CERAMIC TILE ADHESIVES: DETERMINATION OF OPEN TIME BY L_1 REGRESSION

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ABSTRACT

This research study deals with the mechanisms that control the open time of ceramic tile adhesives and new methods to estimate adhesive open time.

Mortars with mix proportion 1:3 (cement:sand, by weight) were formulated with a HEC content of 0 and 0,4 %, and/or EVA of 0 and 10 % (cement weight). The used water/cement ratio was constant and equal to 0,80 (weight). All raw materials as well as mortars were characterized by physical, chemical and mechanical tests. Ceramic tile adhesives obtained from the Brazilian market were also evaluated. The adopted test methods are summarized and results are presented.

The data show that the loss of adhesion strength increases with time. A new method is proposed to evaluate the open time based on the correlation between adhesion strength and time. The correlation is best determined by the L_1 regression method. Extreme values and outliers hardly affect L_1 regression. It is possible to conclude that the open time is controlled by the rate of adhesion strength loss and by the initial strength. HEC controls the rate of adhesion strength loss. EVA controls the initial strength and has little effect on the adhesion rate.

1. INTRODUCTION

Open time is one of the main properties of ceramic tile adhesives. Open time is the period of time after spreading the ceramic tile adhesive on the concrete slab, wherein it is possible to set the ceramic with appropriate tensile adhesion strength. This strength is defined by NBR 14083 [1] (ABNT, 1998) and CEN/TC 67 (CEN, 1993)[2] as greater or equal to 0,50 MPa.

The open time is a direct consequence of the progressive reduction of ceramic tile adhesion with the time that elapses between the distribution of mortar over the surface and the ceramic tile application on the mortar. The reduction of the adhesion strength is a result of the increasing water loss of the mortar to its base and the atmosphere. The insufficient adhesion of the tiles can cause adhesion failure after short aging. In the case of façades, the fall of ceramic tiles are risks to users. However, this correlation is not normally evaluated, as the laboratories are limited to performing the tensile adhesion strength in the minutes that before and after the expected open time.

It is not possible to produce ceramic tile adhesive without admixtures, because the mortar would lose water very quickly, and the ceramic tile will not adhere on the base (PÓVOAS, 1999)[3]. Thus, water-retentive admixtures are used, for example, HEC (hydroxyethyl cellulose). Other polymers, like EVA (ethylene poly (vinyl acetate)), are also used to increase tensile adhesion strength.

Water retention is the main property of HEC. HEC is a cellulose derivative produced by etherifying some hydroxyl groups with alkali cellulose, and the alkali cellulose reacts with the ethylene oxide for the formation of HEC (UNION CARBIDE, [1997])[4]. In contact with water, HEC forms a gel that traps most of available water and increases the plasticity and cohesion of the dry-set mortar, reducing the slippage of tile on vertical or inclined installation. It is also an air-entraining admixture.

EVA, a synthetic and water-insoluble polymer, can be produced by the polymerization of vinyl acetate. It becomes water-soluble after hydrolyzed to yield PVA (poly (vinyl alcohol)) (SEYMOUR & CARRAHER, 1984)[5]. EVA increases flexibility and plasticity of the dry-set mortar. According to some producers (WACKER [1996])[6] the EVA powder admixture also has a weak water-retentive capacity because it contains a protective colloid derived from cellulose in its composition. EVA increases compressive, flexural and tensile adhesion strength, besides reducing the permeability of the mortar (BROCARD & CIRODDE, 1960).[7]

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[1] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. *Argamassa colante industrializada para assentamento de placas de cerâmica. Especificação* - NBR 14081. Rio de Janeiro, 1998.

— *Argamassa colante industrializada para assentamento de placas de cerâmica: substrato-padrão e aplicação de argamassa para ensaios.* Procedimento - NBR 14082. Rio de Janeiro, 1998.

— *Argamassa colante industrializada para assentamento de placas de cerâmica: determinação do tempo em aberto* - NBR 14083. Rio de Janeiro, 1998.

