INFLUENCE OF SETTING TEMPERATURE ON THE ADHESION STRENGTH OF CERAMIC TILE ADHESIVES

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ABSTRACT

The influence of temperature on the setting of cementitious binders is well known, hence the manufacturers' recommendations regarding the limit temperatures for use.

The present study, conducted by students of the Universitat Jaume I Ceramics Classroom, seeks to evaluate the change in pull-off strength of different types of adhesives in which different setting conditions were used: an ideal situation of 20ºC, a situation in slightly cold weather at 5ºC, and one in warm weather at 40ºC.

The test method used was the method defined in standard UNE EN 12004, however, subjecting the test pieces during the first 14 days to the above temperatures, and the following 14 days to laboratory temperature.

The test with adhesives Ci, C1, and C2, made by different manufacturers, allowed their performance under the above situations to be verified, and thus the appropriateness to be verified of each type of adhesive for external uses, as set out in the Ceramic Tile Guide.

1. INTRODUCTION AND BACKGROUND

In June 2006, a collaboration agreement was made between Universitat Jaume I and ASCER (the Spanish Ceramic Tile Manufacturers' Association), in which, under the name Ceramics Classroom, an experience is realised that is already effective in other universities under the name Ceramics Chair. The objectives of this Ceramics Classroom are to better understand the ceramic materials used in coverings and their installation techniques, in order to enhance the quality of these materials in service, by further training of the future technicians responsible for their specification and installation control.

For this purpose, a free-choice subject has been organised, called Ceramic Covering Materials. The subject has a theoretical part, which is delivered in the course of 14 lectures, and a practical part, in which students carry out a research assignment or documentary compilation assignment on some specific aspect of ceramics.

This communication is the result of one of these assignments by students on cementitious adhesives used in ceramic tile installation.

2. OBJECTIVES

According to standard UNE-EN 12004/2008: *Adhesives for ceramic tiles. Requirements, conformity evaluation, classification and designation*, the adhesives used for ceramic tile installation are divided into three types, based on their chemical composition, and into several classes as a function of their characteristics.

Types of adhesive	Classes
Cementitious adhesives **(C)** Dispersion adhesives **(D)** Reactive resin adhesives **(R)**	**1** Normal adhesive **2** Improved adhesive **F** fast setting **T** with reduced slipping **E** cwith longer open time **S** deformable

Table 1.

There are also other adhesives, which are not included in the standard, but which conform to the minimum requirements set out in Annex ZA-UNE EN 12004, whose scope is restricted only to tile installations in interiors. These adhesives are designated Ci.

From a tile installation standpoint, one of the most important adhesive characteristics is its adhesion strength. This may be determined by verifying adhesive pull-off strength. The recommended test method, according to the

above standard, is described in *UNE-EN 1348/2008: Adhesives for ceramic tiles. Determination of the tensile adhesion strength of cementitious adhesives.*

The adhesion strength requirements for the different classes of cementitious adhesives are detailed in the following table:

	Ci	C1	C2	Ud	Test
Initial adhesion	≥0.5	≥0.5	≥1.0	N/mm²	EN 1348
Adhesion after immersion in water	≥0.5	≥0.5	≥1.0	N/mm²	EN 1348
Adhesion after heat action		≥0.5	≥1.0	N/mm²	EN 1348
Adhesion after heat action		≥0.5	≥1.0	N/mm²	EN 1348
Open time after 20 min		≥0.5	≥0.5	N/mm²	EN 1348

Table 2.

This paper focuses solely on the study of cementitious adhesives and the variation of their adhesion strength with temperature. The following objectives were targeted:

- To study adhesion performance with various setting and hardening temperatures for different types of commercial cementitious adhesives.
- To compare the performance of each type of adhesive produced by different commercial manufacturers.

3. METHODOLOGY

The work methodology is based on the determination of adhesion strength using a method set out in standard *UNE-EN 1348/2008: Adhesives for ceramic tiles. Determination of the tensile adhesion strength of cementitious adhesives*, mentioned above. Some details have been adapted or modified in order to address the intended objectives.

3.1. Adhesives.

The following table details the adhesives chosen for the tests: seven adhesives were selected, from two different manufacturers. The table shows the type of adhesive, a letter indicating the manufacturer, the indications for use and constraints for each according to its manufacturer and, finally, the reference code used in the study.

