes are containing also hydraulic components (reactive compounds of SiO_2 and/or Al_2O_3). Hydraulic lime may be "natural hydraulic lime" (NHL; hydraulic oxides are naturally contained in limestone used for lime production) or "hydraulic lime" (HL; mixture of air lime and a hydraulic compound – pozzolana). The hydraulic limes have beneficial effect, compared to air lime, on the mortar's strength, rate of strength gain and durability [5, 6]. The usage of hydraulic limes in general building practice has been replaced during the 20th century by adding of a small amount of Portland cement to the air lime [7].

Another way to improve durability of a lime based mortar is modification of its properties by an admixture. Admixture, in this context, is a matter (liquid or solid), dosed to binder typically in range between 0.5 to 2% (by weight of cement or lime), modifying the various properties of fresh and/or hardened mortar (concrete). Natural admixtures in lime systems have been used since ancient times [8, 9]. Nowadays, the application of synthetic admixtures (i.e. better defined, more effective, with higher stability) is dominant in engineering practice, but the traditional admixtures of natural origin, are of importance for the restorations works, and possibly also for sustainable and green construction.

The natural species useable as lime admixtures can be classified, according to their chemical nature, to three groups: proteins, polysaccharides and triglycerides (glycerine esters of fatty acids, i.e. oils and fats). The proteins used as lime admixtures has been collagen (animal glue), casein or egg proteins [10 - 12]. Among polysaccharides, e.g. starch or carrageenan were studied as viscosity modifying admixtures [13, 14]. The present paper deals with influence of linseed oil on the engineering properties of aerial lime based mortar. This oil was identified in historic mosaic mortars and it is also cited in ancient recipes [15, 16]. The primary motivation of oil utilization in the mortar was its water-repealing ability which results to the improved freeze-thaw resistance of the mortar [17]. Ventola et al. [10] used olive oil as admixture and observed significant reduction of porosity and average pore size. In China, tung oil has been used as water – repealing agent in mortars [9]. In modern age, spent cooking oil or engine oil were

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Table 1

Composition of prepared mortars (in g).

Mortar	Lime	Quartz 0-0.5 mm	Quartz 0.5-1 mm	Limestone	Water	Oil
MC	1000	750	250	500	900	0
M0.5	1000	750	250	500	900	5
M1	1000	750	250	500	900	10
M1.5	1000	750	250	500	900	15
M2	1000	750	250	500	900	20

tested as water repealing admixtures in cementitious concrete and lime mortar [18, 19].

The goal of the present paper is to evaluate possibility to utilize linseed oil as water repealing admixture in air lime based mortar. The particular composition of mortar was inspired by a mortar found in 1890´ mosaic mortar in Krásná Lípa (Czech Republic).

2.Experimental methods

The historic mosaic mortar was characterized by X-ray diffraction and by scanning electron microscopy in order to get its fundamental composition characteristics. X-ray diffractograms were recorded by help of Malvern PANalytical Aeris diffractometer equipped with $Co_{K\alpha}$ source operating at 7.5 mA and 40 kV. The incident beam path consisted of iron beta-filter, Soller slits 0.04 rad and divergence slit 1/2°. The diffracted beam path was equipped with 9 mm anti-scatter slit and Soller slits 0.04 rad. The used detector was PIXcel1D-Medipix3 detector with active length 5.542°. Data were evaluated by Profex software (ver. 3.12.1) [20]. The SEM micrography by help of Phenom XL (15 kV acceleration voltage and BSE detector were used).