[2] COMITE EUROPÉEN DE NORMALISATION. *Adhesivos para baldosas cerámicas: requisitos mínimos* - CEN/TC 67. Milán, 1993.

– *Adhesivos para baldosas cerámicas: determinación del tiempo abierto* - CEN/TC 67. prEN C, n.62. Milano, 1993.

[3] PÓVOAS, Y.V. *Open time of dry set mortars: method for measurement and effect of HEC and EVA admixtures (Tempo em aberto da argamassa colante: método de medida e influência dos aditivos HEC e resina PVAc).* Escuela Politécnica, Universidad de São Paulo. São Paulo, 1999. p.154. Tesis de licenciatura en Ing. Civil (en portugués).

[4] UNION CARBIDE. *Cellosize - HEC - Versatilidade para a construção civil: aplicação em argamassas adesivas.* (Folder). [1997].

[5] SEYMOUR, R.B.; CARRAHER, C.E. *Structure-property relationships in polymers.* New York, Plenum Publishing Corporation, 1984.

[6] WACKER. *Vinyl Acetate Polymers - Vinnapas - Redispersible Powders.* (Folder). [1996].

[7] BROCARD, M; CIRODDE, M.R. *Utilisations des matières plastiques dans le gros oeuvre.* Annales de l'Institut Technique du bâtiment et des Travaux Publics. Série: Matériaux, n.156, p.1355-417, 1960.

The admixture influences the open time controlled by the dropping rate of tensile adhesion strength and the initial strength. The studied determination of the open time is based on the correlation between tensile adhesion strength and elapsed time of spread dry-set mortar, using L_1 regression.

2.. MATERIALS AND METHODS

In this paper the correlation among tensile adhesion strength and time elapsed between the distribution of the mortar over a surface and the ceramic tile application is studied. The correlation was evaluated by L_1 regression.

Mortars with a different content of HEC and EVA admixtures were produced in the laboratory. The mortar identified as HEC has 0,4 % of the weight of cement of HEC. EVA mortar has 10 % of the weight of cement of EVA powder and EVA + HEC has a combination of the two admixtures, 0,4 % and 10 %, respectively. The mix proportion was kept constant 1:3 (cement:sand, in weight) and w/c ratio was 0,80. As a control, one mortar acquired on the Brazilian market mortar was also studied.

2.1. CEMENT

The cement used in the formulation of the dry-set mortar is a Brazilian CP II-E, with ground granulated blast furnace slag content up to 30 %, as well as up to 10 % of calcium carbonate filler. Figure 1 presents its particle size distribution determined by laser granulometry. Table 2 presents its chemical analysis and Table 2 its physical analysis.

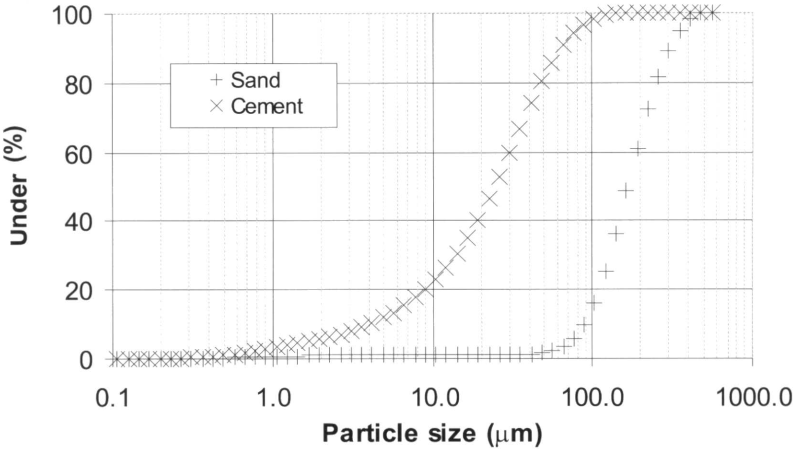

Figure 1. *Laser granulometry of Portland cement and sand.*

LOI	5,94	Na_2O	0,19
SiO_2	22,8	K_2O	0,48
Al_2O_3	7,35	S^{2-}	0,28
Fe_2O_3	2,16	Free CaO	1,54
CaO	56,2	Insoluble	1,19
MgO	2,72	CO_2	4,34
SO_3	2,01	$CaCO_3$	9,85