ADH	MANU	MANUFACTURER'S INDICATIONS	CONSTRAINTS ON USE	REF
Ci	P	Internal cladding and flooring. Tiles with medium and high absorption (>3%)	Tiles with low absorption (<3%) and gypsum substrates	Ci/P
Ci	P	Internal cladding. Tiles with medium and high absorption (>3%) on a gypsum substrate	Tiles with low absorption (<3%)	Ciy/P
C1TE	P	Internal cladding and internal and external flooring. Special swimming pools, appropriate on gypsum wallboard	Tiles with low absorption (<3%)	C1TEy/P
C1TE	P	Internal cladding and internal and external flooring. Especially for tiles with low absorption (<3%)	Substrate with radiant heating	C1TE/P
C1T	K	Internal and external cladding and flooring	Non-cementitious substrates	C1T/K
C2TES1	P	Internal and external cladding and flooring. Especially for façades	Polyester substrates	C2TES1/P
C2E	K	Internal and external cladding and flooring. Especially for porcelain tile and façades with large sizes (also natural stone)		C2E/K

Table 4.

3.2. Test pieces.

To make the test pieces to which cementitious mortar was to be applied, BIIa type glazed ceramic tiles with a smooth finish were chosen.

The dimensions of these test pieces were chosen such that they allowed the metal disk that was to be pulled to be installed. As a result, test piece dimensions were 60 mm x 60 mm.

Four test pieces were prepared for each adhesive and temperature.

3.3. Substrate.

With a view of having an adhesive contact surface as similar as possible to that of one on a building site, cement mortar tiles (terrazzo) were used on the back, as the base substrate on which to fix the ceramic tile test pieces.

3.4. Experimental procedure.

The tests consisted of various phases, which are described below:

3.4.1. Preparation of the adhesive, installation of the test pieces, and adhesive setting.

Each of the adhesives described in the previous section was subjected to three different setting temperature conditions:

- Warm conditions: 40ºC.
- Ideal conditions: 22ºC.
- Slightly cold weather conditions: 5ºC.

These conditions were obtained by means of an oven (40ºC), a laboratory environment (20ºC), and with a large chest freezer (5ºC), the conditions being held for 14 days, at which time setting was considered to have ended.

A sample of 0.5 kg each of the cementitious adhesives was used for mixing, at each of the three programmed temperatures.

The amount of water used for mixing was the quantity indicated in each case by the manufacturer.

Mixing was carried out in a mixer that allowed various mixing speeds and which, in addition to the normal rotation, also performed a planetary movement.

The procedure used for mixing is schematically illustrated below:

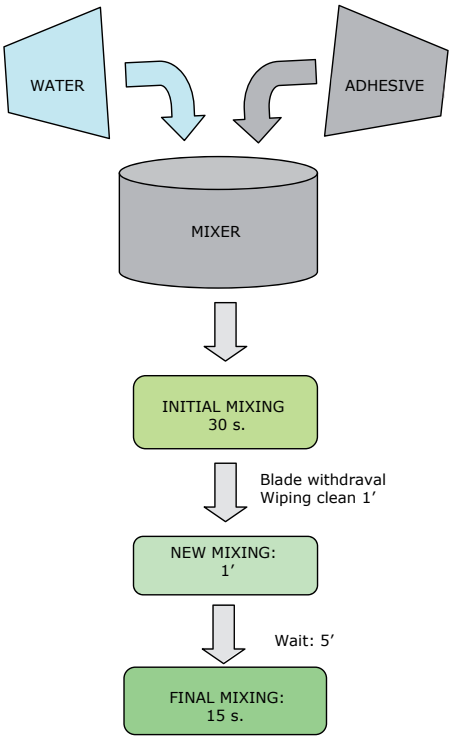


Figure 1.

After the adhesive had been mixed, a thin bed was applied on to the mortar base, with a straight-edge trowel. A thicker layer was then applied and combed with a notched trowel. The trowel must form an angle of about 60º with the substrate, at right angles with the edge of the slab, and move parallel to that edge (in a straight line).

A 5-min wait followed and the four test pieces were then installed, leaving sufficient space between them to be able to fit the pull-off test accessories.

The substrate with the test pieces was then left for 14 days under the corresponding test conditions: in an oven, in the laboratory environment, or in a large chest freezer.

Figures 2,3. Fixing of the test pieces on the substrate.

