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Evaluation of Water Vapor Permeability in Mortars Produced with Crystallizing Additive

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Abstract

The durability of mortar coatings is directly linked to their permeability. Crystallization additives have the potential to modify the pore structure and connectivity within the mortar, thereby improving its permeability. These additives serve the dual function of preventing water ingress while allowing water vapor permeability, which is crucial to promote moisture elimination in mortars. This study aims to analyze the effectiveness of a crystallization additive in enhancing the water vapor permeability of mortars. For this evaluation, samples with proportions of 1:1 and 1:3 (cement: sand, dry materials, by volume, produced by weight) were prepared with the addition of 0%, 1%, and 3% crystallizer based on the cement mass. These proportions were evaluated over time in both the fresh and hardened states, following the Portuguese standard NP EN 1015-19 for determining the water vapor permeability of hardened plastering mortars. Scanning electron microscope (SEM) images were also captured. During the fresh state, the addition of 3% crystallizer impacted the workability of the mortars. However, through rigorous statistical analysis, it was determined that the incorporation of the crystallizing additive in the mortar did not significantly affect its steam permeability. As a result, the additive effectively prevents water from infiltrating and filling the pores of the material. The SEM images depicted the presence of potential crystal formations in the 1:3 mortar mixture containing 3% additive, which underwent wet curing (WC) for 97 days. Moreover, the observed crystallization process facilitated the ability to self-heal a crack, with Energy dispersive spectroscopy (EDS) analysis revealing a predominance of calcium hydroxide particles ($Ca(OH)_2$). This behavior demonstrates the viability of incorporating this particular additive in coating mortars, introducing a novel approach to these construction systems.

Keywords

Mortars, Additives, Water vapor permeability

Introduction

Cementitious materials tend to crack, allowing the ingress of deleterious agents that lead to the manifestation of pathological indications directly linked to the reduction of structural longevity [1]. Furthermore, the porous structure of mortars governs their attributes, including water vapor permeability and capillary absorption. Heightened porosity corresponds to a decrease in mechanical properties, while simultaneously augmenting the diffusion of vapor through the material [2].

Crystallizing additives present themselves as a promising solution for infiltrations that deteriorate coatings, as their primary purpose is to facilitate the closure of capillary pores in the cement matrix. Simultaneously, they effectively impede water penetration while maintaining permeability to water vapor, thereby promoting the evaporation of moisture [3]. Azarsa et al. [4] state that these additives have been employed in the construction sector for several decades, specifically on concrete structures subject to hydrostatic pressure. They have already demonstrated their capability to diminish permeability and enhance the longevity of such materials.

In cementitious materials, these additives undergo a reactive process wherein they disassociate the pores and fill the cracks, however, their action is contingent upon the presence of moisture [5]. Moreover, these capillary crystallization-based waterproofing systems yield materials of higher durability due to their application method involving the addition to the admixture. This distinguishes them from alternative systems that function by forming a surface barrier and merely filling the pores [6].

The majority of studies regarding crystallizing additives focus on their application in concrete materials. For instance, Pazderka and Hájková [7] stated that the addition of a 2% crystallizing additive to concrete resulted in compressive strength values similar to those of plain concrete after 28 days. Additionally, the water vapor permeability of the concrete was reduced by 16 - 20%. The authors further noted that the complete waterproofing effect of concrete with a crystalline admixture typically manifests around the 12th day following concrete creation.

However, to gain a deeper understanding of the impact of crystallizing additives on mortars, several researchers have investigated their behavior when incorporated into these materials. For example, Jaroenratanapirom and Sahamitmongkol [8] examined the performance of mortars with various additives and observed that the mortar containing 1% of crystallizing additive exhibited the most significant rate of self-healing for cracks measuring up to 0.05 mm in thickness.

Krelani and Ferrara [9] conducted a study investigating the crack self-healing capacity of cementitious materials with the incorporation of crystallizing additives. With a particular focus on the impact of different curing methods, their findings indicated that the efficacy of the crystallizing additive was notably enhanced in specimens subjected to water immersion for curing. Similarly, Žižková et al. [10] explored the influence of curing conditions on the performance of crystallizing additives in mortars. The results revealed that samples stored at 95% relative humidity exhibited a significant reduction in the overall volume of accumulated pores.