Table 1. *Chemical analysis of Portland cement (with values in %)*

# 325 NBR 9202[1] (%)	Blaine NBR 7224[2] (m^2/kg)	w/c (%)	Time of setting (min) NBR 11581[3]		Compressive strength (MPa) NBR 7215[4]			
			Initial	Final	1 day	3 days	7 days	28 days
9,0	390	27,8	177	284	9,0	21,0	28,7	37,8

1— ABNT. NBR 9202 - *Cimento Portland e outros materiais em pó - Determinação da finura por meio da peneira 0,044 mm* (número 325) - Método de ensaio. Rio de Janeiro, 1985.
2— NBR 7224 - *Cimento Portland e outros materiais em pó* - Determinação da área específica - Método de ensaio. Rio de Janeiro, 1984.
3— NBR 11581 - Cimento Portland - Determinação dos tempos de pega - Método de ensaio. Rio de Janeiro, 1991.
4— NBR 7215 - Cimento Portland - Determinação da resistência à compressão. Rio de Janeiro, 1996.

Table 2. *Physical and mechanical characterization of Portland Cement*

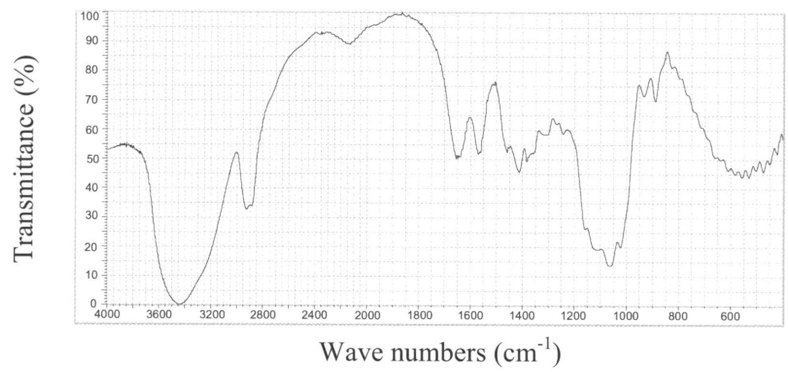

Figure 2. *HEC spectrogram.*

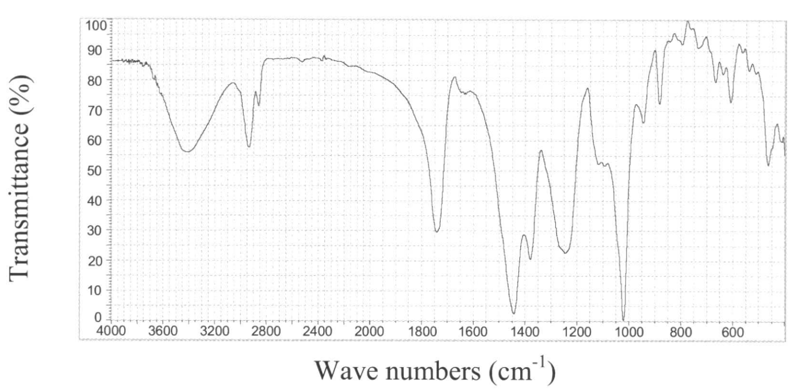

Figure 3. *EVA spectrogram*

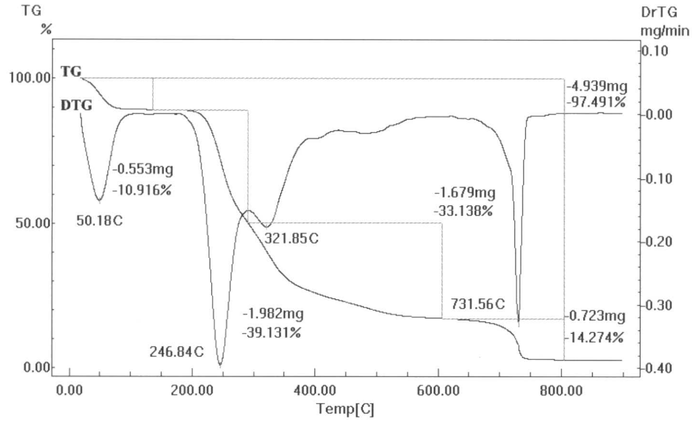

Figure 4. *HEC thermogravimetry result (TG)*

2.2. AGGREGATE

The quartz natural sand used in the formulation had a maximum diameter of 0,42 mm. Figure 1 shows its particle size distribution. The grain form is smooth and rounded, providing better workability.

2.3. ADMIXTURES

Both powder admixtures were obtained on the Brazilian market.

Figure 2 presents the infrared spectrogram of HEC. The analysis reveals that the main component was a cellulose-based structure. Figure 3 shows the infrared spectrogram of EVA, which reveals the presence of the poly (vinyl acetate) as well as another not identified phase, probably inorganic.

Figure 4 is the result of thermogravimetry of HEC. The first peak of the DTG curve at 50,2 °C, shows 10,9 % of humidity. The weight loss between 200 °C and 600 °C, with two peaks centred respectively at 246,8 °C and 321,8 °C, represents the oxidation of the organic phase, which was 72,3 % of the total sample weight. The peak at 731,6 °C presents the decomposition of the inorganic fraction. The inorganic fraction content can be estimated by the weight loss of its decomposition plus the residue at 1000 °C. The samples show an inorganic fraction content of 16,8 % of the total weight or about 18,9 % on a sample dry basis.

