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Tensile bond strength of adhesive mortars with hydroxypropyl methyl cellulose and vinyl acetate-ethylene polymers after thermal storage

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Abstract: The properties of adhesive mortars can change due to heating, compromising the durability of the coating systems. The aim of this article was to evaluate the influence of cement and polymer contents on the tensile bond strength of adhesive mortars after thermal storage. Ceramic tiling system specimens were prepared with seventeen formulations of adhesive mortars. These specimens were stored under dry (reference) at temperature of 23 ± 2 °C and 60 ± 5% of relative humidity and thermal (temperature of 70 ± 2 °C) conditions. The results showed that the cement content was the major factor concerning tensile bond strength. The vinyl acetate-ethylene (VAE) polymer improved the tensile bond strength of mortars under thermal storage. However, the hydroxypropyl methyl cellulose (HPMC) content contributed to the tensile bond strength only when higher cement contents were used. Besides, microstructure analysis showed that ettringite was degraded during thermal storage.

Key words: Adhesive mortars, polymers, vinyl acetate-ethylene, tensile bond strength, thermal storage.

INTRODUCTION

Adhesive ceramic tiling is a traditional system used as inner and outer building coating. The main assignment of the ceramic tiles is to provide adherence to the base and to level any uneven spots. For that reason, a contact bridge must be used to ensure the tiles adhere correctly. The materials commonly used for this function are adhesive mortars. Moreover, once the systems are properly applied, these materials will ensure a long service life to the building coating, protecting the structural systems and allowing ceramic tiles to serve their technical and aesthetic purpose (Modler et al. 2021, İnceoğlu et al. 2021).

The main component of adhesive mortars is Portland cement. The content of this binder in the adhesive mortars can range from 20 wt.% to 40 wt.%. Also, there are fine aggregates (particle size ≤ 0.6 mm) with the binder/aggregate ratio between 1:2 or 1:3 by weight. However, these proportions can vary considerably depending on the type of the adhesive mortar. The modified polymers used in this mixture are typically cellulosic ethers, such as water retainers. Also, vinyl polymers are commonly used as redispersible powders (RP). The particular application will determine what requirements are to be made on the adhesive mortar´s adhesion and plasticity and, therefore, how much polymer should be added (Wacker 2004, Maranhão et al. 2011).

Therefore, the formulations of adhesive mortars can be quite different. These compositions will depend on their application, for example, if external or internal ceramic tile systems will be applied (Wacker 2004). The external ceramic tile system suffers stress because of the demanding environmental conditions, the action of gravity, and greater background deformation. In this sense, considering it is a complex multi-layer system, each layer displays different properties, requiring distinct rigid bonds characteristics. Thus, polymer-modified mortars are used to set ceramic tiles, mainly because they improve mechanical properties and bond strength and flexibility (de Azevedo et al. 2017, Peng et al. 2020).

The main purpose of the polymers is to significantly offer some properties on the adhesive mortar, such as water retention, plasticity, flexibility, fluidity, permeability, adhesiveness, chemical resistance, mechanical resistance, and to provide more durability of the ceramic tile systems in buildings (Zhang et al. 2021, Wang et al. 2016). The solutions of these polymers, like any other real system, exhibit an intermediate behavior between a perfectly solid and an ideal liquid, which means they exhibit viscoelastic behavior. Consequently, such admixtures may interact with the other components of the formulation, in particular with the binder, thus modifying the properties of the mortars in the fresh and hardened state (Wang et al. 2020).

The cellulose ether (CE) has a notable influence on the adhesive mortars in the fresh state. This material improves workability and water retention due to its high water retention capacity. Also, CE allows good application time (subsequently defined as open time) in a thin bed of mortar to a substrate for the tile installation. The most widespread cellulose ethers used to formulate the adhesive mortars are the hydroxyethyl methyl cellulose (HEMC) and the hydroxypropyl methyl cellulose (HPMC) (Metalssi et al. 2014, Wang et al. 2021). According to literature studies (Silva et al. 2001, Jenni et al. 2006), the microstructure of the polymer-modified mortar is typically dominated by a large number of air voids (more than 20 vol.%) and this characteristic improves the workability in the fresh state, but it can negatively affect the mechanical strength in a hard state.

Another type of material used in cement composites is the redispersible powder (RP), which positively influences the rheological characteristics of adhesive mortars. RP's main function is to improve the final characteristics of adhesive mortars, such as adhesion, flexibility, and deformability (Wacker 2004). The polymer film provides a better bond between the hydrated cement and the aggregates, filling the porosity of the cement co-matrix and increasing the flexural strength of the systems (Wang et al. 2016, Li et al. 2021).

In this context, it is important to understand how each component of the adhesive mortars influences a better final performance of the entire ceramic tiling system, especially when it is exposed to difficult conditions such as heating. Therefore, the aim of this article was to evaluate the tensile bond strength of mortar composites with hydroxypropyl methyl cellulose (HPMC) and redispersible vinyl acetate-ethylene powder (VAE) polymers after thermal storage.