The set of reproduced mortars, inspired by the historic one, was prepared (Tab. 1). Slaked air lime (CL 90 S, Lhoist Czech Republic) was used as binder. Two size fractions of quartz sand and crushed limestone were used as filler. Food-grade linseed oil was dosed from 0.5 to 2% by mass of the lime. According to [21], the linseed oil contains more than 50% of linolenic acid (with three double bonds), about 40% of mono and di-unsaturated (oleic and linoleic) acids and the rest are saturated palmitic and stearic acids. The mixing procedure [22] consisted in mixing of all dry components; the oil was dispersed separately in small amount of dry mix and then this suspension was mixed with the rest of dry components and finally water was added. The specimens for hardened mortar testing were prepared by help of standard 40 x 40 x 160 mm moulds; the prisms were stored in laboratory condition (22 ± 1 °C; 55 ± 5% of RH) and kept moist in order to prevent fast drying and to ensure the carbonation course. A set of auxiliary cubic specimens (without limestone flour and with initial composition 2:1 lime:quartz) was prepared in order to monitor the influence of linseed oil on the carbonation course. These auxiliary specimens were prepared and stored the in same way as the "main" samples. The consistency and entrained air content of fresh mortar was measured by standard procedures (EN 1015-3 and 7) by help of flow table test and pressure type air content meter. Rheology of mortars was studied by help of Rheoloab QC (Anton Paar) with ball measuring system. This system was designed for the structural materials, where immersed steel ball of diameter 8 mm is rotating in the studied material. Used measuring system accomplishes just one round during the test with logarithmically increasing shear rate to eliminate footprint, which occurs in the measured material. Integrated software RheoCompassTM V1.23.403 ensures acquisition of the actual shear rate γ (s^{-1}), shear stress τ (Pa) and the viscosity η (Pa·s), which allow the determination of the main rheology characteristics. The compressive and flexural strength was determined after 14, 28 and 62 days of curing by MTS Criterion 43 machine. The 62 days old samples were subjected to pore size distribution measurement (mercury intrusion porosimetry by devices Thermo Pascal 140 and 440). The mortars carbonation course was examined by X-ray diffraction (XRD) analysis with the same parameters as described above. Samples were collected from the surface of specimens and stored, until the analysis, in sealed containers in desiccator. The rate of carbonation was evaluated in terms of degree of conversion (α) of portlandite ($Ca(OH)_2$) defined as Eq. (1) where w_0 is the initial content of portlandite, w is actual content of portlandite in given time.

$$\alpha = 100\frac{w^0 - w}{w^0}\,[\%] \qquad (1)$$

The rate of liquid water transport in mortars was characterized by help of absorption coefficient A [kg m^{-2} $s^{-1/2}$] determined by help of automated imbibition experiment with laterally insulated cubic samples [23]. In this experiment, the amount of soaked water i [kg m^{-2}] is measured in time t (Eq. 2).

$$i = At^{1/2} \qquad (2)$$

The freeze-thaw resistance of mortars was tested by cycling of prismatic specimens according to the temperature/time program shown in Fig. 1. The samples were first let to soak by water for twice 24 hours (from both lateral sides, depth of water pool 1 cm), then sealed to a plastic bag and subjected to cycling between 8 and -18 °C. The samples were sealed for the whole test period in order to keep the moisture content constant. The compressive and flexural strength were measured after 50, 100 and 150 cycles. The experiment was evaluated in term of index of frost resistance – i.e. ratio of flexural strength after given number of cycles and initial flexural strength of the material.

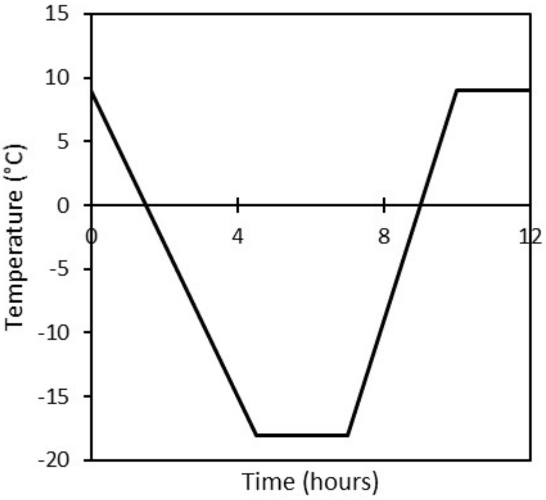


Fig. 1 - Time/temperature program for a single freeze/thaw cycle.