In their study, Hodul et al. [11] examined the microstructure of mortars containing a crystallizer additive following a 540-day curing period. Notably, they observed the emergence of well-defined crystals within the porous structure of the mortars, as depicted in figure 1. The authors proposed that this crystal formation played a crucial role in enhancing the properties of the mortar.

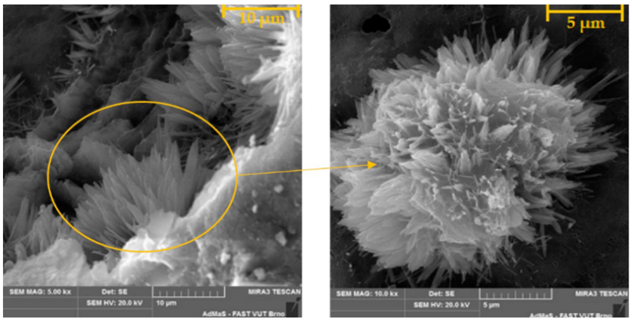


Figure 1: SEM of mortar with crystallizing additive (HODUL, ŽIŽKOVÁ, BORG, 2020).

In an investigation conducted by Jantsch [3], the assessment of water vapor permeability was performed on stabilized mortars aged 36 and 72 hours. These mortars underwent surface treatment using two distinct crystallizing additives. The findings revealed that one of the additives led to a reduction in vapor transmission by forming a surface layer that hindered the material. In contrast, the second additive rendered the mortar more permeable to vapor compared to the untreated mortar, aligning with the manufacturer's claims of enhancing vapor permeability [3].

Regarding vapor permeability in conventional mortars, Temp [12] conducted a study revealing that medium and stabilized mortars exhibit higher vapor permeability (9.00 ng/m.s.Pa) compared to high porosity industrialized mortars (7.00 ng/m.s.Pa). Mortars formulated with light aggregates, surfactant additives, cellulose ether, and powder resin to enhance thermal performance demonstrated resistance coefficients to water vapor (μ) close to 15 [13]. When examining hydraulic and aerial lime mortars, Penas [14] found water vapor permeability values ranging from 25.89 ng/m.s.Pa to 30.56 ng/m.s.Pa, with the air lime component notably improving the water vapor permeability in the mortar.

Intending to enhance comprehension regarding water vapor permeability in mortars containing crystallizing additives, this study focused on the experimental assessment of water vapor transport within the mortar coating. Specifically, it aimed to investigate whether the inclusion of crystallizing additives influenced this property. The evaluation procedure followed the guidelines outlined in the Portuguese standard NP EN 1015-19: Determination of water vapor permeability of hardened plastering mortars [15].

Materials and Method

Figure 2 provides a concise overview of the materials employed and the methodology devised to accomplish the stated research objectives.

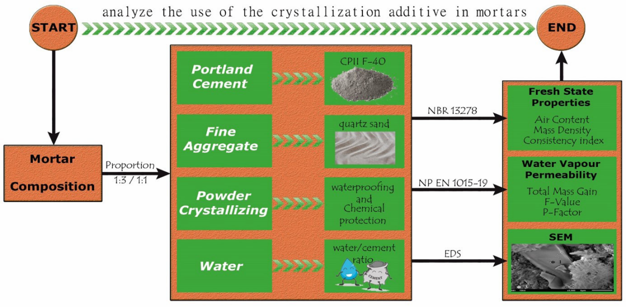


Figure 2: Materials and methods summary.

The binder used in the composition of the mortar in this research was Portland cement composed of filler (CPII F-40, equivalent to CEM I) with a specific mass of 3.03 g/cm^3.

The fine aggregate utilized in this study consisted of quartz sand, which underwent a manual granulometric composition process to achieve similar characteristics to the natural sand found in the study region. This approach was adopted to enhance control over the granulometric composition of the fine aggregate. In this composition, the percentages of fine aggregate passing through the 2.4 mm sieve and retained in the 1.2 mm, 0.6 mm, 0.3 mm, and 0.15 mm sieves were, respectively, 20%, 25%, 35%, and 20%, resulting in unit mass values of 1.51 g/cm^3, the maximum diameter of 2.36 mm, and fineness modulus of 1.45.