The influence of polymers additives on adhesive mortars' properties has been the focus of several scientific investigations, including some studies that evaluated the action of water on adhesive mortars (Modler et al. 2021, Peng et al. 2020, Zhang et al. 2021, Wang et al. 2020, Liao et al. 2019). However, the main innovation of this article is evaluating the tensile bond strength after thermal storage. To achieve the purpose of this study, some formulations of adhesive mortars were developed in a laboratory, varying the content of cement and polymers. Furthermore, composite systems (concrete substrate - adhesive mortar – ceramic tile) were prepared and stored under two different conditions.

MATERIALS AND METHODS

Materials

Brazilian Portland cement CP-II F 32 (9.5% limestone) equivalent to Portland-limestone cement CEM II/A-L 32.5N was used. Table I shows the chemical and physical characteristics of the cement. Quartz sand was used as fine aggregate.

Table I. Cement chemical and physical characteristics.

Property	Unit	CP-II F
CaO	%	55.29
SiO_2	%	23.16
Al_2O_3	%	3.20
MgO	%	9.09
SO_3	%	2.19
Fe_2O_3	%	2.84
Na_2O	%	0.36
K_2O	%	0.99
Mn_2O_3	%	0.008
P_2O_5	%	0.10
Free CaO	%	2.48
Insoluble residue	%	1.02
TiO_2	%	0.18
ZnO	%	0.02
SrO	%	0.08
Specific gravity	g/cm^3	3.13
Initial setting time	min	230
Final setting time	min	305
Fineness Blaine	cm^2/g	3530
# 200	%	1.30
# 325	%	6.10
Compressive strength (3 days)	MPa	27.0
Compressive strength (7 days)	MPa	32.1
Compressive strength (28 days)	MPa	40.1

Also, HPMC and VAE polymers were used in some mortars. Table II shows the main characteristics of these polymers.

Mortar mixtures

Seventeen adhesive mortar mixtures were produced in this study. Table III shows the proportion of materials in the different adhesive mortars. As common in the mortar industry, the percentages shown in Table III refer to 100 wt% of the dry mix without considering the percentage of the polymers in this amount. These compositions were selected based on values found in the literature and taking to account the recommendations from the polymer's manufacturers.

The definition of the 17 mixtures was done in three steps. In the first step (mortar mixtures 1-4), the Portland cement content varied in proportions 25%, 30%, 35%, and 40%, and the polymer contents were fixed (0.20% HPMC and 2.0% VAE). In the second step (mortar mixtures 5-8), the HPMC content varied in proportions 0.15%, 0.20%, 0.25%, and 0.30%, and the cement and VAE contents was fixed at 25.0% and 2.0%, respectively. Finally, the VAE polymer content varied in the contents of 1.0%, 1.5%, 2.0%, and 2.5% in the third step, while the HPMC content was kept constant at 0.20%, and the amount of cement varied between 25% and 35% (mortar mixtures 9-17). Thus, a wide range of mortar types was used in this article, allowing a high reproducibility of results. The water content of the mixtures was determined according to the slip test proposed by NBR 14081-5 (2012b) standard.

The adhesive mortar mixtures were casted in a laboratory under controlled environmental conditions, temperature of 23 ± 2 °C and 60 ± 5% relative humidity, according to NBR 14081-2 (2015) standard. Individual mortar components were weighed and placed into a plastic mixing bag for

Table II. Portland cement physical and mechanical characteristics.

Hydroxypropyl methyl cellulose (HPMC)		Vinyl Acetate-Ethylene (VAE)	
Form:	**Powder**	**Protective colloid:**	**Polyvinyl Alcohol (PVA)**
Solubility:	Water-soluble	Solids content:	(99 ± 1) %
Particle size:	< 177 µm: min. 95%	Ash content:	8 - 12%
Viscosity (mPa s):	48,000~56,000	Tg:	12 – 18 °C
Moisture content:	Max. 8%	Particle size:	90 µm
pH:	6.0~8.0 (2% solution)	Bulk Density:	0.5 (g/ml)
		Viscosity at 25 °C:	2000 mPa.s
		pH:	7

preparing the dry mix materials. Subsequently, the bag was sealed and shaken vigorously by hand for approximately 90 seconds. This type of mixing is an industry-proven simulation for blending operations in the manufacturing of dry mix mortar products (Brien & Mahboub 2013).

After the prior preparation of the dry materials, the mortar casting continued in accordance with the NBR 14081-2 (2015) standard. The initial water content used for the mixing of the mortars was based on an average indicated for market mortars, as recommended by the manufacturers, and this content was adjusted corresponding to the performance of the mortars in the slip test (NBR 14081-5 2012b). A limit slip of less than 2 mm was considered, according to NBR 14081-2 (2015) standard.

Preparation of specimens (ceramic tiling system)

The ceramic tiling system specimens were composed of three layers: concrete substrate, adhesive mortar bed, and ceramic tile. The specifications of the concrete substrate used in the ceramic tiling systems followed the NBR 14081-2 (2015) standard. The concrete substrate was slabs with dimensions of (25 x 50 x 2) cm and the concrete had a tensile strength greater than 2.5 MPa and water absorption of 0.30%. Besides, the ceramic tile dimensions of (10 x 10) cm had water absorption of less than 0.5%.

The freshly mixed mortars (Table III) were applied to the concrete substrate using the notched side of the trowel, making longitudinal strings with a height of (5 ± 0.5) mm. Then, after 5 minutes of the mortar´s application (open time), the (50 x 50) mm ceramic tiles were placed and each tile was loaded with a weight of 2 kg for 30 seconds. After that, the ceramic tiling system was ready to be stored.