2.4. TENSILE ADHESION STRENGTH

The tensile adhesion strength of each mortar at time T was measured using (5 x 5) cm ceramic tiles with 16,8 % of total water absorption (NBR 13818[5]). The CEN/TC 67 (CEN, 1993) and NBR 14083 (ABNT, 1998) consider the ceramic tile water absorption (15 ± 3) % by mass. These ceramic tiles are applied over a standard concrete slab with water absorption at the surface after 4 hours in the range of 0,15 cm^3. This absorption was as specified by the Brazilian Standard NBR 14082 (ABNT, 1998) but is outside the CEN/TC 67-prEN B[6] specified range of 0,5-1,5 cm^3.

All sample preparation and curing was held in a room with (23 ± 2) °C, relative humidity of (60 ± 5) % and air speed lower than 0,2 m/s (NBR 14082 (ABNT, 1998)).

The mortar mixture procedure was the same as specified by CEN/TC 67-prEN C (CEN, 1993) and NBR 14082 (ABNT, 1998). After mixture, a thin layer of the adhesive was applied over the concrete slab surface using a straight edge trowel. Immediately afterwards a thicker layer was applied and combed with a notched trowel having (6 x 6) mm to produce a final layer of (5 ± 0,5) mm.

Ten tiles were pressed in the mortar, at a distance of 50 mm from each other, five minutes after the mortar application. The adhesion was improved by applying (2 ± 0,5) kg weight for 30 seconds. The operation was repeated at intervals of 5 minutes (10, 15, 20, 25 and 30 minutes or more).

5— ABNT. Placas cerâmicas para revestimento. Especificação e métodos de ensaio - NBR 13818. Rio de Janeiro, 1997.

6— CEN. Adhesivos para baldosas cerámicas: preparación de la placa de hormigón para el ensayo - CEN/TC 67. prEN B, n.61. Milán, 1993.

The tensile adhesion strength test was performed after 28 days of curing, using DYNATEST equipment, with a loading rate of (250 ± 50) N/s. The individual tensile adhesion strengths are determined to 0,1 N/mm^2.