3. Results

3.1 Historic mosaic mortar

The experimental program presented within this paper was inspired by mosaic found in Dittrich sepulchre in Krásná Lípa (German: Schönlinde) in Northern Bohemia (Czech Republic) (Fig. 2). The currently heavily damaged mosaic dates back to ca 1890 and its authorship has not been ascertain indisputably. It belongs the oldest "modern" (not mediaeval) mosaics in Czech Republic. The X-ray diffractogram (Fig. 3) revealed that the mortar contains calcite ($CaCO_3$; AMCSD 0000989) and quartz (SiO_2; AMCSD 0008971) as the principal components, accompanied by traces of feldspar, mica and kaolinite coming from the used sand (where quartz was the main component). The binder was thus lime, possibly weakly hydraulic as

Fig. 2 - Mosaic in Dittrich sepulchre in Krásná Lípa (German: Schönlinde) in Northern Bohemia (Czech Republic).

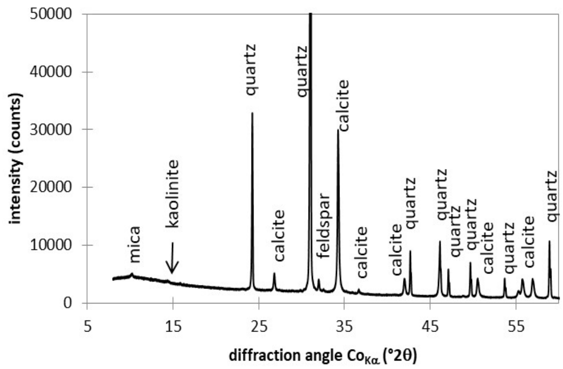


Fig. 3 - X-ray diffractogram of historic mosaic mortar.

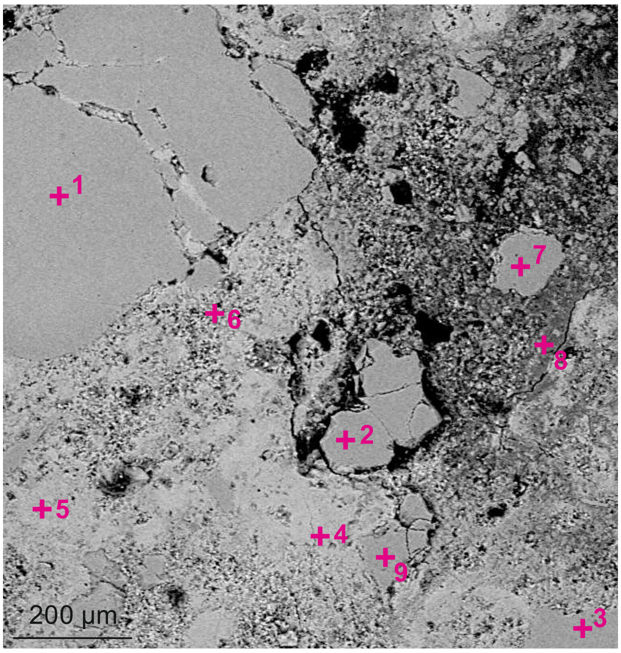


Fig. 4 - SEM micrography of historic mosaic mortar. Points 1, 2, 3, 7 and 9: quartz grains. Points 6 and 8: carbonated lime. Points 4 and 5: marble powder used as filler.

it is indicated by the small amorphous hump in XRD. The SEM micrography (Fig. 4) shows various areas found in the mortar. Points 1, 2, 3, 7 and 9 are quartz grains (identified by EDS spectroscopy). Points 6 and 8 are indicating porous areas – the carbonated lime ($CaCO_3$) phase (EDS indicated Ca, C and traces of Si and Al). The denser areas of similar composition (points 4 and 5) are grains of calcite; the crushed calcite (marble) was frequently used as component of mosaic mortars [24].

3.2. Prepared mortars

3.2.1. Rheology of mortars with linseed oil

The standard method for the rheology characterization of mortars according to EN 1015-3 is based on the flow table (Fig. 5); the results indicate that the linseed oil is linearly increasing the flow value, i.e. it is acting as plasticizer. Moreover the oil – already in its lowest dosage - is increasing the content of entrained air from 2 to 4%; . The rheometer with ball measuring system was used for more extensive rheology investigation. The viscosity curves (Fig. 6 left) indicate that the mortar is non-Newtonian fluid; its viscosity is decreasing with applied shear rate and growing oil content. The flow curves (Fig. 6 right) obey the Bingham model (3), i.e. the mortar can be called "Bingham plastic"; the Bingham-like behaviour was in lime based mortars already identified [25]. In Bingham model, τ is shear stress [Pa], τ_0 yield stress [Pa], η_{pl} is plastic viscosity [Pa·s] and γ is shear rate [1/s].