Two different storage conditions were applied. The first was called “dry” in this article and consisted of the storage of ceramic tiling system specimens for 28 days at laboratory environmental conditions (temperature of 23 ± 2 °C and 60 ± 5% relative humidity). The second was called “thermal” in this article and consisted of the storage of ceramic tiling system specimens initially for 14 days at laboratory environmental conditions and later for another 14 days inside a heater. The heater was configured with a temperature of (70 ± 2) °C and had air circulation and humidity control, as suggested by NBR 14081-4 (2012a) standard.

Table III. Mortar mixtures formulations.

Code	Portland cement	Fine aggregate	HPMC	VAE	Water	w/b ratio
1	25.0%	75.0%	0.20%	2.0%	23.0%	0.92
2	30.0%	70.0%	0.20%	2.0%	23.6%	0.79
3	35.0%	65.0%	0.20%	2.0%	24.0%	0.69
4	40.0%	60.0%	0.20%	2.0%	24.5%	0.61
5	25.0%	75.0%	0.15%	2.0%	23.0%	0.92
6	25.0%	75.0%	0.20%	2.0%	23.0%	0.92
7	25.0%	75.0%	0.25%	2.0%	23.0%	0.92
8	25.0%	75.0%	0.30%	2.0%	23.0%	0.92
9	35.0%	65.0%	0.15%	2.0%	23.0%	0.66
10	35.0%	65.0%	0.20%	2.0%	23.0%	0.66
11	25.0%	75.0%	0.20%	1.0%	23.0%	0.92
12	25.0%	75.0%	0.20%	1.5%	23.0%	0.92
13	25.0%	75.0%	0.20%	2.0%	23.0%	0.92
14	25.0%	75.0%	0.20%	2.5%	23.0%	0.92
15	35.0%	65.0%	0.20%	1.0%	23.0%	0.66
16	35.0%	65.0%	0.20%	1.5%	23.0%	0.66
17	35.0%	65.0%	0.20%	2.0%	23.0%	0.66

Tensile bond strength and microstructure tests

The pull-off test was applied after 28 days of storage (dry or thermal) to determine the tensile bond strength of the adhesive mortars, according to NBR 14081-4 (2012a) standard. A Proceq Dyna Z6 Pull-off Tester equipment with a Digital Manometer force gauge was used. The loading speed was 250 N/s. Twenty specimens per mortar mixtures were used, representing the high repeatability and reliability of the results.

Cement pastes were casted to study the microstructure of the composites (Table IV). The formulations in Table IV were selected according to their performance in the tensile bond strength test. X-ray diffraction (XRD) and scanning electron microscopy (SEM) analyses were performed.

The mortar paste was used to complement the investigation of the physical and chemical modification mechanism in the tensile bond strength mainly after thermal storage. The formulations were prepared with cement, polymers, and water. The fine aggregate (quartz sand) was eliminated from the composition in order to remove this variable since the elements affected by thermal storage are the hydrated cement phases. The water-to-binder (w/b) ratio was 0.46 since it was noted that this w/b ratio provided smooth paste.

The cement powder was mechanically mixed for 1 min and 30 seconds with water, achieving a homogeneous and lump-free mixture. The specimens were obtained from the casting of a 2 cm thick layer of the paste in a plastic container. After three days, these specimens were removed from the plastic container and cut in the middle with an iron cutting saw. Thus, two 1 cm thick specimens were obtained. These specimens were stored under the same conditions (dry

Table IV. Cement pastes mixtures select for microstructure analyses.

Formulation	A	B	C	D	E	F	G	Pure Paste
Portland Cement	25.0%	25.0%	35.0%	25.0%	25.0%	35.0%	35.0%	25.0%
HPMC	0.20%	0.30%	0.15%	0.20%	0.20%	0.20%	0.20%	-
VAE	2.0%	2.0%	2.0%	1.5%	2.0%	1.0%	2.0%	-
w/b ratio	0.46	0.46	0.46	0.46	0.46	0.46	0.46	0.46

or thermal) performed for the tensile bond strength test.

The samples for SEM investigation were reduced in size by manual sanding performed with thick sandpaper (N°320). Then, the polishing of the samples was performed using a polishing agent to leave the surface of the cement pastes free of irregularities. The samples were coated with a thin gold film by sputtering using a low deposition rate. The sample holder was inserted into the scanning electron microscope with low vacuum and coupled EDS. The equipment features the Gatan MonoCL 4 plus Luminescence (CL) system, capable of obtaining panchromatic, monochromatic, and spectral images in the range of 165 to 930 nm.

The samples to XRD analysis were milled in the pan mill for 10 seconds, leaving the material in powder form. Samples with manual pressing in the sample holder were then prepared and inserted into the diffractometer. XRD assays were conducted in PW1710, Philips equipment. The parameters used for this analysis were scanning time of 20 minutes and counting speed of 40 seconds per step at angles 2Θ between 5° to 60° and with step time of 0.017 degrees/second. As an X-ray source, the copper anode ceramic tube with a wavelength of 1.540598 Å was used, at a maximum power of 1600 W.