The powder crystallizing additive employed in this study possesses a grey color and is characterized by the manufacturer as a chemical treatment for waterproofing and chemical protection. It is specifically formulated for addition during the concrete batching process at the plant. The manufacturer specifies an apparent specific mass of 1.02 kg/L and an absolute specific mass of 2.90 kg/L for the additive. Additionally, water sourced from an artesian well was included in the mixture.

The mortars formulated in this study were prepared using proportions of 1:3 and 1:1 (cement:sand), where the volumes of dry materials were converted to mass. The adoption of the 1:3 proportion was based on its widespread usage as a standard practice in various academic research studies, as well as its recommendation in the Brazilian standard NBR 16072 [16], which specifically addresses waterproof mortars. On the other hand, the selection of the 1:1 proportion was driven by the understanding that cement-rich mixtures tend to yield more favorable outcomes concerning the effectiveness of the additive due to increased interaction between the reactions of Portland cement and the crystallizing agent.

The mixture preparation followed the guidelines outlined in the Brazilian standard NBR 16541 [17], which recommends adjusting the amount necessary to reach the consistency index of 260 mm ± 5 mm. Three different contents of crystallizing additive were incorporated relative to the mass of cement used: 0% (serving as the reference), 1% (as per the manufacturer's recommendation), and 3% (intended for assessing the effects of overdosage).

Consequently, to achieve the recommended consistency index, the water-cement ratio employed for the reference mortars was determined. For the 1:1 reference mortar, a water-cement ratio of 0.40 was fixed, while for the 1:3 reference mortar, a water-cement ratio of 0.75 was established. These ratios were determined by conducting tests following NBR 13276 [18]. Following the consistency index test, the air content and mass density of the fresh-state mortars were determined for characterization purposes using NBR 13278 [19].

The water vapor permeability test, as per NP EN 1015-19 [15], was conducted on hardened samples. For each proportion and additive content, a total of 20 specimens were molded with a thickness of 20 mm and a diameter of 150 mm. Two distinct curing procedures were employed: half of the specimens underwent WC in an acclimatized environment, while the remaining half were subjected to cycles of wetting and drying based on the methodology proposed by Cappellesso et al. [1]. The proposed method suggests immersing the samples in water for 2 days followed by 12 days of drying at a temperature of 30 ± 2 °C. However, in this research, WC was chosen as an alternative to immersion. The mortars were analyzed at 28 and 97 days of curing.

The performance of the vapor permeability test underwent some adaptations of the standard. The dimensions of the test cups were adjusted to meet the recommendation of a reduced opening of 0.02 m^2 (160 mm in diameter). To facilitate the cup fabrication process and enable comparison with the results obtained in the study by Temp [12], which also employed the same reduction, the bottom of the cup was designed with a diameter of 100 mm.

Ultimately, to evaluate the formation of crystals resulting from the incorporation of the crystallizing additive in the mortars, SEM combined with EDS was conducted utilizing a JOEL JSM-6510LV device. The samples subjected to analysis were meticulously prepared and metalized to ensure accurate examination within the equipment. The crystallizing additives were nanoscale materials.

Results and Discussion

Fresh state properties

Table 1 presents the characterization of the mortars in their fresh state. Notably, the inclusion of 3% crystallizing additive content leads to a significant reduction in the consistency index results. This observation can be attributed to the pulverulent nature of the additive, which increases the mass of dry materials within the mortar. Consequently, this alteration affects the workability as the grains of the additive absorb a portion of the moisture present in the mixture.

Furthermore, in the case of the 1:1 ratio, an elevation in the incorporated air content is observed, accompanied by a corresponding decrease in the mass density for higher additive content. This observation may indicate greater challenges in compacting the specimens of this mortar due to its lower consistency index. Conversely, for the 1:3 ratio, a decrease in the incorporated air content is noted, increasing the mass density. Despite a slight decrease in the consistency index, it was not significant enough to hinder the molding process of the mortar specimens.

Table 1: Characterizations of the mortars in the fresh state.

