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SOME PHYSICAL PROPERTIES OF LIGHTWEIGHT MORTARS WITH EXPANDED VERMICULITE

Abstract

Expanded vermiculite was added to the cement-lime mortar at 10%, 20% and 30%, which reduced the density of the hardened mortar (HM) without expanded vermiculite (EV) by an average of 8,3% for the HM with 10% EV, 14,7% for the HM with 20% EV and 24,2% for the HM with 30% EV. Compressive and flexural strength and static modulus of elasticity were investigated and the relationships between these properties were determined. The effect of the added amount of EV on: total porosity, air void content and capillary suction of HM according to SIA 262, Appendix A; capillary water absorption; gas permeability and air permeability; water permeability and internal freeze/thaw resistance up to 200 cycles was investigated and determined.

Keywords: lightweight cement-lime mortar, expanded vermiculite, physical properties of mortar

НЕКА ФИЗИЧКА СВОЈСТВА ЛАКИХ МАЛТЕРА СА ЕКСПАНДИРАНИМ ВЕРМИКУЛИТОМ

Сажетак

У цементно-кречни малтер је додат експандирани вермикулит са 10%, 20% и 30%, чиме је смањена густина очврслог малтера (ОМ) без експандираног вермикулита (ЕВ) у просјеку за 8,3% за ОМ са 10 % ЕВ, 14,7% за ОМ са 20% ЕВ и 24,2% за ОМ са 30% ЕВ. Истражена је чврстоћа на притисак и савијање и статички модул еластичности и утврђени су односи између ових својстава. Утицај додате количине ЕВ на: укупну порозност, садржај ваздушних шупљина и капиларно усисавање ОМ према СИА 262, Додатак А; капиларна апсорпција воде; пропустљивост гаса и пропустљивост ваздуха; испитана је и одређена водопропусност и унутрашња отпорност на смрзавање/одмрзавање до 200 циклуса.

Кључне ријечи: лаки цементно-кречни малтер, експандирани вермикулит, физичка својства

1. INTRODUCTION

In the framework of the project "Study of the production, placeability and characteristics of final filling and sealing mortars for the N2d disposal container" [1-4], investigations were carried out, the results of which served as a basis to determine the optimal mix proportion of the filling mortar and to optimize the technology for the preparation and placing of the mortars in the disposal container for low and intermediate level radioactive wastes.

The starting point are the results of the tests of the filling and sealing mortar developed in the framework of the project "Development of a prototype and certification of a disposal container for the disposal of low and intermediate level radioactive waste (LILW)" [5]. Based on the results of the gas permeability tests [6, 7], it was concluded that the gas permeability of this filling mortar is too low. The compressive strength of the mortar at 28 days is also considered to be too high (85,0 MPa on average).

Based on the findings of several foreign researchers, the use of cement-lime mortar would be preferable, as the presence of lime, which has a high pH, increases the binding capacity of radionuclides. The presence of lime in the filling mortar is also important to protect the metal drums from corrosion, as lime provides a sufficiently high alkalinity despite the release of CO_2 released by the anaerobic degradation of the stored (mainly organic) LILW. Cement-lime mortars show increased porosity, which is favorable in terms of increased gas permeability, but decreased compressive strength. Based on the calculation models of the container, the strength class of the filling mortar is sufficient C12/15.

Increased porosity and thus increased gas permeability of the aggregate mortar, as well as its ability to accumulate more gas, would be further achieved by the addition of porous (light) aggregate. The three types of lightweight aggregates mainly considered were: expanded clay, perlite and expanded vermiculite. As the use of perlite is suspected to be problematic in the long term [8], it was excluded from the study. In the following study, we consider expanded vermiculite, which has already been used at Nuclear Power Plant Krško for waste encapsulation.

2. SELECTION OF BASIC MATERIALS FOR THE PREPARATION OF MORTARS

The selection of the base materials was based on data and information from the literature, the container testing programme [9], our own experience from investigations of cement-lime mortars and the requirements of the project [10].

2.1. CEMENT

We used pure cement CEM I 42.5 N - SR 0, manufactured by Salonit Anhovo. This type of cement was chosen because it is more coarsely ground and therefore slower in reactions compared to CEM I 52,5 R and CEM I 42,5 N. This is important from the point of view of lowering the temperature of the mortar due to the hydration of the cement because the filling mortar must fill at least 2,6 m^3 in the container. Additionally, this cement ensures the sulphate resistance of the mortar, in case of aggression that could occur due to the possible formation of an aggressive medium during the degradation of the waste material.

2.2. LIME

The lime used was hydrated lime produced by InterCal Slovenija, Zagorje ob Savi. It is calcium hydroxide - $Ca(OH)_2$, containing CaO > 92%, MgO < 1,5%, CO_2 < 4%, SO_3 < 0,5%.

2.3. SUPERPLACTICIZER

In order to reduce the amount of water in the cement-lime mortar (to lower the w/c ratio and to reduce subsequent water extraction - bleeding), polycarboxylate-based superplasticizers were used.

2.4. CRUSHED LIMESTONE AGGREGATE AND STONE FLOUR

We used crushed limestone aggregate and, as part of preliminary trial mixing, stone flour from the Calcit Kamnik separation plant. The justification for the choice of limestone aggregate was given in the project "Study on the production, workability and characteristics of ready-mixed concrete for the secondary reinforced concrete lining of the silo of the LILW repository" [11]. In preliminary laboratory investigations, we looked for the optimum type of stone flour, particularly with regard to workability and bleeding of fresh mortar. The stone flours varied according to their granulometric composition: 0-120 μm (CalPlex VP), 0-70 μm (CalPlex 15) and 0-17,5 μm (CalPlex 5).

2.5. EXPANDED VERMICULITE

As mentioned in the introduction, based on the assessment of the long-term durability of lightweight aggregates in cement-lime mortar, we selected expanded vermiculite as potentially the most suitable. Expanded vermiculite was also used at Nuclear Power Plant Krško to encapsulate waste in drums. This use of expanded vermiculite has been eliminated because the vermiculite mixture occupies a large volume, so the amount of waste material in the barrel has been reduced. The increase in volume of the expanded vermiculite mortar shall not affect its use for filling the space between the drums (TTC) in the container, if such mortar fulfils all other criteria.

Since vermiculite contains 38 to 46 % w/w SiO_2, the question immediately arises whether an alkali-silicate reaction (ASR) can occur in the mortar. In the paper [8], the results of investigations show that there is no ASR potential in a cementitious composite with expanded vermiculite.

A similar conclusion was reached in the IRMA laboratory, where the potential alkali-silicate reactivity of expanded vermiculite was determined according to ASTM C1260-05a. The fractions of expanded vermiculite used were EXV0 (0,25-1mm), EXV1 (0,5-2.5mm) and EXV2 (1-4mm), produced by Vermit Group, Krško. Three combinations were investigated, varying the binder: (1) CEM I 52,5 R-SR from the Lafarge cement plant in Mannersdorf (Austria), (2) CEM I 42,5 N - SR 0 from the Salonit Anhovo cement plant, and (3) CEM I 42,5 N - SR 0 from the Salonit Anhovo cement plant and GIRK Kalun lime (lime/cement ratio = 1/2,65). For all three combinations, we measured practically no expansion after 16 days or 14 days of exposure to an alkaline environment.

3. PRELIMINARY INVESTIGATIONS - FINDING THE OPTIMUM MORTAR MIX PROPORTION

Preliminary investigations were carried out to find the optimal mix proportion of the filling mortar (FM) with respect to its rheological properties: workability (measured by the grout spreading method according to SIST EN 445:2008, section 4.3.2), segregability and bleeding, which were assessed visually.