Finally, an analysis of variance (ANOVA) with 95% reliability (5% significance level) was performed to verify the significance between the differences in results.

RESULTS

Fig. 1 shows the tensile bond strength of adhesive mortars formulated with different cement contents (25 to 40 wt.%). According to Fig. 1, mortars with higher cement contents showed higher tensile bond strength, mainly for thermal storage. The 40% cement content mortar had an 87% greater tensile bond strength than the 25% cement content mortar. The exception was the mortars with cement contents of 30% and 35% in dry storage. In this case, the tensile bond strength remains statistically the same.

The increase in tensile bond strength with cement content is due to the lower w/b ratio (Table III). This lower w/b ratio reduces the amount of air incorporated in the mortars, directly influencing the performance of the material in the hardened state. To prove this hypothesis, the amount of air incorporated was measured in the mortars. The results showed that the 40% cement content mortar had 26.7% less air incorporated than the 25% cement content mortar.

The effect of cement content was greater in mortars with thermal storage. Elevated temperatures can increase the possibility of early release of water from the mortar, increasing the number of voids in the internal structure of the material (Kjellsen et al. 1990). This effect explains the lower tensile bond strength of mortars with 25% and 30% cement content in thermal storage compared to dry storage. However, high

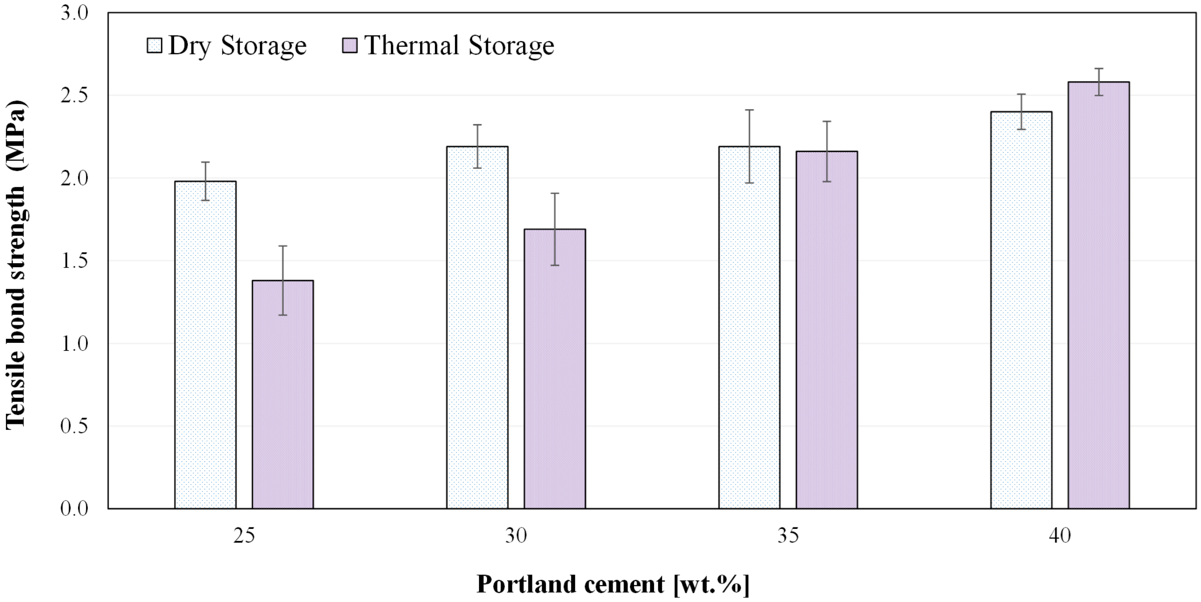

Figure 1. Tensile bond strength of mortars with 0.20% HPMC and 2.0% VAE.

temperatures can also increase the kinetics of cement hydration reactions. This explains why mortars with higher cement contents (35% and 40%) showed a marked increase in tensile bond strength when cured at 70 °C.

Fig. 2 shows the effect of the HPMC polymer on the tensile bond strength of mortars. The cement content was the most important factor to impact the tensile bond strength of mortars, independently of the HPMC content.

According to Fig. 2, increasing the HPMC content slightly reduced the tensile bond strength. According to Beeldens et al. (2005), this phenomenon occurs due to the formation of a polymer film that can partially or completely encompass the cement grains causing delay or even interruption of the cement grain hydration. However, this effect was greater for the dry storage. Thus, the difference between the tensile bond strength between the two storage conditions (dry and thermal) decreased as the HPMC content increased. This means that these polymers can partially contribute to the strength of mortars when exposed to high-temperature conditions.

Fig. 3 shows the tensile bond strength results of adhesive mortars with different VAE contents. The VAE polymer provided an increase of more than 100% on the tensile bond strength from the lower (1.0%) to the higher (2.5%) contents in the mortars with 25% of cement concerning thermal storage. This growth was also observed for dry storage up to 2.0% VAE concentration.

Therefore, the different VAE content influences mortar behavior contributing to higher tensile bond strength when exposed to high temperatures indifferent to the amount of cement. According to Beeldens et al. (2005), the addition of redispersible powders in cementitious materials promotes the formation of a polymeric film that interacts in the microstructure of these materials, acting as a bridge. This behavior may favor the formation of microcracks instead of larger cracks, providing greater compressive strength and adhesion, in addition to increasing the ductility of the materials (Beeldens et al. 2005). Thus, the interaction of the VAE polymer in the composition of the adhesive mortars is important for the mortars to reach their properties even on the effect of temperature.