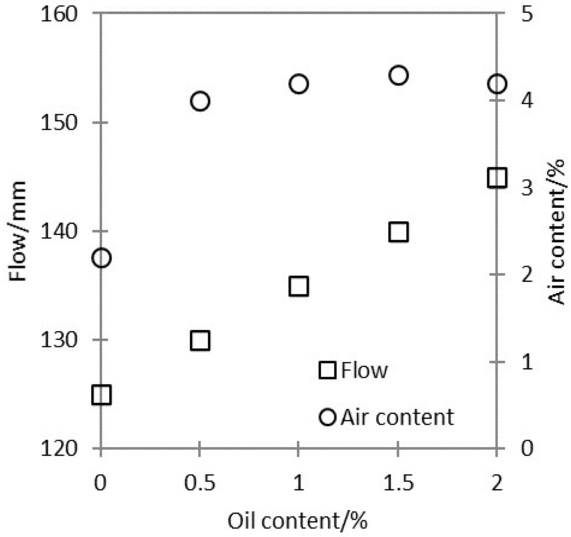
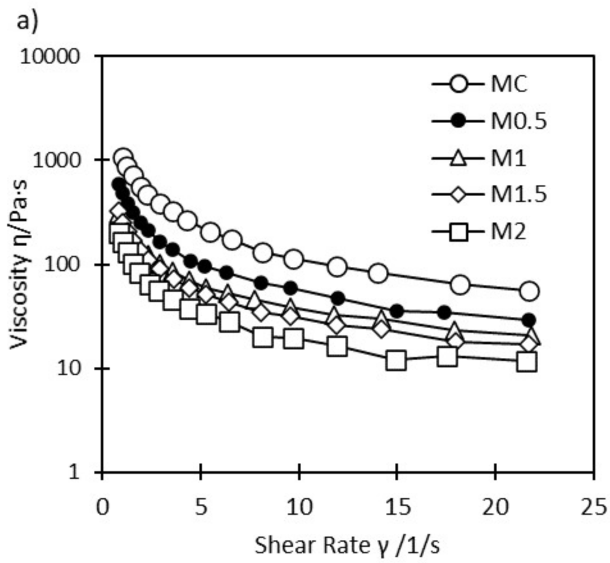


Fig. 5 - Dependence of flow consistency and entrained air content on the linseed oil content.

$$\tau = \tau_0 + \eta_{pl} \cdot \gamma \qquad (3)$$

The rheology parameters obtained from Bingham model (Tab. 2) clearly indicates, that the oil content reduces significantly the yield stress; this measure is inversely proportional to the flow measured by flow table [26].



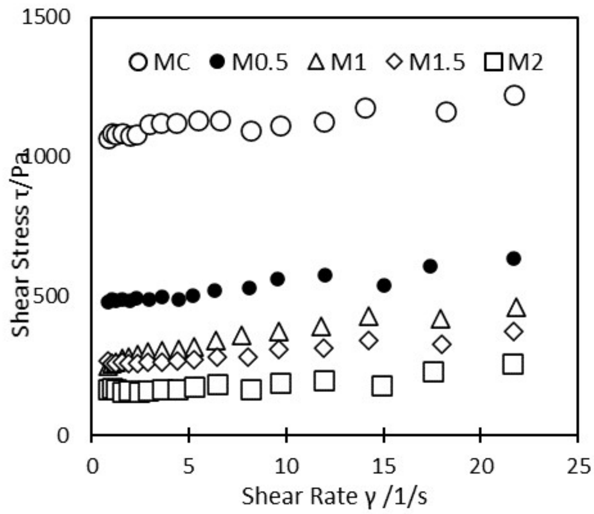
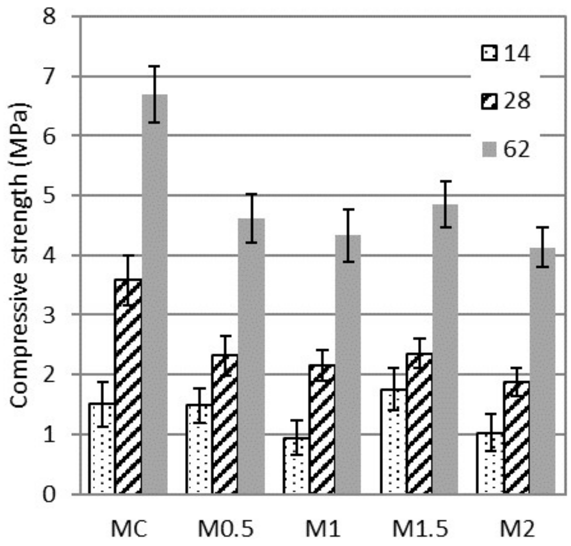