To measure the spread, we use a cylinder (plastic tube) with an inner diameter of 39 mm and a height of 60 mm, standing on a smooth (glass) plate. Pour the mortar up to the top of the cylinder. After 30 s from the rising of the cylinder, measure the diameter of the spread in two mutually perpendicular directions (Figure 1).

Figure 1. Measurement of the diameter of the mortar spread.

The mortar was placed in 40 × 40 × 160 mm prism moulds, which were then cured according to SIST EN 1015-11:2020, Table 1: the day after preparation, the prisms were placed in polyethylene bags and stored for 7 days; after 7 days, they were removed from the bags and stored until the test in a chamber at 20°C (+3°C/-2°C) and 65%±5% relative humidity; when the mortar was tested at 3 and 7 days of age, the prisms were removed from the bags before the test.

Fresh mortar is considered suitable for filling disposal containers if the average spread according to SIST EN 445:2008, clause 4.3.2 is $PR_{per} \geq 120$ mm, with no segregation and bleeding observed by visual assessment.

Based on the calculation models carried out by the designer of the container, the compressive strength of the mortar is sufficient to meet the strength class C12/15.

The following tests were carried out to assess the applicability of the mortar:

- **in a fresh state:**
  - spread according to SIST EN 445:2008, clause 4.3.2;
  - visual assessment of segregation and bleeding;
- **in a hardened state:**
  - compressive strength according to SIST EN 1015-11:2020 converted to the compressive strength of a cube with an edge of 15 cm.

In addition, the following tests were carried out on selected mortars:

- **in a fresh state:**
  - air content according to SIST EN 1015-7:1999, pressure method;
  - water-cement ratio according to SIST 1026:2016, Appendix NC;
  - density according to SIST EN 1015-6:1999/A1:2007;
  - air and mortar temperature;
- **in a hardened state:**
  - density according to SIST EN 1015-10:2001/A1:2007.

Table 1 gives the mix proportions of all the filling mortars (FMs) from the preliminary investigations, together with an assessment of the applicability of each FM in relation to the results of the workability (spread) test of the fresh FMs, the visual assessment of segregation and bleeding of the fresh FMs, and the results of the compressive strength test of the hardened FMs.

Table 1. Mix proportions of all FMs from the preliminary investigations and assessment of applicability

set of FM mixtures	filling mortar designation	binding component		stone flour (μm)			superplasticizer				expanded vermiculite (mm)			crushed limestone (mm)		water	appropriate workability	segregation	bleeding	strength class C12/15	appropriate for application
		cement (% m/m)	lime (% m/m)	0-120	0-70	0-17,5	RC277	RC897	RC187	RC687	0,25-1	0,5-2,5	1-4	0-2	0-4						
1	»NIREX«, CalPlex VP	62	38		-	-	-	-	-	-	-	-	-	-	-		yes	no	yes	no	no
	»NIREX«, CalPlex 15	62	38	-		-	-	-	-	-	-	-	-	-	-		yes	no	yes	no	no
	»NIREX«, CalPlex 5	62	38	-	-		-	-	-	-	-	-	-	-	-		yes	no	yes	no	no
2	»NIREX«-mod, CalPlex VP	62	38		-	-		-	-	-	-	-	-	-	-		yes	no	yes	yes	no
	»NIREX«-mod, CalPlex 15	62	38	-		-		-	-	-	-	-	-	-	-		yes	no	yes	yes	no
	»NIREX«-mod, CalPlex 5	62	38	-	-			-	-	-	-	-	-	-	-		yes	no	yes	yes	no
3	FM-VER-1	62	38		-	-		-	-	-				-	-		no	no	no	yes	no
	FM-VER-2	62	38		-	-		-	-	-				-	-		no	no	no	yes	no
	FM-VER-2a	62	38		-	-		-	-	-				-	-		yes	no	no	no	no
	FM-VER-2b	62	38		-	-		-	-	-				-	-		no	no	no	no	no
	FM-VER-2c	62	38		-	-		-	-	-				-	-		yes	no	no	no	no
	FM-VER-2d	62	38		-	-		-	-	-				-	-		yes	no	no	no	no
	FM-VER-2e	62	38		-	-		-	-	-				-	-		no	no	no	no	no
	FM-VER-2f	62	38		-	-		-	-	-				-	-		no	no	no	no	no
4	FM-VER-3	62	38		-	-		-	-	-				-			yes	no	no	no	no
	FM-VER-3a	62	38		-	-		-	-	-				-			no	no	no	no	no
	FM-VER-4	62	38		-	-	-		-	-				-			no	no	no	no	no
	FM-VER-4a	62	38		-	-	-		-	-				-			yes	no	no	no	no
5	FM-VER-5	62	38	-	-	-	-		-	-		-	-	-			no	no	no	no	no
	FM-VER-5a	62	38	-	-	-	-		-	-		-	-	-			no	no	no	no	no
	FM-VER-5b	62	38	-	-	-	-		-	-		-	-	-			yes	yes	-	-	no
	FM-VER-5c	62	38	-	-	-		-	-	-		-	-	-			yes	yes	no	no	no
	FM-VER-6	62	38	-	-	-		-	-	-		-	-		-		yes	no	no	yes	yes
6	FM-VER-E-1-187	62	38	-	-	-	-	-		-	-	-	-		-		yes	yes	yes	yes	no
	FM-VER-E-1-897	62	38	-	-	-	-		-	-	-	-	-		-		no	no	no	yes	no
	FM-VER-E-1-687	62	38	-	-	-	-	-	-		-	-	-		-		yes	no	no	yes	yes
7	FM-0-EV	62	38	-	-	-	-	-	-		-	-	-		-		yes	no	no	yes	yes
	FM-10-EV	62	38	-	-	-	-	-	-			-	-		-		yes	no	no	yes	yes
	FM-20-EV	62	38	-	-	-	-	-	-			-	-		-		yes	no	no	yes	yes
	FM-30-EV	62	38	-	-	-	-	-	-			-	-		-		yes	no	no	no	no

The mix proportions of FM set 7 were accepted as default for further laboratory tests.

4. LABORATORY INVESTIGATIONS OF SELECTED MORTARS

In the four selected FM mixtures (from set 7 in Table 1, designated FM-0-EV, FM-10-EV, FM-20-EV and FM-30-EV), we vary the volume fractions of the expanded vermiculite EXV0 (0,25-1 mm): 0, 10, 20 and 30 vol.%. The proportions of crushed limestone aggregate 0-2 mm shall be reduced accordingly. The proportions of cement CEM I 42,5 N SR0, lime and superplasticizer RC 687 remain the same. Water was added to each FM mix in such a quantity as to achieve a suitable workability of the fresh FM. Therefore, as the proportion of expanded vermiculite (EV) increased, more water had to be added, increasing the total water-cement ratio $(w/c)_{tot}$. In the mix proportions, we assume that the EV and the aggregate do not contain water. Therefore, we have also used dry EV and aggregate in the preparation of the FM.

4.1. INVESTIGATIONS OF FRESH MORTARS

4.1.1. TESTS ON FRESH MORTARS IN THE PREPARATION OF TEST SPECIMENS

The following tests and measurements were carried out in the preparation of the test specimens:

- air and mortar temperature;
- spread according to SIST EN 445:2008, clause 4.3.2;
- air content according to SIST EN 1015-7:1999 , pressure method;
- water-cement ratio $(w/c)_{tot}$ according to SIST 1026:2016, Appendix NC;
- density according to SIST EN 12350-6:2019.