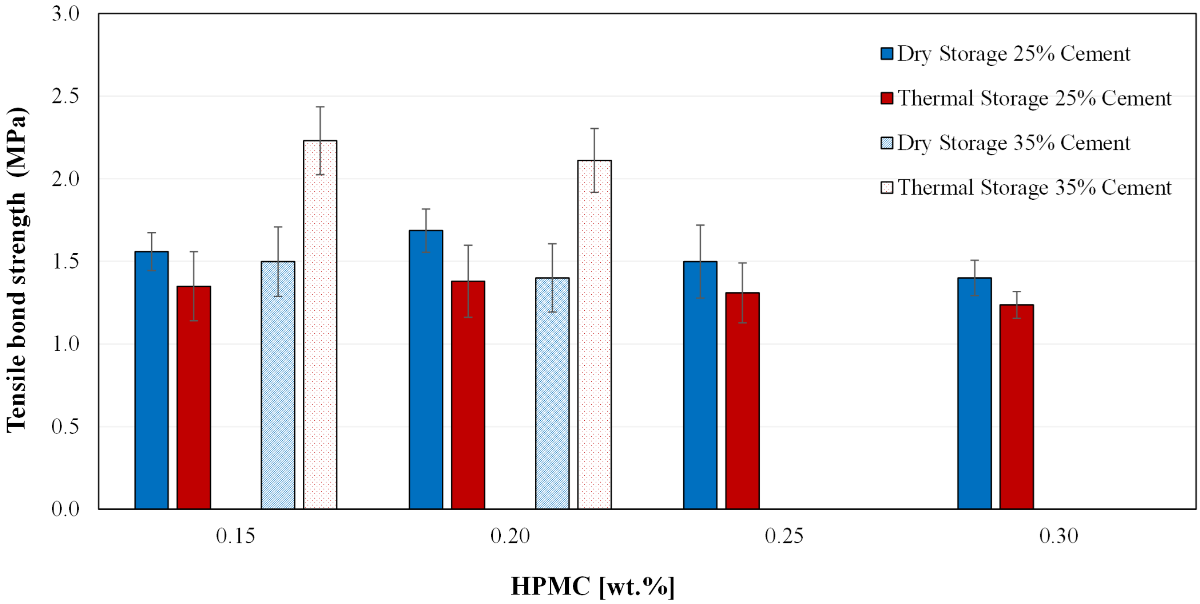

Figure 2. Tensile bond strength of mortars with different HPMC content.

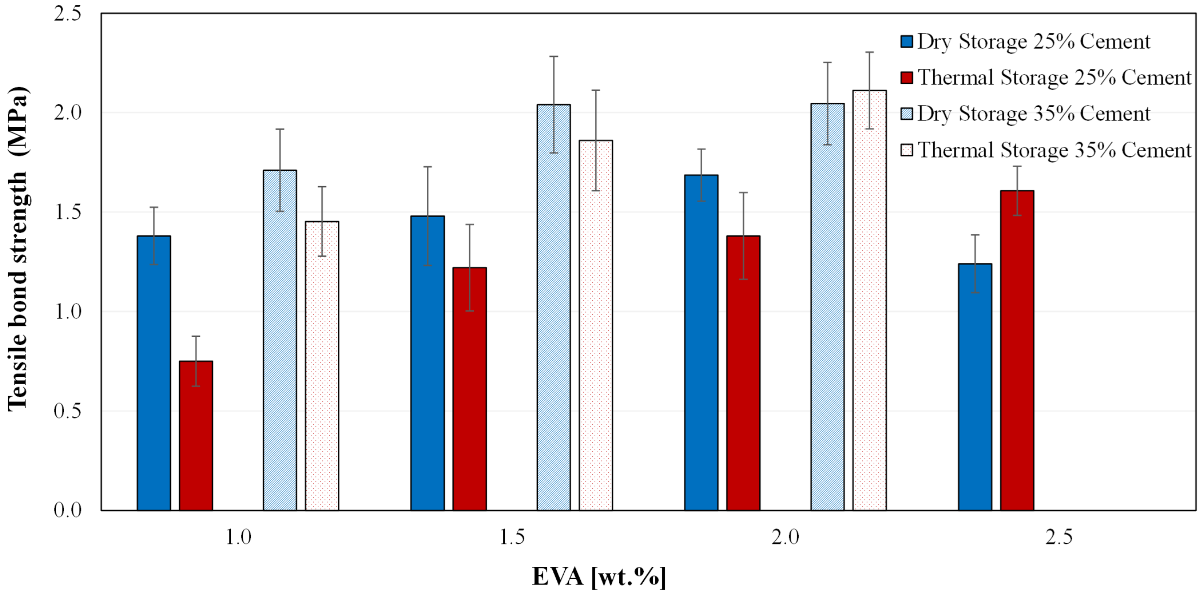

Figure 3. Tensile bond strength of mortars with different VAE content.