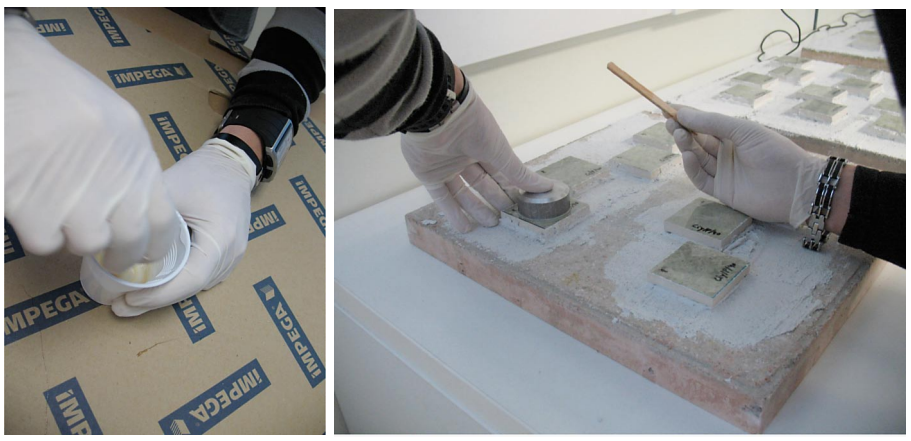
3.4.2. Second drying phase.

After the 14-day setting phase, all the samples were left under laboratory conditions for the following, metal disk-adhering phase.

3.4.3. Disk adhering.

Twenty-seven days after the beginning of the test, the metal disks were adhered using a high-strength two-component epoxy resin.

A screw was coupled to these disks, to which the pull-off devices used for measuring the tensile adhesion strength fitted.



Figures 4,5. Resin preparation and adhering of the metal disks.

3.4.4. Pull-off test.

The tensile adhesion strength test was conducted 24 h after the metal disks had been adhered, ensuring the most uniform possible tensile force rate.

The results were recorded in newton.

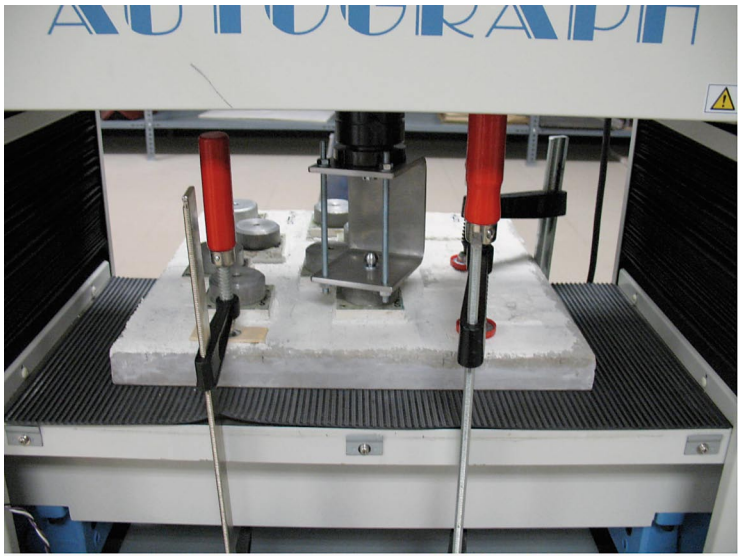


Figure 6. Tensile adhesion strength test.

3.5. Expression of the results.

The values for each tensile adhesion strength test were determined from the following expression:

$$A_s = \frac{L}{A}$$

Equation 1.

where

As is the individual tensile adhesion strength (N/mm^2);

L is the total load (N);

A is the adhesion surface in square millimetres (3,600 mm^2).

Tensile adhesion strength was calculated for each type of adhesive and manufacturer, the arithmetic mean being calculated of the individual values (the values that differed more than 20% from this average value were considered outliers). The mean thus obtained was considered the characteristic tensile adhesion strength of the relevant product.