The results of the creep measurements show that the workability of all FM was good immediately after mixing. The average values are 154 mm for FM-0-EV and FM-10-EV, 156 mm for FM-20-EV and 151 mm for FM-30-EV. After 30 minutes, workability decreases, but this varies considerably from mixture to mixture, even for mixtures of the same type of FM. Several factors influence the change in workability: the temperature of the air and mortar; the degree of humidity of the EV and aggregate, which affects their water absorption; and the precision of the measurements.

The air content increases slightly on average with increasing EV content for slightly higher dispersion results.

As EV increases, the density of fresh PM decreases (Figure 2).

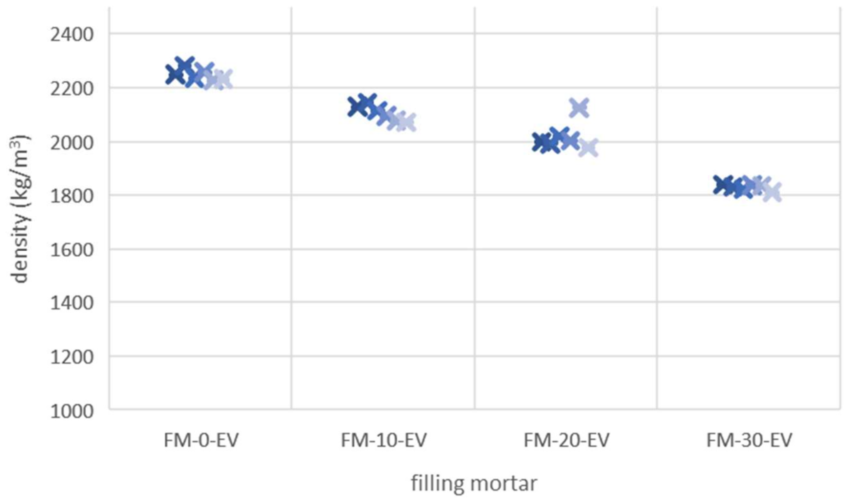

Figure 2. Results of the measurements of the density of fresh FM.

As the amount of EV was increased, more water had to be added, so for the same amount of cement in all FM mixtures, the total water-cement ratio $(w/c)_{tot}$ also increased.

4.1.2. INITIAL SETTING TIME OF FM

The penetration resistance is measured in the initial setting time test of FM. Measurements were carried out using a CT-421A concrete and mortar penetrometer according to the method given in ASTM C403/C403M -23. The mortar starts to set when it reaches a penetration resistance of 3,448 kPa (500 psi). As the amount of EV increased, more water was added and for the same amount of cement, the total water-cement ratio increases, so as the amount of EV increases, the FM initial setting time increases (Figure 3).

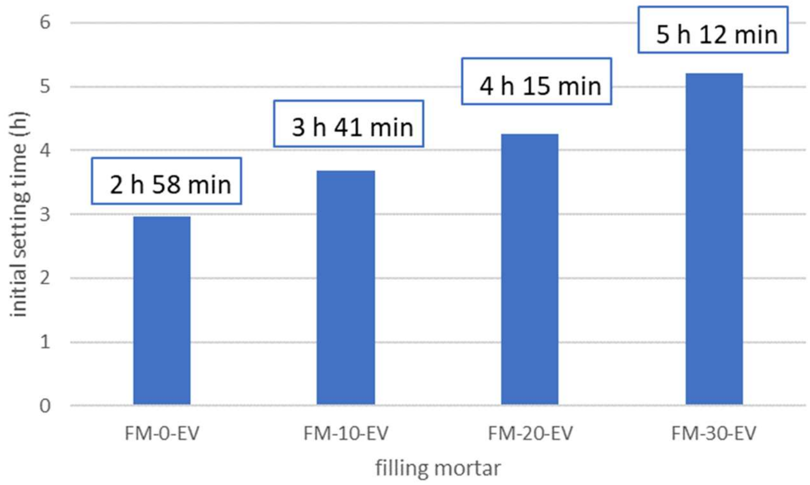

Figure 3. Initial setting time as a function of increasing amount of EV in the FM.

4.1.3. ADIABATIC THERMAL CHARACTERISTICS OF FM

The adiabatic thermal characteristics of the FM were measured in an ATC chamber (adiabatic calorimeter). The results of the measurements are given for each FM in Table 2: initial temperature T_i; maximum measured temperature $T_{max,meas}$; time at which the maximum temperature measured $t_{Tmax,meas}$; corrected maximum temperature $T_{max,corr}$; temperature increment ΔT.

Table 2. Initial temperature T_i; maximum measured temperature $T_{max,meas}$; time at which the maximum temperature measured $t_{Tmax,meas}$; corrected maximum temperature $T_{max,corr}$; temperature increment ΔT

parameters	FM-0-EV	FM-10-EV	FM-20-EV	FM-30-EV
Ti (°C)	21,57	26,49	19,58	21,62
$T_{max,meas.}$ (°C)	65,54	66,59	57,81	54,33
$t_{Tmax.meas}$ (day, h, min)	1d,14h,15m	1d,16h,05m	2d,2h,45m	2d,2u,15m
$T_{max,corr}$ (°C)	72,69	75,19	64,3	60,96
increase ΔT (°C)	51,12	48,7	44,7	39,34

As the amount of expanded vermiculite increases, the insulating capacity of the FM increases and therefore the temperature increment ΔT decreases.

4.1.4. SHRINKAGE OF FM DUE TO DRYING

FM shrinkage due to drying was measured according to DIN 4227 - Part 1, on 3 prisms of dimensions 10 × 10 × 50 cm, at a relative humidity of 65 (± 5)% and a temperature of +20 (± 4)°C. Additionally, shrinkage was measured on 3 prisms of identical dimensions, wrapped with PVC foil and stored under the same conditions. In this way, we want to simulate similar conditions to those that will be present in the FM placed in the disposal container.

Shrinkage due to drying of FM without protection is most intense for FM-30-EV and FM-20-EV, and much less intense for FM-0-EV and FM-10-EV (in Figure 4, the four curves below). This shrinkage is due to the water content, which is higher in FM with a higher amount of expanded vermiculite.

The reverse effect is obtained by partially preventing drying by wrapping the test specimens with foil (top four curves in Figure 4). We are talking about partial prevention because the foil is also permeable. In such a test, the evaporation of water from the FM is very slow and the intensity of shrinkage is correspondingly low. Unbound water remaining in the voids of the expanded vermiculite causes first a slight expansion of the grains. As this water slowly evaporates, shrinkage occurs, which is very small in the case of FM with a larger amount of expanded vermiculite, as can be seen in Figure 4 - the top two curves. This figure shows that PM-30-EV has not shrunk from its initial state after 188 days.