Fig. 4 shows the X-ray diffraction results. The most intense peaks identified were the chemical elements common in the mineralogy of the Portland cement pastes. The main peaks found were ettringite (E), C_4AF, portlandite (P), calcite ($CaCO_3$), C_2S, magnesium oxide (M_gO), gypsum (Gyp), and C_3S.

The diffractogram in Fig. 4 represents all the studied cement pastes (Table IV). Therefore, it was not possible to identify any phase of the reaction of the polymers with the cement, since the XRD method only identifies the crystalline phases of a solid. Thus, there was no significant change in the mineralogy of the cement paste when comparing pastes of mortar with different content of polymers and cement. However, when comparing samples under different storage (dry or thermal) (Fig. 5a), the main peak of ettringite

(9.071 ° 2θ) disappears in the diffractogram of the thermal storage (Fig. 5b), indicating that the temperature (70 °C) caused the degradation of this phase.

The disappearance of the main peak of ettringite was also noted by Hacker et al. (2011) after thermal storage for 14 days at 70 °C. This author studied sixty formulations of adhesive mortars modified with cellulose ether. The ettringite and calcium silicate hydrate (C-S-H) are the minerals responsible for the mechanical resistance of cementitious materials. Therefore, the loss of tensile bond strength of the adhesive mortar after thermal storage (Figs. 1-3) can be related to the degradation of ettringite when the temperature increased. Trentin et al. (2022) also observed a loss of strength in cement composites due to the disappearance of the main peak of ettringite.

Fig. 6a shows the images of the pure paste (cement + water, Table IV) from dry storage. the peaks of the common chemical components found in the cement appear in the EDS spectrum (Fig. 6b). A large concentration of calcium phases was noted on thermal storage pastes (Fig. 7a, b).

Moreover, Fig. 8a, b shows the SEM images of the cement paste containing 25.0% Portland cement, 0.15% HPMC, and 2.0% VAE (by cement weight) from dry storage. Thus, ettringite crystals were observed in the paste in dry

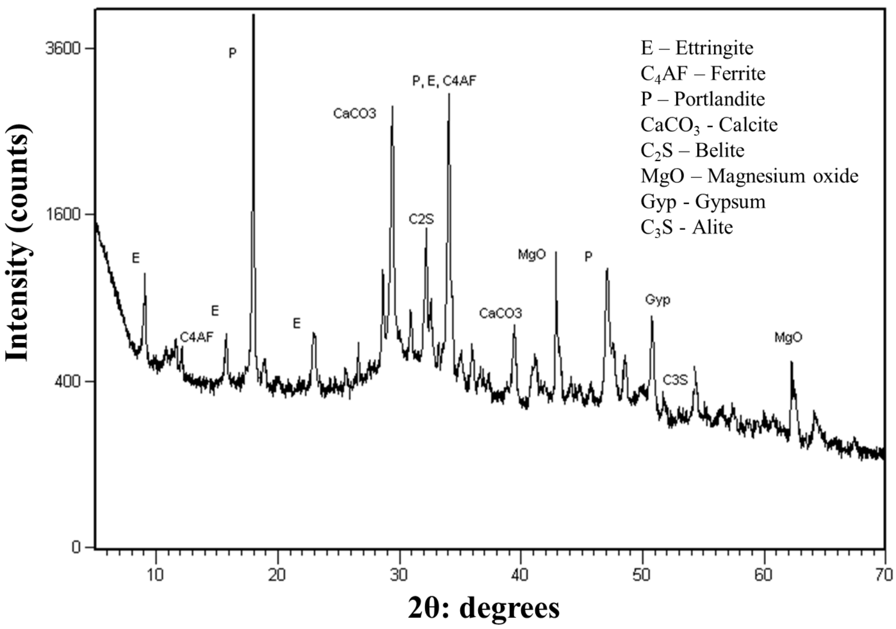

Figure 4. Diffractogram of cement pastes.

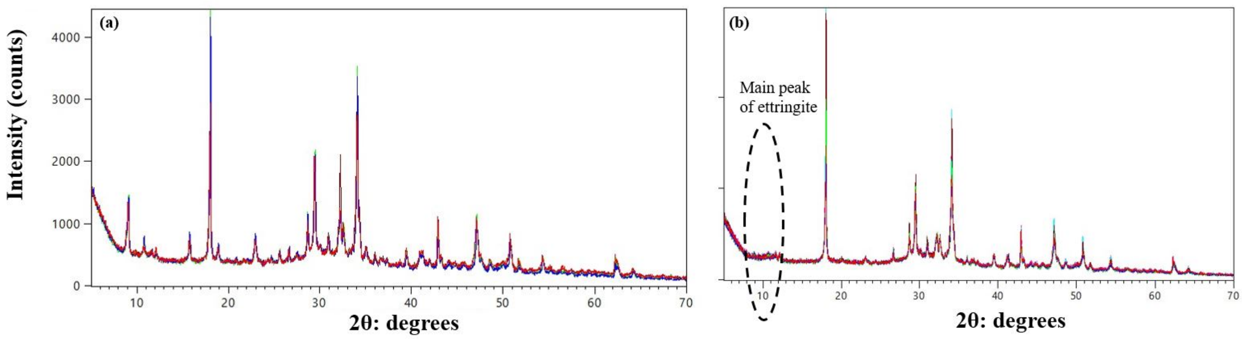

Figure 5. XRD results showing the disappearance of the main peak of ettringite (a) dry storage (b) thermal storage.

storage. However, the presence of ettringite crystals was not identified (Fig. 9a) for thermal storage pastes, but a calcium-rich structure (Fig. 9b) was noted. Therefore, the hypothesis of ettringite decomposition was confirmed again and these results indicate that higher calcium concentrations are found in the sample cured at 70 °C. As noted by Pereira et al. (2013), if there are a low amount of ettringite crystals in the structure to provide mechanical anchoring, the system depends more on the chemical anchoring provided by the polymers additives to exhibit a tensile strength at the interface of those layers. This is why the contribution of polymers to tensile bond strength is greatest in the thermal storage condition (Figs. 1-3).