Fig. 6 - Viscosity (a) and flow curves (b) of mortars with linseed oil.

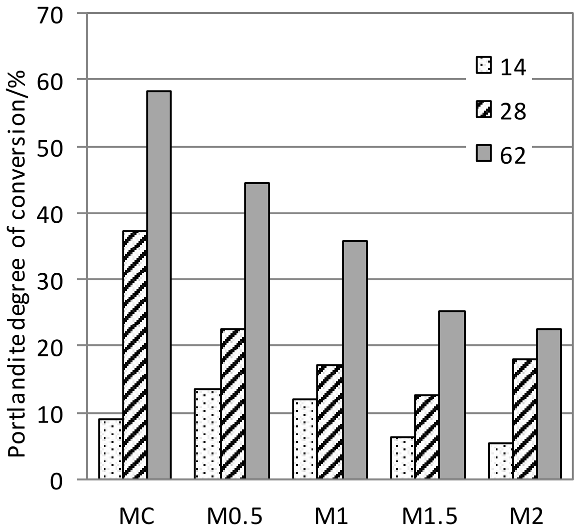
Table 2

Rheology parameters of mortars.

	MC	M0.5	M1	M1.5	M2
plastic viscosity η_{pl} (Pa·s)	5.75	7.20	9.90	5.24	3.87
yield stress τ_0 (Pa)	1078	472	263	249	153
flow (mm)	125	130	135	140	145



a



b

Fig. 7 - Compressive strength (a) and portlandite conversion (b) in time (14, 28 and 60 days).

3.2.2. Hardening of mortars with linseed oil

The hardening of air lime based mortars is supposed to be controlled by the carbonation of portlandite ($Ca(OH)_2$) to calcite according to the reaction (4). Even though water is not present as reactant in the process, its presence is necessary since the calcite precipitation takes place in water solution and the rate of carbonation depends (among other) also on relative humidity of surroundings [27].

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \qquad (4)$$

The hardening process was monitored by measurement of compressive strength of mortars (Fig. 7) in 14, 28 and 62 days. The control mortar reached more than 6 MPa in two months, what can be considered as a good result for that type of material [28]. Apparently the presence of linseed oil reduced the rate of strength development. The simultaneous measurement of the portlandite conversion in auxiliary samples, monitored by XRD, was used to evaluate the rate of carbonation process. The selected diffractograms (62 days of curing, Fig. 8) clearly show that higher oil dosage means higher portlandite content, i.e. slower progress of reaction (4). The portlandite degree of conversion (1) was used to quantify its depletion in pastes (Fig. 7). The conversion of portlandite is obviously increasing in time in all samples and it is the fastest in the control mortar MC. The higher oil content, the slower rate of carbonation. When one compares both plots in Fig. 7, there is an observable difference: the rate of carbonation is gradually (and negatively) dependent on the oil content, while the strength is reduced in roughly the same extent for all tested oil contents (from 0.5% to 2%). It indicated that the strength is not controlled just by the level of carbonation.