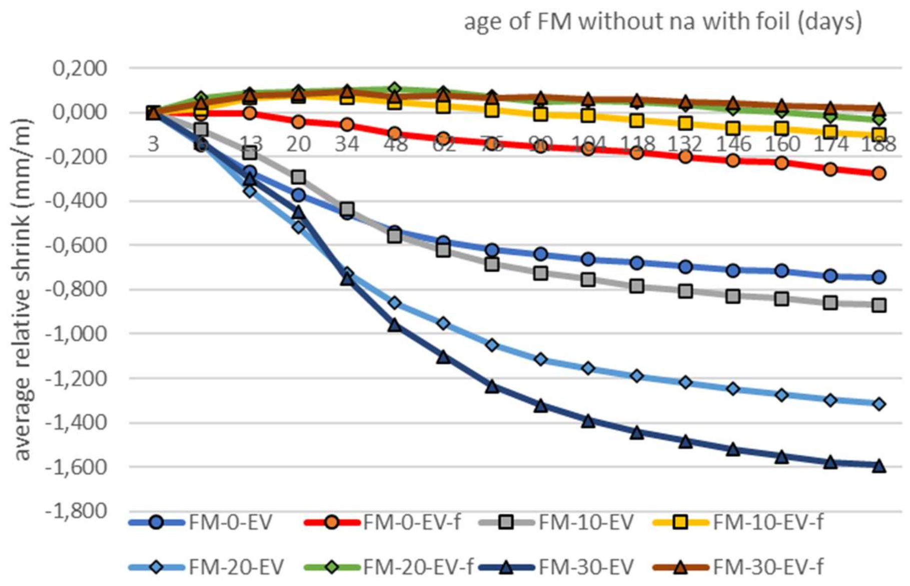

Figure 4. Shrinkage due to drying of FM without and with protection (with foil) as a function of the age of the FM.

4.2. INVESTIGATIONS OF HARDENED MORTARS

4.2.1. COMPRESSIVE AND FLEXURAL TENSILE STRENGTH AND DENSITY OF FM PRISMS 4 × 4 × 16 CM

The compressive and flexural tensile strengths and the density of FM were determined on 4 × 4 × 16 cm prisms according to SIST EN 1015-11:2020 and SIST EN 1015-10:2001/A1:2007 at 3, 7, 28, 56, 90 and 180 days of age.

Density

The average values of the results of the density tests as a function of age of the FM are given in Figure 5.

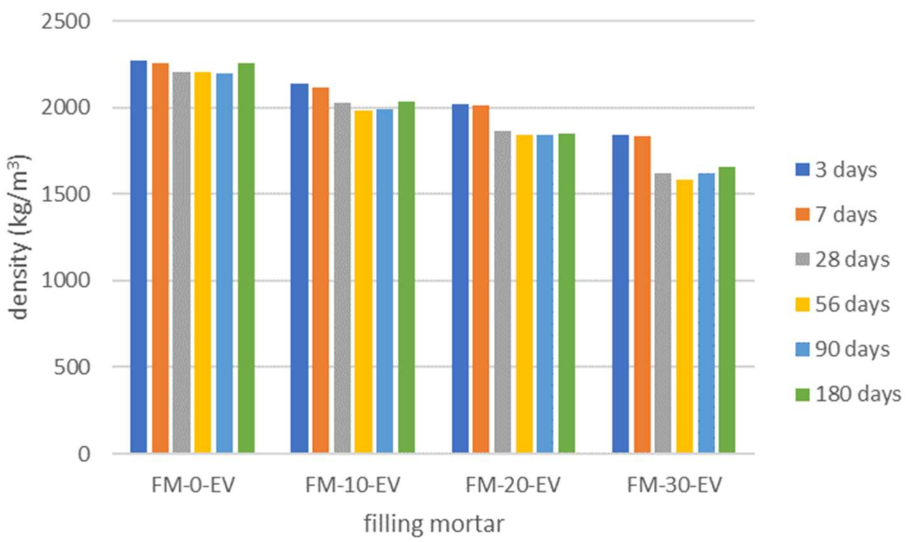

Figure 5. Average values of density test results as a function of FM age.

The density of FM decreases as the amount of expanded vermiculite increases. Compared to the density of FM-0-EV (ρ_0), the density of FM-10-EV (ρ_{10}) decreases on average by 8,3%, ρ_{20} (FM-20-EV) by 14,7% and ρ_{30} (FM-30-EV) by 24,2%.
After 7 days, the foil was unwrapped according to the standard and the test specimens were cured in a chamber at 20°C (+3°C/-2°C) and 65%±5% relative humidity until the test. This change in cure resulted in a decrease in the densities of all FM.

Compressive strength

The average values of the compressive strength test results, as a function of FM age, are given in Figure 6.

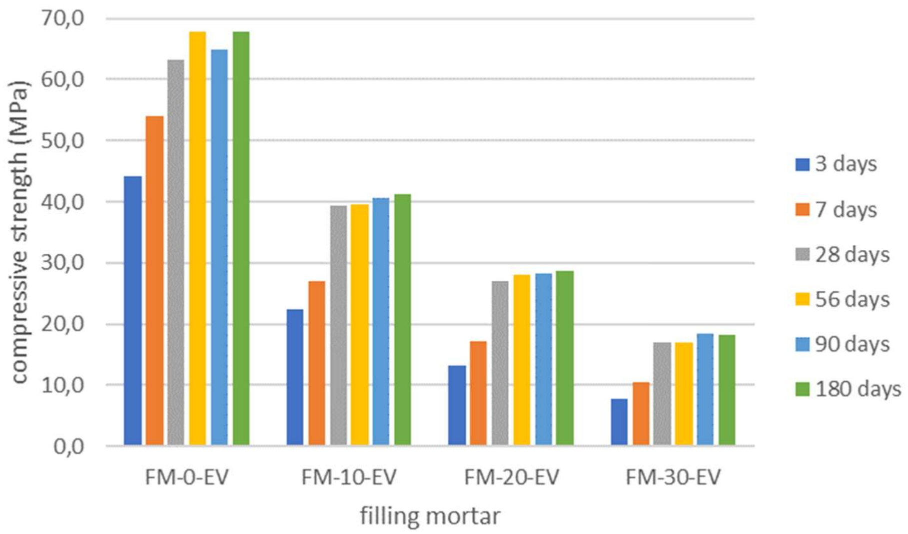

Figure 6. Average values of compressive strength test results as a function of FM age.

The compressive strength of all FM increases with age. Compared to the compressive strength of FM-0-EV (f_{c0}), the compressive strength of FM-10-EV (f_{c10}) decreases on average by 42,5%, f_{c20} (FM-20-EV) by 61,4% and f_{c30} (FM-30-EV) by 76,0%.
Up to the age of FM 7 days, FM with a higher amount of expanded vermiculite also contained a higher amount of water, resulting in lower compressive strengths. As mentioned above, after 7 days, the foil was unwrapped in accordance with the standard and the test specimens were cured until the test in a chamber with a temperature of 20°C (+3°C/-2°C) and a relative humidity of 65%±5%. This change in cure reduced the amount of water in the FM, thereby significantly increasing the compressive strength at FM age 28 days compared to the compressive strength at FM age 7 days (Figure 6). This relative increase is more pronounced in FM with expanded vermiculite (31,0% in FM-10-EV, 36,2% in FM-20-EV and 37,9% in FM-30-EV) compared to FM-0-EV, where it is only 14,5%.

Flexural tensile strength

The average values of the results of the flexural tensile strength tests, as a function of the age of the FM, are given in Figure 7. Compared to the flexural tensile strength of FM-0-EV (f_{f0}), the flexural tensile strength of FM-10-EV (f_{f10}) decreases on average by 18,4%, f_{f20} (FM-20-EV) by 356% and f_{f30} (FM-30-EV) by 47,0%.
The change in curing reduced the amount of water in the FM, thereby significantly increasing the flexural tensile strength at 28 days FM compared to the flexural tensile strength at 7 days FM (Figure 7). This relative increase is much more pronounced in FM with expanded vermiculite (18,7% in FM-10-EV, 30,8% in FM-20-EV and 37,7% in FM-30-EV) compared to FM-0-EV, where it is only 1,1%.

Relationship between flexural tensile strength and compressive strength

Figure 7 shows that for all FMs, the average flexural tensile strengths increase up to an FM age of 56 days and then decrease. However, Figure 6 shows that the average compressive strengths of all FMs increase up to 180 days of age. Figure 8 shows the progress of the flexural tensile strengths with respect to the increase in compressive strengths for each FM.