DISCUSSION

The influence of Portland cement and polymers content on tensile bond strength of adhesive mortars after thermal storage can be described as follows:

(1) The effect of cement content was greater in mortars stored in thermal conditions. The lower tensile bond strength of mortars with 25% and 30% cement content in thermal storage compared to dry storage is explained since elevated temperatures can increase the early release of water from the mortar, increasing the number of air voids. However, mortars with higher cement contents (35% and 40%) showed a marked increase in tensile bond strength

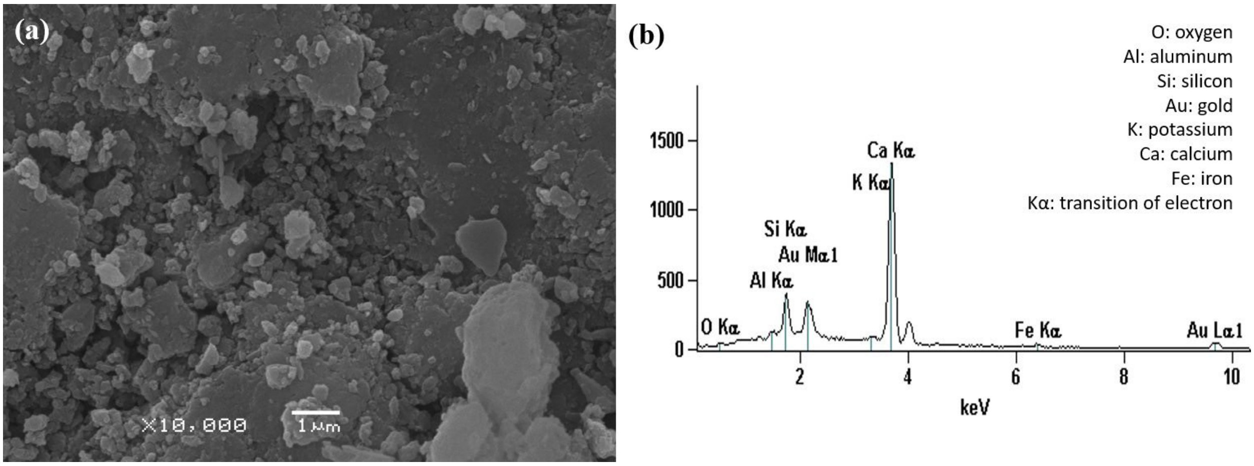

Figure 6. Pure paste sample from dry storage (a) SEM (b) EDS.

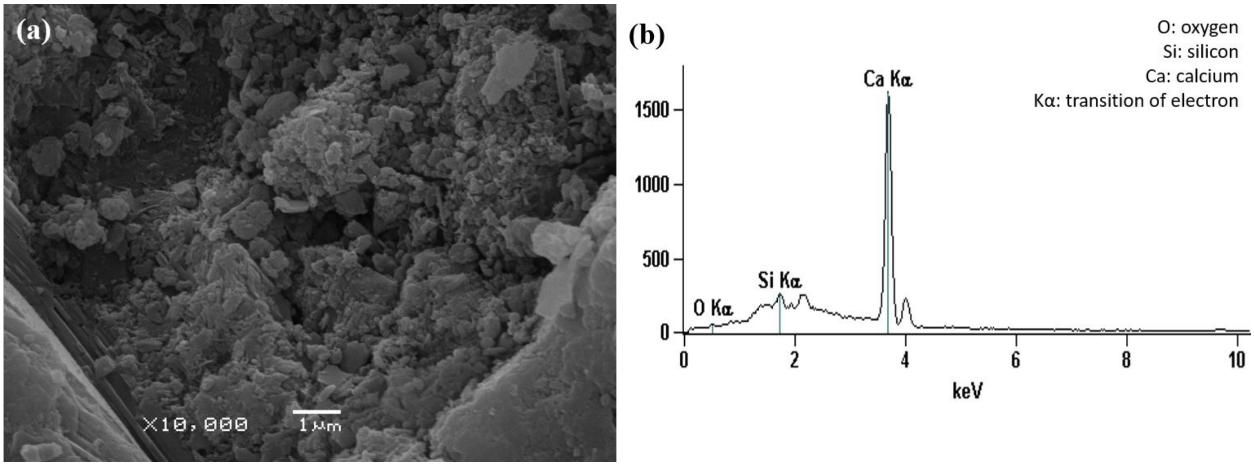

Figure 7. Pure paste sample from thermal storage (a) SEM (b) EDS.

when stored at 70 °C as high temperatures can also increase the kinetics of cement hydration reactions.