Mortar	Additive content (%)	Consistency index (mm)	Mass density (kg/m^3)	Air content (%)
1:1	0	264	1444	35.4
	1	264	1491	33.0
	3	176	1380	37.5
1:3	0	256	1269	43.5
	1	268	1316	41.2
	3	205	1347	39.6

Water vapor permeability

In the hardened state, the specimens were subjected to two different types of curing: WC and curing cycles (CC). Consequently, the results are reported separately for each type of curing. To ensure data accuracy, spurious data points were treated by removing values above the mean plus the standard deviation and below the mean minus the standard deviation. Subsequently, the remaining data points underwent a statistical analysis of variance to determine any significant differences.

By conducting regular weightings over 15 days, excluding weekends, the mean mass gain, standard deviation, and coefficient of variation for this property were determined and presented in table 2. Following the specified normalization [15], the vapor permeability values were also determined. The table additionally provides information on the water vapor resistance factor (μ), Permeance, and total mass gain over time.

The results demonstrate a direct relationship between mass gain and water vapor permeability. These findings align with those obtained by Temp [12], who reported mass gain values ranging from 0.48 g to 0.69 g for the mortars analyzed in their study. Similarly, high coefficients of variation (ranging from 59% to 73%) were observed, reflecting significant variations in mass gain over the days.

Through a comparison of mortar behavior, it was evident that the 1:3 proportion mortar exhibited water vapor permeability mean values that were 107% higher than those of the 1:1 proportion mortar. This observation aligns with expectations, as reducing the cement content in the material is known to elevate water vapor permeability [20].

The obtained results were subjected to a statistical variance analysis using Statistica® 7 Software, with a confidence level of 95%. The analysis aimed to assess the significance of the variables concerning the properties' outcomes. The significance of these variables can be observed in table 3.

Based on the findings, it can be concluded that the inclusion of crystallizing additives does not have a significant impact on the water vapor permeability of mortars. However, for the 1:1 mortar, the age of the specimen demonstrated an influence on this property. On the other hand, for the 1:3 mortar, both the curing process and the interaction between age and curing exerted a notable influence on the observed results.

Pazderka and Hájková [7] conducted a study to examine the influence of a crystallizing additive on concretes. Their findings indicate that this component has a relatively insignificant impact on water vapor permeability. Consequently, the authors concluded that the incorporation of this additive contributes to enhancing the durability of cement materials by mitigating the risks associated with vapor pressure in structures.

Table 2: Mean values of mass gain at 28 and 97 days.

Age	Mortar	Curing	Additive content	Mass gain			Total mass gain	Permeance	Water vapor permeability	Water vapor resistance factor
				Mean	Standard deviation	Coefficient of variation				
			(%)	(g)	(g)	(%)	(g)	(E-10 kg/ m^2.s.Pa)	(E-11 kg/m.s. Pa)	(μ)
28 days	1:1	WC	0	1.05	0.48	45.59	7.78	6.93	1.39	14.00
			1	0.89	0.37	41.56	6.70	5.93	1.19	16.35
			3	1.38	0.62	44.96	9.14	8.19	1.64	11.82
		CC	0	0.83	0.32	38.06	5.56	4.90	0.98	19.84
			1	0.89	0.37	42.16	5.93	5.24	1.05	18.52
			3	1.02	0.35	34.57	7.58	6.74	1.35	14.38
	1:3	WC	0	1.99	0.89	44.59	12.87	11.7	2.34	8.20
			1	2.52	1.07	42.41	13.96	12.8	2.55	7.52
			3	3.14	0.86	27.53	14.54	13.3	2.67	7.17
		CC	0	2.43	1.13	46.48	12.88	11.7	2.34	8.17
			1	2.36	1.33	56.47	12.57	11.4	2.28	8.38
			3	1.70	1.13	66.43	15.17	13.9	2.79	6.76
97 days	1:1	WC	0	0.84	0.51	60.72	7.27	6.46	1.29	14.94
			1	0.81	0.54	67.36	5.76	5.08	1.02	19.03
			3	0.63	0.36	57.88	5.40	4.76	0.95	20.34
		CC	0	0.68	0.60	87.37	5.78	5.10	1.02	19.02
			1	0.42	0.28	66.94	5.36	4.72	0.94	20.58
			3	0.64	0.29	44.66	5.57	4.91	0.98	19.67
	1:3	WC	0	0.96	0.49	50.83	6.50	5.76	1.15	16.67
			1	0.93	0.40	42.80	6.41	5.67	1.13	16.90
			3	1.04	0.49	47.11	7.50	6.67	1.33	14.34
		CC	0	1.85	1.08	58.31	13.55	1.24	2.47	7.61
			1	2.36	1.44	61.28	18.03	16.8	3.36	5.52
			3	1.82	0.63	34.30	18.99	17.8	3.55	5.26