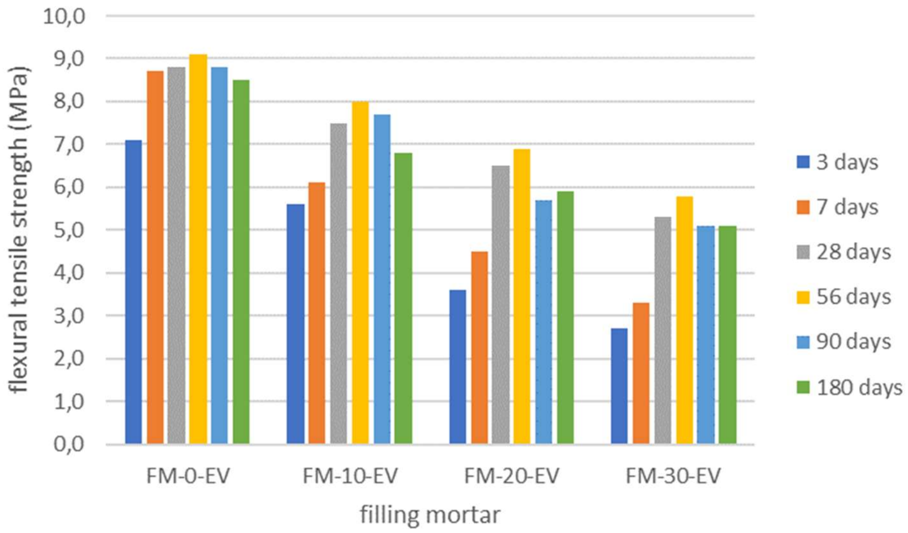

Figure 7. Mean values of flexural tensile strength test results as a function of FM age.

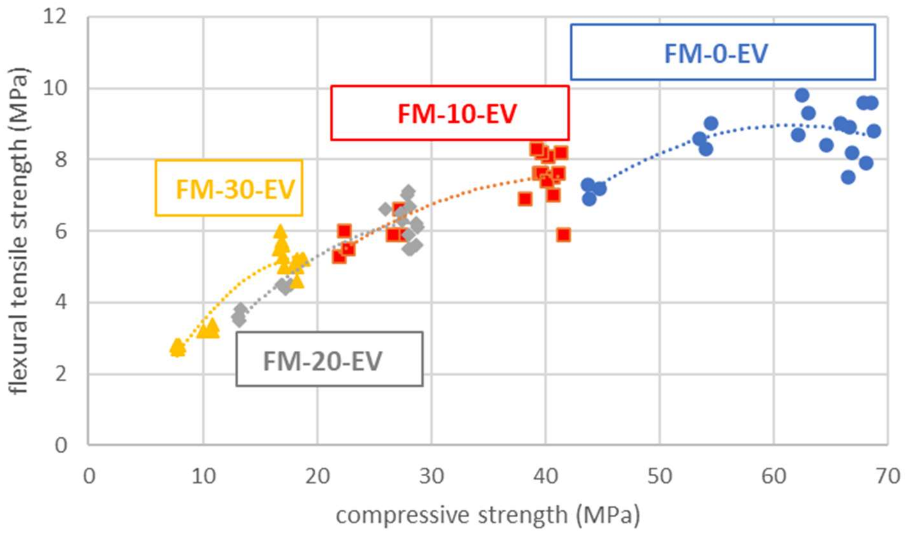

Figure 8. Progress of flexural tensile strengths versus increasing compressive strengths of FM.

Figure 8 shows that there is a trend towards decreasing flexural tensile strengths at higher compressive strengths. This is most evident in FM-0-EV, but much less so in all other FMs with expanded vermiculite. This means that FM becomes more brittle at higher compressive strengths, especially for FM-0-EV, which achieves much higher compressive strengths compared to the compressive strengths of FM with expanded vermiculite.

From the results obtained for the compressive and flexural tensile strengths of the FM, we can also calculate the ratio of the flexural tensile strength f_f to the compressive strength f_c: $f_f/f_c \times 100$ (Figure 9), which increases as the amount of expanded vermiculite in the FM increases.

4.2.2. COMPRESSIVE STRENGTH OF A CUBE WITH A 15 CM EDGE AND STATIC MODULUS OF ELASTICITY FM

The compressive strength was determined on a cube with an edge of 15 cm according to SIST EN 12390-3:2019 and the static modulus of elasticity on a prism of 10 × 10 × 40 cm according to DIN 1048, Part 5, section 7.5 at FM ages 3, 7, 28, 56, 90 and 180 days.

Compressive strength

The average values of the compressive strength test results as a function of FM age are given in Figure 10. The compressive strength of all FM increases with age. Compared to the compressive strength of FM-0-EV (f_{c0}), the compressive strength of FM-10-EV (f_{c10}) decreases on average by 48,9%, f_{c20} (FM-20-EV) by 65,3% and f_{c30} (FM-30-EV) by 78,4%.

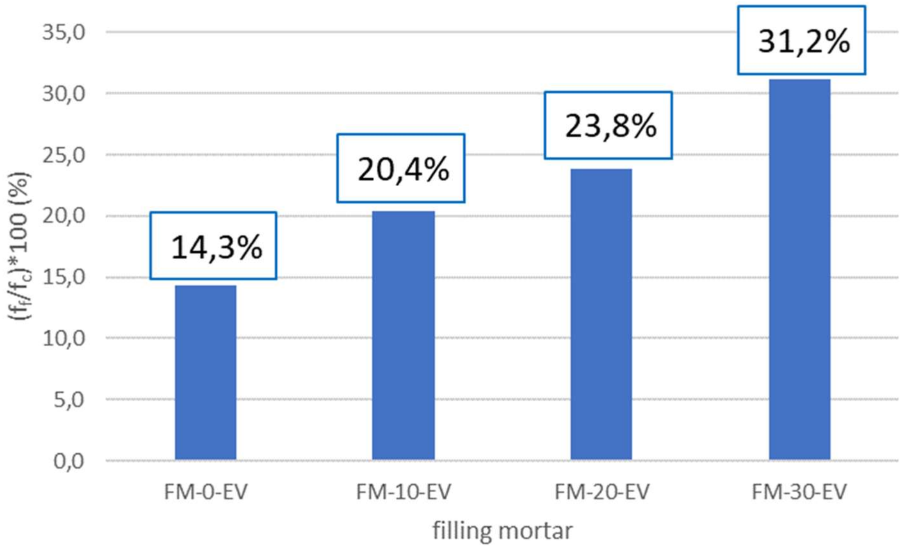

Figure 9. Proportions of the flexural tensile strength f_f with respect to the compressive strength f_c FM.

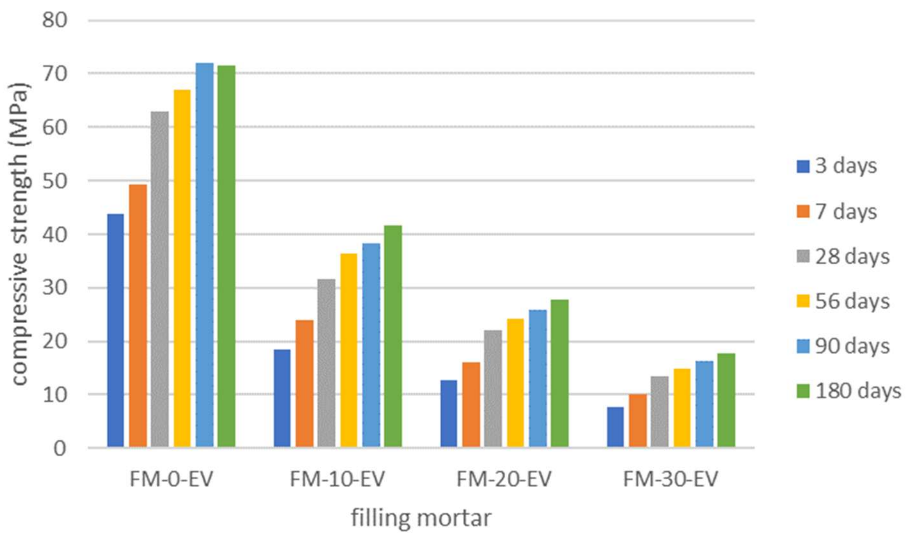

Figure 10. Average values of compressive strength test results as a function of FM age.