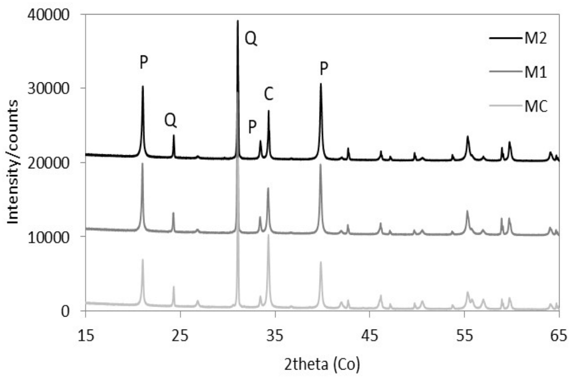


Fig. 8 - Diffractograms of MC, M1 and M2 mortars (age 62 days). P-portlandite, C-calcite, Q-quartz.

3.2.3.Durability characteristics of prepared mortars with linseed oil

The mortars pore size distribution is a crucial parameter, which is closely related to its mechanical and durability properties [29]. The prepared mortars were characterized by mercury intrusion porosimetry (Fig. 9). The mortars have unimodal pore system. The control mortar reached the lowest total pore volume; the increasing oil content caused increasing pore volume. Moreover, the character of the pore system has been changed by the oil presence. The control mortar MC contained considerable number of capillary pores between 1 to 100 μm – volume of these larger pores was reduced by the oil, but the volume of the smaller ones (< 1 μm) gradually increased. The pore size distribution of a material is closely influencing its ability to transport (not only) liquid water [30]; higher porosity means more space for flowing liquid, on the other hand smaller pore diameter means more intensive capillary suction. The rate of water intake by capillary activity to the mortars was measured by help of sorptivity experiment (2). The course of water sorption to the samples (Fig. 10) shows significant differences between individual mortars. The water absorption coefficients are calculated from the initial linear part of the measured data. The obtained values (Tab. 3) are extremally dependent on the oil content. The mortar M2 reached about two orders of magnitude lower rate of capillary water transport compared to the control mortar MC, despite its significantly higher porosity. The final plateau of absorption curves does not have a clear relationship to the absorption capacity of the material; nevertheless the oil containing mortars (with higher porosity) reached also the higher cumulative water content compared to the control.

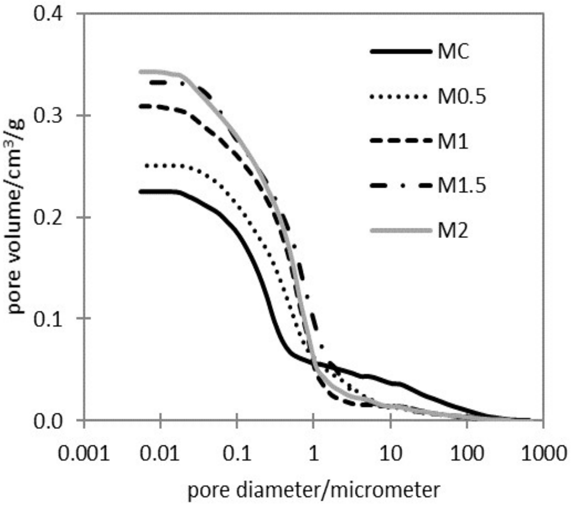


Fig. 9 - Pore size distribution of mortars.

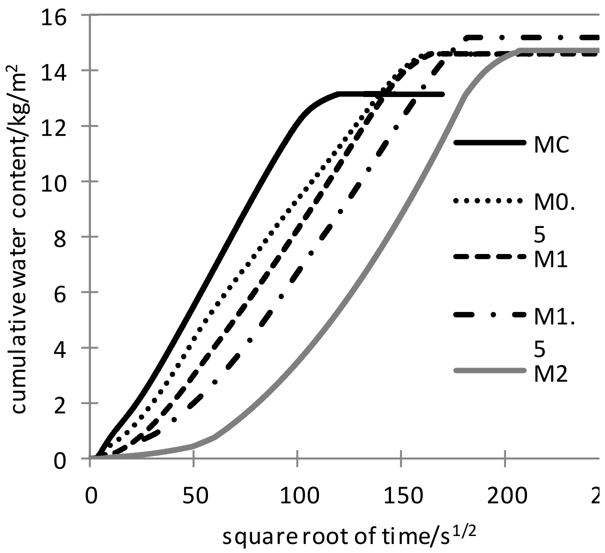


Fig. 10 - Course of free water intake experiments.