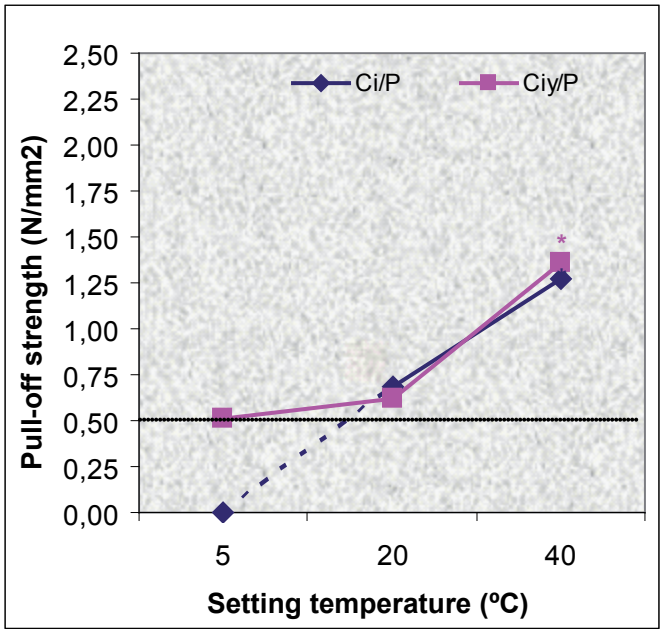
4. ANALYSIS OF THE RESULTS

After the tensile adhesion strength of each adhesive had been calculated, the results, **grouped by type of product**, were plotted to compare them to each other and to draw conclusions.

The results obtained are displayed below:

TYPE Ci ADHESIVES

Temp. ºC	Tension (N/mm²)			
	Ci/P		Ciy/P	
40ºC	1.39	*	1.39	*
	1.26		1.39	*
	1.39	*	1.28	
	1.03		1.39	*
20ºC	0.94		0.50	
	0.54		0.82	
	0.61		0.61	
	0.65		0.53	
5ºC	0.00	**	0.00	**
	0.00	**	0.00	**
	0.00	**	0.00	**
	0.00	**	0.52	**



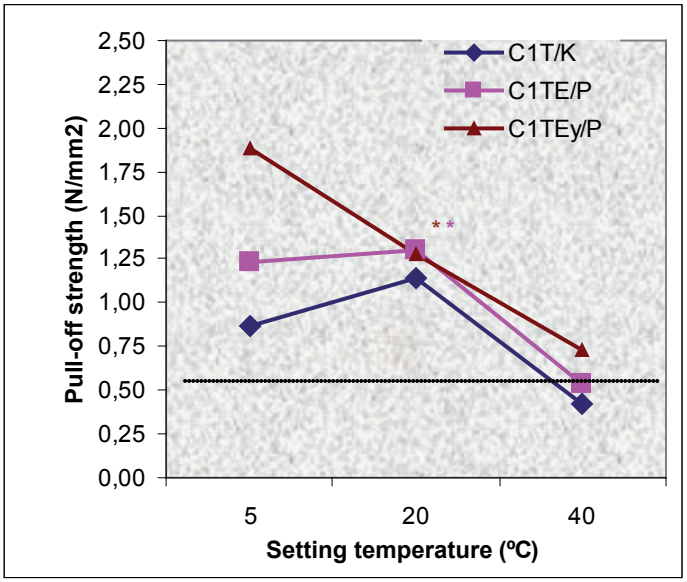
** Limit values of the test device.*
*** Null tests / no strength*
Table 5.

Observations:

- At the setting temperature of 5ºC, the test pieces Ciy/P were not adhered to the substrate, which is why no result is given for their pull-off strength.
- Better tensile adhesion strength performance was obtained at high setting temperatures for both products.

TYPE C1 ADHESIVES

Temp. ºC	Tension (N/mm²)					
		C1TE/P		C1TEy/P		
40ºC	0.17	0.25		0.47		
	0,15	0.33		0.58		
	0,28	0.46		0.46		
	1,08	1.11		1.39	*	
20ºC	1.22	1.01		1.39	*	
	1.22	1.39	*	1.39	*	
	0.72	1.39	*	0.96		
	1.39	1.39	*	1.39	*	
5ºC	0.90	1.60		1.89		
	1.02	0.79		0.00	**	
	0.92	0.91		0.00	**	
	0.63	1.63		1.86		



** Limit values of the test device. ** Null tests / no strength.*

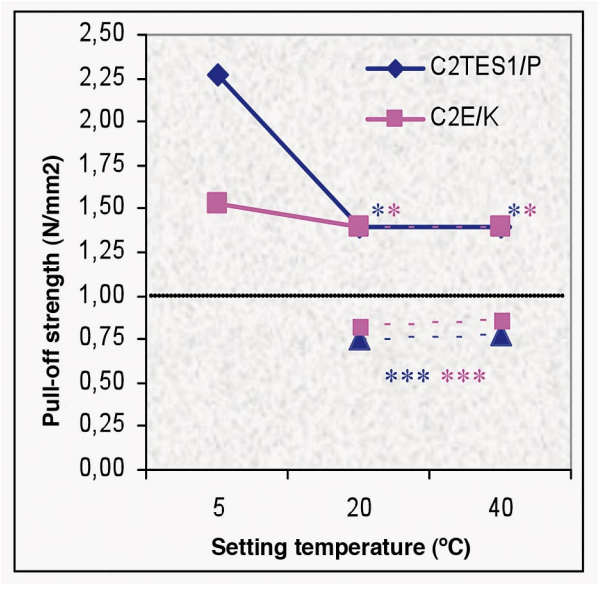
Table 6.