The effect of curing according to SIST EN 1015-11:2020, Table 1 on the increment of the compressive strength of FM cubes with a 15 cm edge is lower compared to the increment of the strength of FM cubes with a 4 cm edge (or 4 × 4 × 16 cm prism halves). The relative increase in compressive strength at FM 28 days compared to the compressive strength at FM 7 days is only slightly higher for FM with expanded vermiculite (24,6% for FM-10-EV, 27,6% for FM-20-EV, 24,6% for FM-30-EV) compared to FM-0-EV, where it is 20,2%.

In addition to FM-0-EV ($f_{cm,K15,28}$ = 62,9 MPa), FM-10-EV ($f_{cm,K15,28}$ = 31,7 MPa) also meets the criterion of the required strength class C12/15 at FM 28 days. The acceptance criterion for the initial compressive strength tests at FM 28 days is:

$$f_{cm,K15,28} \geq f_{ck} + 12,0 \text{ MPa} = 15,0 \text{ MPa} + 12,0 \text{ MPa} = 27,0 \text{ MPa}.$$

Ratio between the compressive strength of an FM cube with an edge of 15 cm and the compressive strength of an FM cube with an edge of 4 cm (or a prism half of 4 × 4 × 16 cm)

The correlations between the average compressive strength test results of an FM cube with 15 cm edge $f_{c,K15}$ and the average compressive strength test results of an FM cube with 4 cm edge $f_{c,K4}$ are given for each FM in Figure 11.

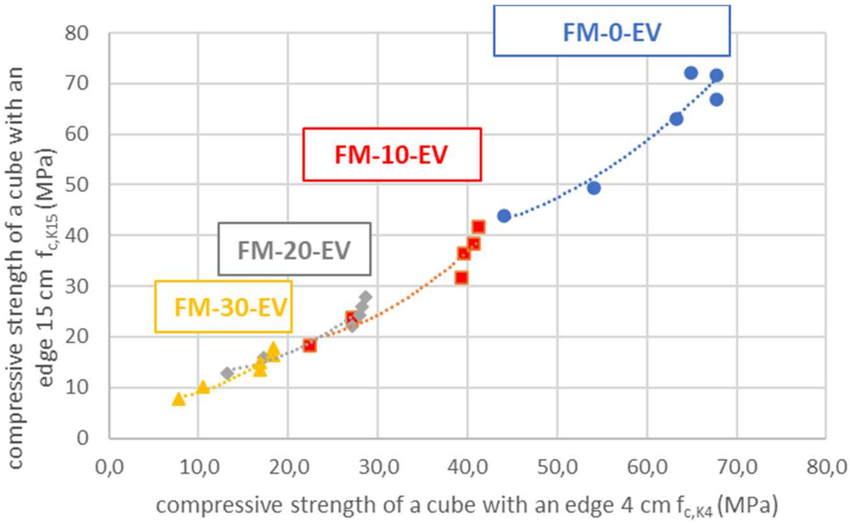

Figure 11. Correlation between $f_{c,K15}$ and $f_{c,K4}$ of individual PMs.

From the results of the compressive strength tests of the FM cubes with a 15 cm edge and the FM cubes with a 4 cm edge, calculate for each FM the ratio between $f_{c,K15}$ and $f_{c,K4}$:

- $f_{c,K15}/f_{c,K4}$ = 1,01 for FM-0-EV,
- $f_{c,K15}/f_{c,K4}$ = 0,90 for FM-10-EV,
- $f_{c,K15}/f_{c,K4}$ = 0,91 for FM-20-EV,
- $f_{c,K15}/f_{c,K4}$ = 0,92 for FM-30-EV.

Taking all the results of the FM compressive strength tests together gives the following average ratio:

- $f_{c,K15}/f_{c,K4}$ = 0,93 for all PMs together.

Static modulus of elasticity

The average values of the results of the static modulus of elasticity tests, as a function of the age of the FM, are given in Figure 12.

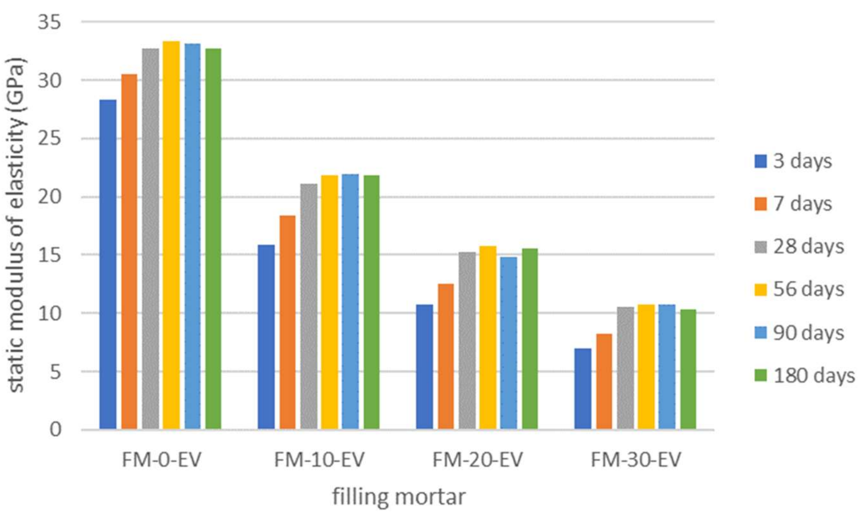

Figure 12. Average values of static modulus of elasticity test results vs. age of FM.

The elastic modulus of elasticity increases in all FM until the age of 28 days, after which the increase is moderate or almost stops until the age of 180 days. As compressive strength increases with FM age up to 180 days (Figure 10), this is also evident from the correlations between the average compressive strength and static modulus of elasticity test results. For intercomparison, all correlations are given together in Figure 13.

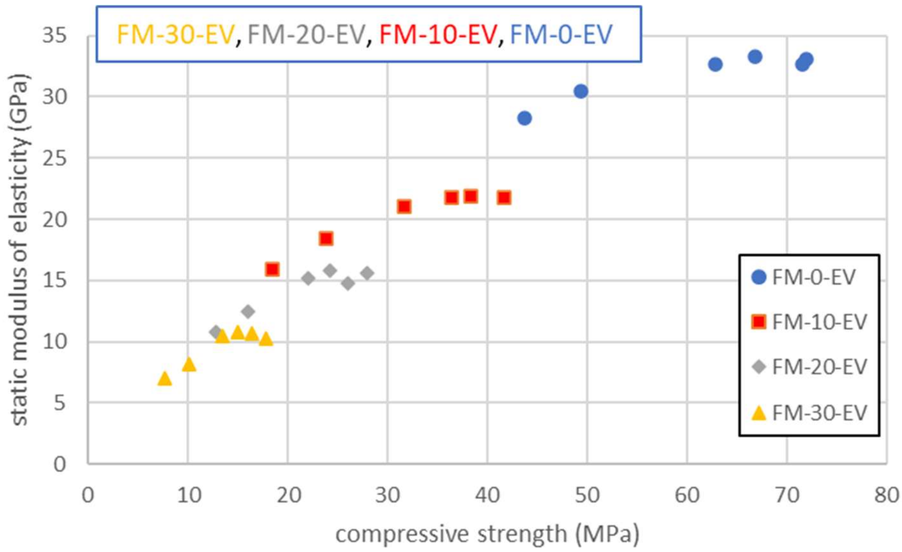

Figure 13. All correlations between compressive strength and static modulus of elasticity of FM together.