(2) The difference between the tensile bond strength between the two storage conditions (dry and thermal) decreased as the HPMC content increased. This means that these polymers can partially contribute to the strength of mortars when exposed to high-temperature conditions.

(3) The tensile bond strength of the adhesive mortars after thermal storage increased by more than 100% when VAE polymer content increased from 1.0% to 2.5%. This increase was also noticed for dry storage up to 2.0% VAE concentration.

(4) The XRD results showed no change in the phases formed between cement pastes with different content of polymers and cement. However, the main peak of ettringite disappears in the diffractogram of the thermal storage when comparing samples under different storage (dry or thermal), indicating that the temperature caused the degradation of this phase.

(5) Finally, the ideal formulation proposed by this article from the studied materials was 35% of Portland cement, 0.2% of HPMC, and 2.0% VAE. According to the results of this article, this formulation showed good performance regarding thermal storage. The cement content is essential to provide the highest tensile

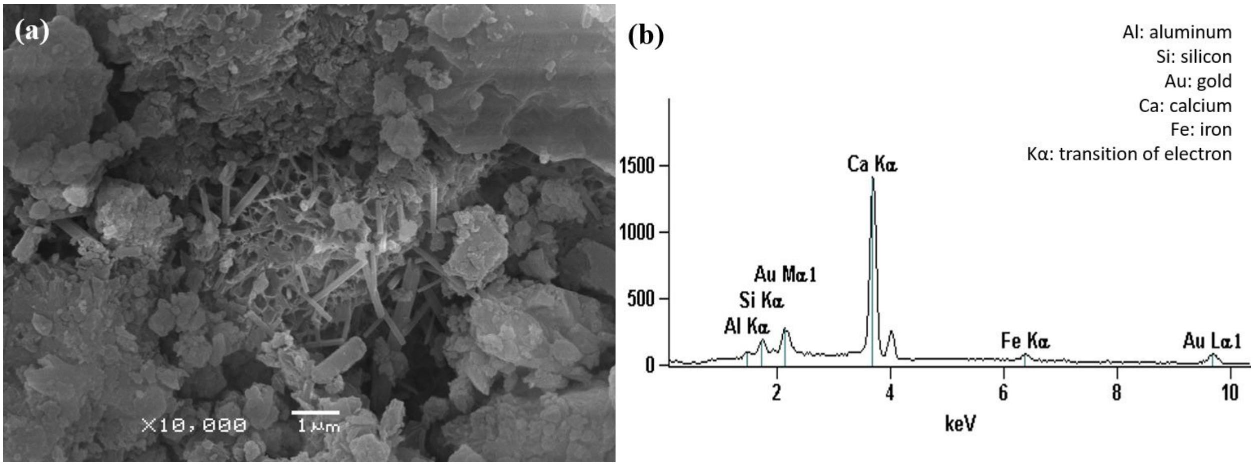

Figure 8. Cement paste with polymer from dry storage (a) SEM (b) EDS.

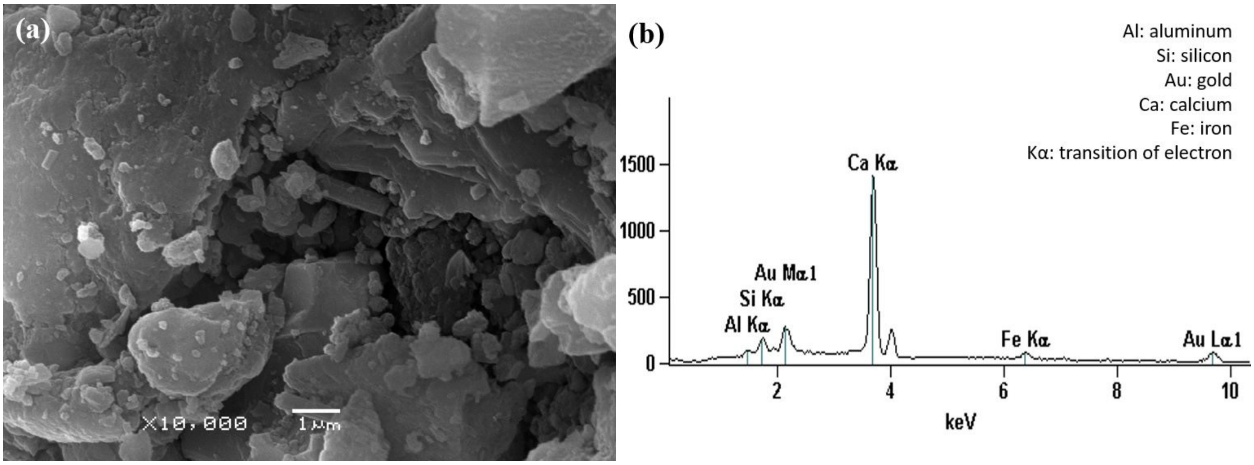

Figure 9. Cement paste with polymer from thermal storage (a) SEM (b) EDS.

bond strength. However, considering that the ceramic tiling system suffers shear stresses on the interface of materials, the adhesive mortar has to provide enough flexibility to keep all layers adhered to each other. Accordingly, the formulation of adhesive mortars should contain vinyl polymers to provide more flexibility to the entire system and avoid failure of a tiling system.

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