Table 3: Analysis of variance of controlled variables on water vapor permeability.

Mortar	Variables	Sum of squares	Degrees of freedom	Mean squares	Calculated F-value	P-Factor	Significant
1:1	Additive content (%)	0.000000	2	0.000000	1.1164	0.330837	no
	Age	0.000000	1	0.000000	4.9843	0.027433	yes
	Curing	0.000000	1	0.000000	3.6227	0.059391	no
	Additive content (%)*Age	0.000000	2	0.000000	2.2162	0.113465	no
	Additive content (%)*Curing	0.000000	2	0.000000	0.5337	0.587811	no
	Age*Curing	0.000000	1	0.000000	0.7233	0.396748	no
	Additive content (%) (%)*Age*Curing	0.000000	2	0.000000	0.1529	0.858390	no
	Error	0.000000	120	0.000000			
1:3	Additive content (%)	0.000000	2	0.000000	0.9784	0.378907	no
	Age	0.000000	1	0.000000	0.8891	0.347628	no
	Curing	0.000000	1	0.000000	10.0129	0.001970	yes
	Additive content (%)*Age	0.000000	2	0.000000	0.1362	0.872809	no
	Additive content (%)*Curing	0.000000	2	0.000000	0.2673	0.765902	no
	Age*Curing	0.000000	1	0.000000	10.6664	0.001422	yes
	Additive content (%) (%)*Age*Curing	0.000000	2	0.000000	0.3382	0.713749	no
	Error	0.000000	120	0.000000			

SEM

SEM was employed to capture images to observe the formation of crystals resulting from the inclusion of a crystallizing additive in the mortars. Additionally, EDS was utilized to identify the chemical components present in the specimens.

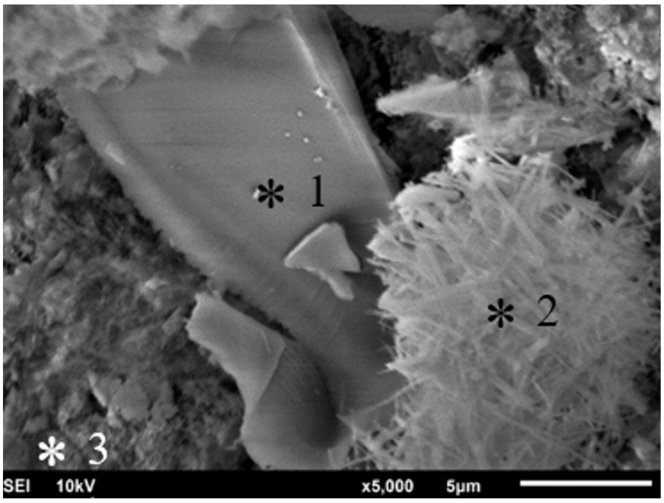
Figure 3 provides a visual representation obtained at a magnification of 5000 times, showcasing the formation of crystals in the 1:3 mortar composition containing 3% crystallizing additive and subjected to 97 days of WC. Additionally, the figure depicts the points analyzed by EDS to determine the chemical components present. Within figure 3, the presence of Portlandite ($Ca(OH)_2$) is observed, which is a plate-type hydrate formed during cement hydration. Furthermore, ettringite, a needle-type hydrate, is also visible. Ettringite exhibits volume expansion during its formation, thereby contributing effectively to crack self-healing [21].

Figure 4 illustrates a 1:3 mortar composition with a 3% crystallizing additive, subjected to wetting and drying cycles for a curing period of 97 days. The SEM image, captured at a magnification of 5000 times, reveals a distinct feature indicating the potential for crack self-healing.