Compared to E_{stat0} (FM-0-EV), E_{stat10} (FM-10-EV) is on average 36,8% smaller, E_{stat20} (FM-20-EV) 55,8% smaller and E_{stat30} (FM-30-EV) 70,0% smaller.

4.2.3. TOTAL POROSITY, AIR PORE CONTENT AND CAPILLARY SUCTION OF FM ACCORDING TO SIA 262, APPENDIX A

Total porosity, air pore content and capillary suction of FM were determined according to SIA 262, Appendix A. The results are given in Table 3, together with a reference mortar from abroad.

Table 3. Total porosity, air pore content and capillary suction of FM.

filling mortar	average values		
	total porosity n (%)	air pore content L_p (%)	capillary suction q_w (g/(m^{2}h))
FM-0-EV	18,8	0,3	4,8
FM-10-EV	26,8	2,3	6,5
FM-20-EV	34,6	5,5	9,3
FM-30-EV	44,2	10,7	10,7
reference mortar	26,9	3,2	20,7

The total porosity and air pore content are given together in Figure 14, and the capillary suction is given in Figure 15.

As can be seen from Figure 14, the total porosity n and air pore content L_p of PM-10-EV are very similar to n and L_p of the reference filling mortar. However, it can be seen from Figure 15 that the capillary suction q_w of the FM-10-EV is much lower compared to the q_w of the reference FM. These results indicate the suitability of the FM-10-EV for its intended use.

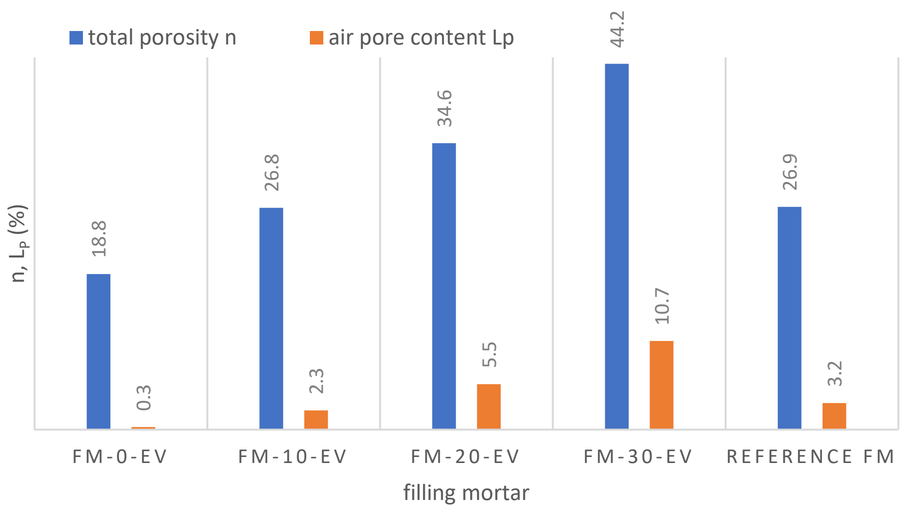

Figure 14. Total porosity n and air void content Lp.

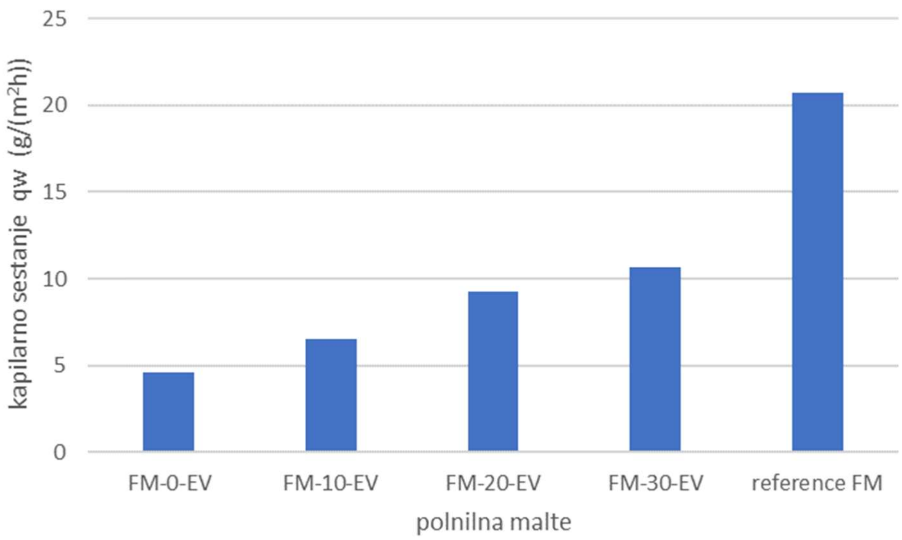

Figure 15. Capillary suction q_w.

4.2.4. CAPILLARY WATER-ABSORPTION

The capillary water-absorption of the FMs was investigated according to SIST EN 13057:2002. The average values of the sorption coefficients at FM ages 28, 90 and 180 days are given in Table 4 and Figure 16.

Table 4. Average sorption coefficient of FM.

age of FM (days)	average sorption coefficient (kg/m² h^0,5)			
	FM-0-EV	FM-10-EV	FM-20-EV	FM-30-EV
28	0,72	1,31	1,99	2,36
90	0,56	1,12	1,98	2,31
180	0,67	1,18	1,99	2,34

Average sorption coefficients were determined for all FMs, which are less than the maximum permissible value of 2,50 $kg/m^2h^{0.5}$. The coefficient of sorption decreases only slightly with the age of FM. For FM-0-EV and FM-10-EV, the sorption coefficients are much lower compared to those of FM-20-EV and FM-30-EV.

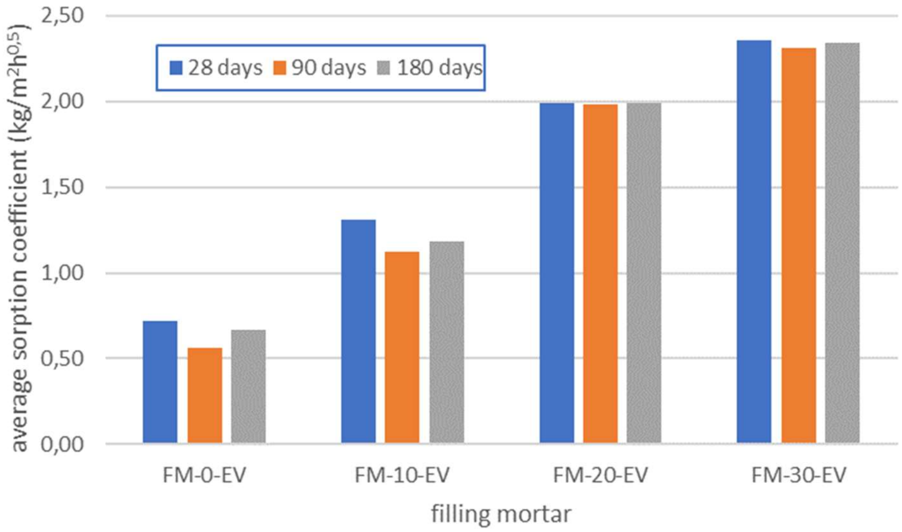

Figure 16. Average sorption coefficient depending on the age of the FM.