3. DETERMINATION OF OPEN TIME BY REGRESSION

For the Brazilian Standard and CEN/TC 67 (CEN, 1993) methods, open time is the maximum time interval at which the adhesive meets the average adhesion $\bar{x} \geq 0,5$ MPa. This average is computed interactively by discarding all outlier values defined as those out of the range $\bar{x} \pm 0,2\bar{x}$. It is not usual have a coefficient of variation of 20 or even 30 %, which implies discarding up to 49 % of the results. If less than 5 results are left after discarding the outliers, the set is dumped. In other words, from a typical test with 40 or 60 adhesion points, only 5 or 6 are actually used as estimators of the open time. This increases the uncertainty of the estimation process.

On the other hand, the statistical regression approach allows the use of all data set to estimate the open time. Because L_1 regressions are more robust to disperse values, there is no need to expurgate outlying results, a procedure that is necessary in traditional regression. This makes the process easy and quicker.

The simple linear L_1 regression model is

$$_i = \beta_0 + \beta_1 T_i + \varepsilon_i \qquad (1)$$

Where: $_i = y_i$: tensile adhesion strength (MPa);

i = experimental result: 1, 2, ..., n;

β_0 : intercept parameter or linear coefficient (MPa);

β_1 : inclination parameter or angular coefficient (MPa/min);

$T_i = x_i$: time (min);

ε_i : random error, that is, the distance between the dispersion graph points and the adjusted linear regression straight line $[\varepsilon_i = {}_i - E({}_i)]$.

When estimating $_i$, the estimated value of ε_i is considered $E(\varepsilon_i) = 0$, giving

$$E({}_i) = E(\beta_0 + \beta_1 T_i) \qquad (2)$$

The estimators of the parameters $E(\beta_0)$ and $E(\beta_1)$ do not have an explicit formula; they are determined in such a way that the sum of absolute errors $\sum_{i=1}^{n} |\sigma_i - E(\sigma_i)|$ is minimum. The algorithm used in this research to estimate the parameters is presented in TAVARES (1998).[8]

After adjusting the linear equation, the parameters undergo the hypothesis test

H_1: $\beta_0 = 0$ e H_2: $\beta_1 = 0$

The significance level adopted in this research is $\alpha = 0,05$.

[8] TAVARES, R.A. *Seleção de variáveis em regressão L1. São Paulo,* 1998. p.82. Dissertação (Mestrado) – Instituto de Matemática e Estatística, Universidad de São Paulo.

A linear equation is adjusted for each mortar and presented together with the determination coefficient (CDM) (Table 3). The CDM with regard to the estimate robust method is similar to R^2 of the least squares method.

The open time will be estimated by the equation, the time $T_{0,5}$ that results on an adhesion strength of 0,5MPa

$$T_{0,5} = \frac{0{,}5-\beta_0}{\beta_0}$$

4. ADHESION STRENGTH VERSUS TIME

The studied mortars had the equations adjusted through L_1 linear regression (Table 3 and Figure 5).

Mortar	Linear Equation	CDM	Estimated open time (min)	
			Regression	NBR 14083
HEC	$_i = 0{,}70 - 0{,}0280\ T_i$	0,67	7,1	5
EVA	$_i = 1{,}37 - 0{,}0396\ T_i$	0,63	22,4	15
EVA + HEC	$_i = 1{,}26 - 0{,}0086\ T_i$	0,14	*87,9*[1]	*> 35*[2]
MARKET	$_i = 1{,}11 - 0{,}0086\ T_I$	0,36	*70,6*[1]	*> 45*[2]

[1] This is a forecast using the available data.

[2] The last ceramic tile application was done at this time.

Table 3. *Adhesion versus time estimated equations for the formulated mortars (i and β_o are in MPa and β_1 in MPa/min).*

The HEC equation (Table 3) shows the smaller value of the linear coefficient, that is, this mortar has the lowest initial ($T_i = 0$) tensile adhesion strength between the studied mortars. On the other hand, the EVA + HEC mortar showed the highest initial tensile adhesion strength, even above the MARKET one. The EVA mortar gave intermediate result.