Table 3

Water absorption coefficients of mortars.

Material	A/kg/(m^{2}s$^{1/2}$)
MC	0.098
M0.5	0.061
M1	0.032
M1.5	0.016
M2	0.002

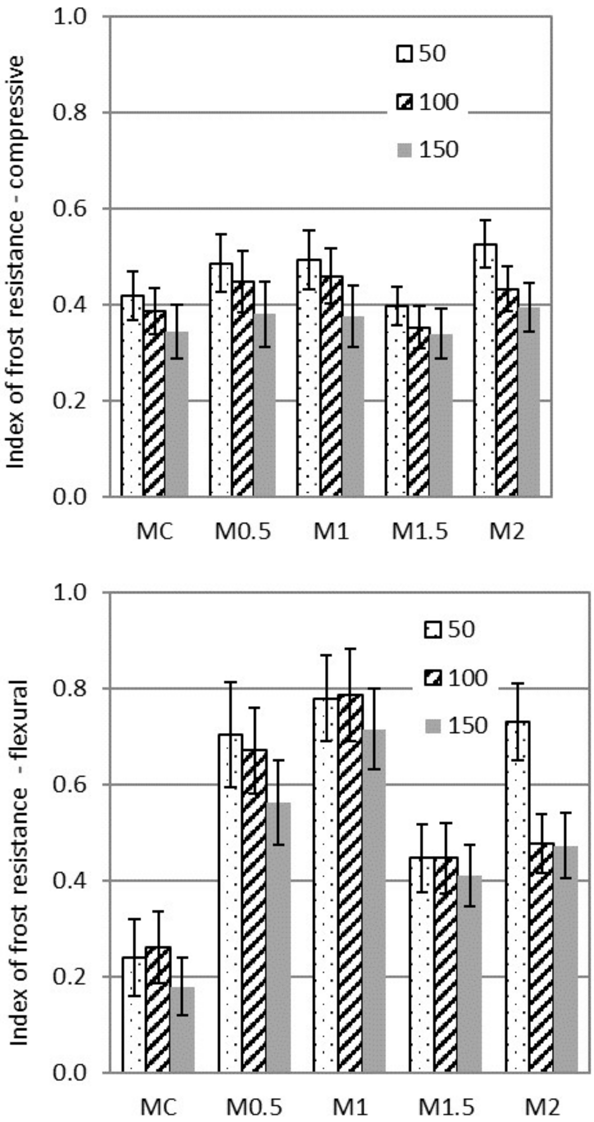


Fig. 11 - Index of frost resistance of mortars determined by means of compressive and flexural strength.

The frost resistance of cured mortars was studied and expressed by help of index of frost resistance (i.e. ratio of strength after the given number of cycles and initial strength of the material) (Fig. 11). The results were plotted for both kinds of studied loading – flexural and compressive. The results of control mortar MC are very poor; already 50 freezing cycles caused loss of strength to 40/20% (compressive/flexural strength). Further cycling caused further loss of strength, although not substantially. It should be pointed out, that even though the strength of mortars decreased due freezing/thawing process, the samples kept their shape and integrity. The admixing of linseed oil improved significantly the frost resistance in terms of flexural strength – especially the sample M1 reached very good results. The difference between compressive and flexural resistance indexes indicates that the oil presence is reducing the fragility of the mortar, what is very beneficial in case of mosaic mortar or generally mortars and plasters for building coatings applications.

4.Discussion

The linseed oil is known for centuries as a mortar admixture aiming to reduce the water penetration into the mortar (i.e. as eater-repealing agent) [22], but also – dosed in much higher amount – the oil was used for preparing a mastic (*stucco* in Italian) for mosaics laying [16]. Here the motivation was not the water repellence, but the reduction of mortar hardening rate. The present experimental program aimed to describe impact of linseed oil on a fine-grained air lime mortar, inspired by a historical mosaic mortar. Such mortar could be used either for restoration purposes, but also in nowadays constructions where linseed oil would be a more sustainable alternative for synthetic admixtures.