The images presented in figure 3 and figure 4 exhibit similar characteristics to those depicted in figure 1, as observed in the study conducted by Hodul et al. [11]. The authors established a correlation between the formation of crystals and the improvement of mortar properties. Notably, the EDS analyses of both images reveal the predominance of $Ca(OH)_2$ particles. However, a distinguishing factor between the two EDS analyses is the presence of sodium (Na) in the second sample, albeit in small quantities. It is worth mentioning that the manufacturer does not disclose the precise chemical composition of the crystallizing additive but confirms its reactivity with $Ca(OH)_2$ to generate a crystalline solution within the pores of the cementitious material.

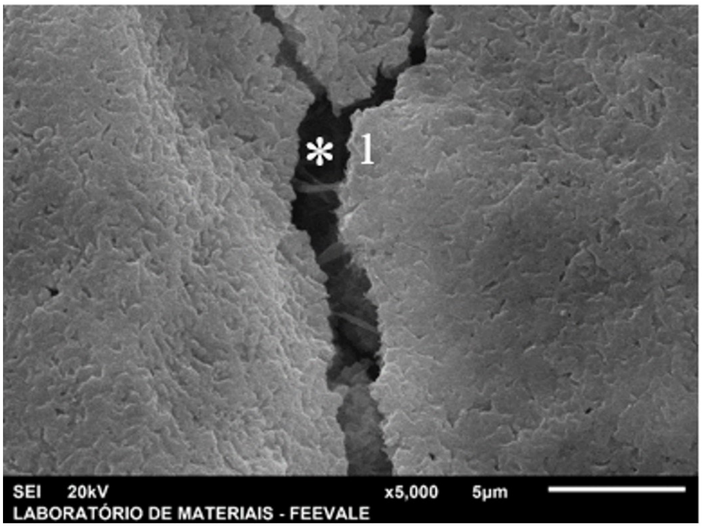
Conclusions

The inadequate consideration given to the performance



Chemical component	Point 1	Point 2	Point 3
C	1.07	1.38	1.02
O	33.46	27.71	26.46
Mg	1.14	1.53	0.51
Al	2.42	2.40	1.25
Si	11.39	12.20	6.15
S	1.27	1.41	0.77
Ca	47.24	52.19	63.83
Ti	0.49	-	-
Fe	1.52	1.18	-

Figure 3: SEM of 1:3 mortar proportioning, with 3% of crystallizing additive and 97 days of wet curing: crystal formation and points analyzed by EDS.



Chemical component	Point 1
C	5.56
O	55.02
Na	0.67
Mg	1.23
Al	1.36
Si	7.26
K	0.64
Ca	27.37
Ti	0.18
Fe	0.71

Figure 4: SEM of 1:3 mortar proportioning, with 3% of crystallizing additive and 97 days of curing cycles.

requirements of coating mortar often leads to the emergence of pathological manifestations during its lifecycle. Therefore, this research aimed to analyze the effectiveness of a crystallization additive in enhancing the water vapor permeability of mortars. It focused on investigating a property that is frequently overlooked but holds significant importance, particularly in the context of the most commonly utilized type of coating in Brazil: the water vapor permeability of mortars. Therefore, the subsequent set of conclusions is presented, based on the materials and methods applied in the present study:

- The incorporation of a 3% dosage of crystallizing additive resulted in a notable modification in the consistency of the mortars, particularly in the 1:1 mixture. Consequently, when considering a higher dosage of the crystallizing agent in the mortar, it is crucial to evaluate the inclusion of additional additives that contribute to prolonging the workability of the mortar.
- WC decreased water vapor permeability.
- The 1:3 mortar proportion had an average water vapor permeability 107% higher than the 1:1 mortar proportion.
- The highest vapor permeabilities found were for mortars tested at 28 days.
- The inclusion of the crystallizing additive did not show a substantial influence on the results of water vapor permeability. This observation supports the notion that the additive does not create a vapor barrier, thus it allows the material to maintain its breathability.

Finally, the utilization of the crystallizing additive in coating mortars is promising, as it did not result in pore plugging. In contrast to other waterproofing additives, the crystallizing additive does not impede the evaporation of water within the mortar.

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Conflict of Interest

None.

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