4.2.5. GAS PERMEABILITY

The gas permeability test was carried out in the CEMBUREAU permeameter in the laboratory of the University of Liège, Department of Architecture, Geology, Environment and Constructions. The cylinders tested were 150 mm in diameter and 50 mm high, drilled from cubes with a 20 cm edge. The test specimens are dried to a constant mass.

The following average values of intrinsic permeability k_{int} are obtained:

- $k_{int,avrg} = 2,80 \times 10^{-16}$ m^2 for FM-0-EV;
- $k_{int,avrg} = 4,21 \times 10^{-16}$ m^2 for FM-10-EV;
- $k_{int,avrg} = 6,21 \times 10^{-16}$ m^2 for FM-20-EV;
- $k_{int,avrg} = 1,18 \times 10^{-15}$ m^2 for FM-30-EV.

For the filling mortar used to fill the prototype containers at the Pomgrad ABI plant in Lipovci, we determined a much lower average intrinsic permeability:

- $k_{int,avrg} = 2,67 \times 10^{-17}$ m^2.

4.2.6. AIR PERMEABILITY

The air permeability of FM was measured in the ZAG laboratory on cubes with a 20 cm edge according to SIA 262/1:2019, Appendix E, at FM ages 28, 50, 90 and 180 days.

The following average values of the air permeability kT of 180-day-old FM are obtained:

- $kT_{avrg} = 0,043 \times 10^{-16}$ m^2 for FM-0-EV;
- $kT_{avrg} = 0,202 \times 10^{-16}$ m^2 for FM-10-EV;
- $kT_{avrg} = 0,525 \times 10^{-16}$ m^2 for FM-20-EV;
- $kT_{avrg} = 1,668 \times 10^{-16}$ m^2 for FM-30-EV.

The measured values show a lower permeability compared to the gas permeability measurements (paragraph 4.2.5) where the test specimens have been dried to a constant mass, which is not the case in the air permeability test.

4.2.7. WATER PERMEABILITY

The FM water permeability test in a triaxial cell at constant hydraulic gradient according to SIST EN ISO 17892-11:2019 was carried out in the ZAG laboratory.

The coefficient of water permeability at room temperature was calculated in the test:

- $k_T = 3,10 \times 10^{-14}$ m/s, for FM-10-EV;
- $k_T = 4,78 \times 10^{-13}$ m/s, for FM-20-EV.

By applying a temperature correction factor for a reference temperature of T_{ref} = 10°C, the water permeability coefficient at T = 10°C was calculated:

- k_{10} = 2,24 × 10^{-14} m/s, for FM-10-EV;
- k_{10} = 3,45 × 10^{-13} m/s, for FM-20-EV.

The results show that increasing the amount of expanded vermiculite in the FM from 10% to 20% increases the water permeability coefficient significantly.

4.2.8. INTRINSIC FREEZE/THAW RESISTANCE (IRFT-200)

The intrinsic freeze/thaw resistance of the FM to 200 freeze/thaw cycles (IRFT-200) was tested according to SIST 1026:2016, Appendix ND, at an FM age of 90 days.

Table 5 gives for each FM the average relative dynamic modulus of elasticity after 200 freeze/thaw cycles P_n and the minimum relative dynamic modulus of elasticity after 200 freeze/thaw cycles P_{min}.

Table 5. P_n and P_{min} after 200 freeze/thaw cycles.

average relative dynamic modulus of elasticity	FM-0-EV	FM-10-EV	FM-20-EV	FM-30-EV
P_n (%)	54	103,3	103,4	104,1
P_{min} (%)	27,7	103,1	102,9	103,0

Only FM-0-EV does not demonstrate intrinsic freeze/thaw resistance up to 200 cycles (IRFT-200) because P_n < 75% and P_{min} < 65%. However, for all FM with expanded vermiculite, the dynamic modulus of elasticity increases after 200 cycles, indicating that no intrinsic freezing/thawing damage has occurred.

5. CONCLUSION

The addition of expanded vermiculite to lime-cement mortar reduces the fresh and hardened density of the lightweight mortar (FM). The reduction in density is in direct proportion to the increase in the amount of expanded vermiculite.

Increasing the amount of expanded vermiculite (EV) added has the effect of prolonging the initial setting time of the FM and reducing the temperature rise (ΔT) due to the hydration of the cement in the FM.

Shrinkage due to drying is most intense in FM with higher amounts of EV (30% and 20%), but much less in FM without EV and with lower amounts of EV (10%). This shrinkage is due to the water content, which is higher in FM with a higher amount of EV. The reversal effect is obtained by partially preventing drying by wrapping the test specimens with foil. In such a test, the evaporation of water from the FM is very slow and the intensity of shrinkage is correspondingly low. Unbound water remaining in the voids of the expanded vermiculite causes first a slight expansion of the grains and then, as this water slowly evaporates, shrinkage occurs, which is very slight in the case of FM with a larger amount of expanded vermiculite.

As the amount of EV increases, the compressive and flexural strengths of lightweight FM decrease. We found a trend towards decreasing flexural tensile strengths at higher compressive strengths. This is most evident for FM without EV, but much less so for all other FM with EV. This means that at higher compressive strengths the FMs become more brittle, especially FM-0-EV, which achieves much higher compressive strengths compared to those of the FMs with EV. From the results obtained for the compressive and flexural tensile strengths of the FM, we also calculated the percentage of the flexural tensile strength f_f with respect to the compressive strength f_c: $f_f/f_c \times 100$, which increases with increasing the amount of expanded vermiculite in the FM.

According to SIA 262, Appendix A, the total porosity n and air void content L_p of FM-10-EV are very similar to the n and L_p of the reference filling mortar. The capillary suction q_w of the FM-10-EV is much lower compared to the q_w of the reference FM. These results indicate the suitability of the FM-10-EV for its intended use.

All the results of the sorption coefficients determined by the capillary water-absorption test are less than the maximum permissible value, with the sorption coefficients of FM-0-EV and FM-10-EV being significantly lower compared to the sorption coefficients of FM-20-EV and FM-30-EV.

The average values of the intrinsic permeability k_{int} determined by the gas permeability test in the CEMBUREAU permeameter increase as the amount of EV in the FM increases and are higher than the k_{int} of the filling mortar used to fill the prototype containers. The measured air permeability values determined according to SIA 262/1:2019, Appendix E show a lower permeability compared to the gas permeability measurements (in the CEMBUREAU permeameter) where the test specimens were dried to a constant mass, which is not a coincidence in the air permeability test.

From the results of the water permeability test of the FM in a triaxial cell at constant hydraulic gradient, it is evident that the increase in the amount of EV in the FM from 10% to 20% results in a significant increase in the water permeability coefficient.

The results of the FM intrinsic freeze/thaw resistance test up to 200 freeze/thaw cycles (IRFT-200) show that only FM-0-EV does not exhibit intrinsic freeze/thaw resistance up to 200 cycles because the average relative dynamic modulus of elasticity is $P_n < 75\%$ and the minimum relative dynamic modulus of elasticity is $P_{min} < 65\%$. However, for all FM with expanded vermiculite, the dynamic modulus of elasticity increases after 200 cycles, indicating that no intrinsic freeze/thaw damage has occurred.

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