Observations:

- For two of the adhesives, it may be observed that the conditions that provide the best pull-off strength results are those of the intermediate setting temperature.
- Although the third product (C1Tey/P) exhibits a higher average value at the 5ºC temperature, it should be noted that in this situation, two samples provided zero values, which indicates the poor reliability of the average value. This adhesive is indicated by the manufacturer for use in swimming pools.

TYPE C2 ADHESIVES

Temp. ºC	Tension (N/mm²)					
	C2TES1/P		***	C2E/K		***
40ºC	1.39	*	0.82	1.39	*	0.93
	1.39	*	0.69	1.39	*	0.90
	1.39	*	0.79	1.39	*	0.83
	1.39	*	0.81	1.39	*	0.79
20ºC	1.39	*	0.80	1.39	*	0.56
	1.39	*	0.73	1.39	*	1.01
	1.39	*	0.85	1.39	*	0.90
	1.39	*	0.64	1.39	*	0.84
5ºC	0.00	**		0.00	**	
	2.34			1.30		
	0.00	**		0.00	**	
	2.19			1.75		



** Limit values of the test device. ** Null tests / no strength. *** Pull-off strength with water-saturated test pieces.*

Table 7.

Observations:

- It was not possible to determine the tensile adhesion strength of these adhesives at 20 and 40ºC setting temperatures; it may be noted, however, that the pull-off strength exceeded 1,39N/mm^2 (limit of the pull-off strength tester). However, for both adhesives, the pull-off strength of the water-saturated samples was about 0,75 and 0,85 N/mm^2, which is below that required by the standard.

5. COMPLEMENTARY TESTS

5.1. Immersion in water.

Some of the adhesive test pieces of the C2 type were immersed for 24 hours in water at room temperature, after which the tensile adhesion strength test was conducted. The purpose of this test was to establish possible changes in the pull-off strength of the cementitious adhesives.

A reduction in the tensile adhesion strength was observed, independently of the temperature at which setting had occurred. The same performance was evidenced for both manufacturers.

Figure 7. Immersion in water.

5.2. Modification of open time with temperature.

A last test was conducted to verify the possible reduction in the open time of the adhesives, under forced drying conditions (the substrate with the adhesive being subjected to a continuous stream of hot air).

For this purpose, the installation of the test pieces on the substrate was performed at three different times: immediately after mixing the adhesive and spreading it on the substrate, at half the open time, and at the end of the open time fixed by each manufacturer.

After one hour, the pull-off strength of the test pieces was qualitatively verified.

The findings matched the expected results:

- No adhesive kept its adhesion strength when the samples were installed at the end of the open time.
- Only some of the C2 adhesive samples were able to achieve good adhesion strength when they were installed at half the open time. Adhesives Ci and C1 were unable to achieve adhesion under these conditions.
- All test pieces installed after mixing displayed good adhesion capability.

6. CONCLUSIONS

The following conclusions may be drawn from the study:

- Under ideal conditions, with a perfect installation, temperature variations during the setting and hardening phases of adhesives influence their final strength. It is therefore of great importance to observe the temperatures for use indicated by the manufacturers for their products.
- The detected strength variations under these conditions are influenced by the class of adhesive, their additional characteristics, and the manufacturer, which is why valid conclusions cannot be extrapolated for all adhesives.
- A smaller influence on the final strength of the C2 adhesive is noted, which, in all cases, yields higher results than those specified by the standard.
- The results obtained suggest that Ci adhesives behave differently from C1 and C2 adhesives under heat situations, which may be due to a smaller binder content in the Ci adhesives, which enhances its performance under heat.
- As is well known, and envisaged as such by the standard, the presence of water in adhesives significantly reduces adhesion strength.
- Under extreme environmental conditions (high temperatures or wind), it is essential to reduce application times as much as possible, because adhesion strength diminishes when the application time is prolonged.

ACKNOWLEDGEMENTS

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