The linseed oil acted as a plasticizer of fresh mortar – it increased the flow of the mortar in terms of EN 1015-3 standard. The same behaviour of observed when a mineral oil was used as admixture in concrete [18, 31]. The performed study of lime-oil mortars rheology confirmed the – for lime expected – Bingham-like behaviour [25]; the oil reduced the yield stress of mortar and thus acted in opposite way to viscosity enhancing polysaccharides [14]. The positive effect of oil on plasticity is something what one would expect since its generally lubricating nature, but triglycerides are not used nor studied as cement concrete plasticizers due to their detrimental effect on strength [18]. The observed positive effect of oils on air entrapment was reported both in cement and lime based systems [17, 18, 31]. This finding seems to be supported by the porosimetry results (Fig. 9), but one has to realize that air entrapment is responsible for larger pores than those which are detected by MIP. The retarding effect of oil on lime carbonation rate was published many times [19, 21, 22]. Similar effect was observed when a cementitious mortar was treated by water-repealing silanes – the presence of hydrophobic admixture retarded the cement hydration. This is explained by fact, that both processes – carbonation of lime and hydration of cement – are taking place only in the presence of liquid water. Nevertheless the retarded carbonation/hydration is not necessarily accompanied with the strength gain retardation [32, 33]. In this study, the strength loss was not proportional to the oil content, but just to the oil presence. The oil is not just retarding the rate of carbonation, but it also changes the microstructure of carbonated lime – mortars with oil contains higher amount of amorphous $CaCO_3$ compared to the control [21]. The effect on microstructure was indirectly observed also in this study by help of porosimetry – the oil caused increase of porosity and of the mean pore diameter.

So far in this text, linseed oil was considered as a stable admixture, but in fact, the triglycerides are decomposing in alkaline environment to glycerol and calcium salts (called soaps) of corresponding fatty acids (saponification process) [34]. Obviously, salts of fatty acids, e.g. stearates, are commonly used water repealing admixtures [33], the saponification is not influencing the water-repealing ability. Since the linseed oil contains mainly polyunsaturated fatty acids, they (or their salts) may undergo oxidative polymerization, so finally, in the mortar is not present the original oil, but the product of its polymerization. The positive effect of oil admixture on frost resistance is reached by the combination of its air-entraining ability [35] and hydrophobicity. As the optimum oil dosage may be considered 1%, what is in agreement with [17]. This dose is likely the optimum between the oil's positive (air entrapment, water repellence) and negative effects (strength reduction). Interesting is the observed (Fig. 11) discrepancy between the frost resistance indexes obtained from compressive and flexural load. Elsewhere [17] an opposite behaviour was observed and explained by the presence of cracks in samples, which are more critical for flexural strength. The better performance in the flexure load compared to the compression was observed by authors of [36] in slag-based geopolymer modified by styrene-acrylate polymer latex; an optimum (2.5%) latex dose improved the flexural strength while the compressive one was gradually decreasing with the latex content. The explanation of such behaviour lies probably in formation of polymer film in the material, which positive effect is at higher content outweighed by the polymer's effect on porosity. Nevertheless the complete mechanism of the oil influence on the mortar mechanical performance is not clear yet.

5. Conclusions

The performance of linseed oil used as admixture in lime mortar was thoroughly tested. The research was motivated by the knowledge that the linseed oil was used as admixture prior the modern synthetic admixtures were invented and introduced in building practice. The linseed oil acts as plasticizer of lime mortar, reducing its yield stress. It also raises the air entrapment of the mortar, what contributes to very good frost resistance of mortar expressed in terms of flexural index of frost resistance. The oil, or products of its polymerization in alkaline (lime) environment, is working as water-repealing agent in the mortar. On the other hand, this property is reducing the rate of carbonation. The presence of oil is also reducing the strength of mortar, but not just because of the lower carbonation, but because of more complex changes of mortars microstructure. The linseed oil dosage 1% of lime mass can be considered as the optimum with respect to the frost resistance. The results might be of importance for preparation of renovation mortars or mortars which are intended to be more sustainable due to absence of synthetic admixtures.

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