The HEC angular coefficient (β_1) is smaller than the EVA mortar. These results show that the rate of adhesion strength loss of EVA is greater than the HEC mortar. However, the mortar with EVA has a greater open time than HEC due to its superior initial tensile adhesion strength. EVA mortar again gave intermediate results.

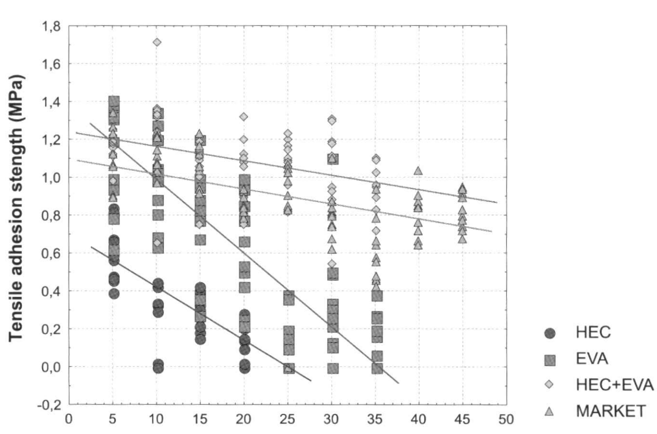

Figure 5. *Open time linear regression for the studied mortars.*

The open times estimated by regression and by NBR 14083 (ABNT, 1998) for HEC mortar are equivalent. The discrepancy between those values probably occurs only because in the standard procedure the open time is a discrete variable, varying by multiples of 5 minutes. The source of the discrepancy on both open times for EVA mortar is the standard procedure of discarding the outlier results. After discarding the 20 minutes results outliers, only four good results were left and the set of data was discarded. For the last two mortars, the last ceramic tile application was done at times that are inferior to their actual open times. The equations allow doing forecasts of the open time, but at this state of knowledge these results must be considered carefully. Indeed the open time results of the forecasts are in complete agreement with the results of the standard procedure.

It is possible to conclude that the mortar with only HEC admixture is not capable of providing great open times, because although it increases the water retention, expressed by a low angular coefficient, its effect in the initial strength is limited. The EVA admixture alone does not significantly improve water retention but it improves the open time because it increases the initial adhesion strength. The combination of both admixtures gives better results in term of open time and initial adhesion strength.

5. CONCLUSIONS

A significant linear correlation among tensile adhesion strength and time between the spreading of the mortar and the ceramic tile application was found for the set of studied mortars. Further investigations are required to confirm the linear correlation.

In this model the open time is controlled by two factors: (a) initial strength and (b) rate of tensile adhesion strength loss.

It was also concluded that both admixtures affect the period of open time, but in different ways. HEC increases the open time because it reduces the rate of tensile adhesion strength loss. It is probably due to its water retention capacity. EVA, in spite of its small water retention capacity, yielded high open time, because this admixture increases the initial tensile adhesion strength.

The estimation of the open time by regression analysis is a simple and quicker procedure that uses all the available data, and allows better comparative analysis among different ceramic tile adhesives. In general, the results of open time estimated by regression are in agreement with those obtained by standard procedure.

6. REFERENCES

— AMERICAN NATIONAL STANDARD SPECIFICATIONS. *American National Standard Specifications for dry-set Portland cement mortar* - ANSI 118.1 - 1992. Tile Council of America, 1992.

— BRITISH STANDARD INSTITUTION. *Adhesives for use with ceramic tiles and mosaics* - BS 5980:1980. London, 1989.

— UNION EUROPÉENNE POUR L'AGRÉMENT TECHNIQUE DANS LA CONSTRUCTION. *Guía Técnica UEAtc para la evaluación de Colas para Revestimientos Cerámicos.* Madrid, ICCET, n.403, 1990.

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