FIXING OF STONEWARE "PORCELANIC" TILES BY MEANS OF CERAMIC TILE ADHESIVES MODIFIED WITH REDISPERSIBLE POLYMER POWDER

Dr Hardy Herold

Technical Service Construction
Polymer Division
L-B-E-T5/WB
Wacker Polymer Systems GmbH & Co. KG
Burghausen plant
D-84489 Burghausen
Phone + 49-8677- 83 53 98
Fax + 49-8677- 83 50 90

ABSTRACT

Traditionally, tile adhesives were simple Portland cement mortars which were applied using the so-called thick-bed method, eg, the mortar was first applied (buttered) on the back of the (prewetted) tiles to a thickness of 10 to 25 mm, and the tiles were then pressed into position on the wall or substrate.

This method was fairly successful for earthenware tiles, which have a high water-absorption capacity. The pure cement lime was able to penetrate to a certain extent into the tile's surface, thus forming an anchorage between tile and adhesive.

However, this mechanism does not apply to modern ceramic tiles (stoneware tiles, "porcelanic tiles"), which absorb very little water. On account of their low water uptake, they offer higher abrasion resistance and increased durability, and are increasingly being used instead of earthenware tiles.

With stoneware tiles, only modern ceramic tile adhesives (so-called thin-bed mortars) modified with redispersible polymer powders are able to guarantee durable adhesion to the substrates.

Redispersible polymer powders improve the adhesion properties as well as flexibility and workability. They work by way of forming "resin-domains" within the mortar as well as between the mortar and the surfaces of substrate and tile. The adhesion mechanism is thus different from that of non-modified mortars.

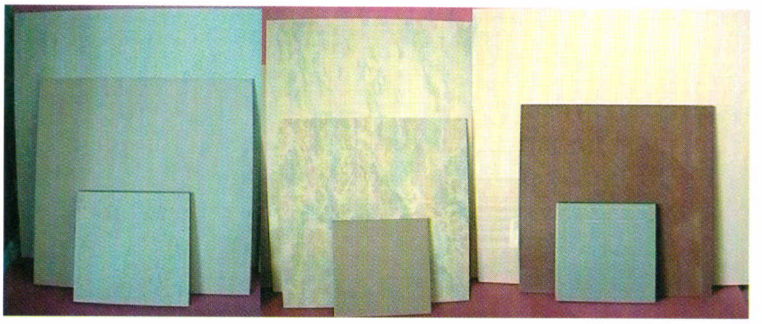

Figure 1. Spanish ("porcelánico") Tiles.

INTRODUCTION

The problems connected with the laying of stoneware tiles with water-absorption values of less than 0.5 % by weight have been pointed out repeatedly in various scientific studies and by well-known manufacturers of tile adhesives. Accordingly, manufacturers offer special "flexible" tile adhesives (deformable, polymer-modified tile adhesives) for such tiles.

Spain has a world-wide reputation for the excellence of its high-quality tiles, which explains why Spanish tile manufacturers succeed in exporting a substantial proportion of their products. This study deals with the laying of "porcelánico" tiles from three well-known Spanish manufacturers (Figure 1) with polymer-modified tile adhesives.

It quickly became evident that tile grades of this kind, which absorb even less water (practically none) than "normal" stoneware tiles, place extremely high demands on the tile-laying materials.

STANDARDIZATION OF TILE ADHESIVES IN EUROPE

The European Committee for Standardization has agreed on the DIN EN 12004 [1] for the standardization in Europe of materials used for laying ceramic floor coverings. This Standard, which is currently still in draft form, regulates the requirements and classification of tile adhesives. Other Standards are cited for the corresponding test methods. The new Standard differs from previous tile adhesive Standards in that cementitious tile adhesives are now categorized in two groups: standard adhesives (C1), and adhesives for special requirements (C2). In addition, the term "open time" has been redefined as the tensile adhesive strength after 28 days' conditioning under standard climatic conditions, the tiles having been bedded on a concrete substrate at various intervals from the time the adhesive was applied [2].

Some of the requirements prescribed in the Standard are summarized in the following table.

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[1] prEN 12004, *Adhesives for Tiles, Definitions and Specifications* (November 1995).

[2] DIN EN 1346, *Adhesives for Tiles, Determination of Open Time* (March 1997).

EN 12004: Adhesives for Tiles, Definitions and Specifications

Demands for class C1:

Tensile adhesion strength

- 28 days (d) standard conditions (sc)	≥ 0,5 N/mm²	initial strength
- 7 d sc + 21 d under water	≥ 0,5 N/mm²	water immersion
- 14 d sc + 14 d at 70°C + 1 d sc	≥ 0,5 N/mm²	heat ageing
- 7 d sc + 21 d under water + 25 cycles freeze/thaw	≥ 0,5 N/mm²	freeze thaw cycles
Open time (tensile adhesion strength)	≥ 0,5 N/mm² ≥ 20 min. (waiting time)	

Demands for class C2:

Tensile adhesion strength

- 28 days (d) standard conditions (sc)	≥ 1 N/mm²	initial strength
- 7 d sc + 21 d under water	≥ 1 N/mm²	water immersion
- 14 d sc + 14 d at 70°C + 1 d sc	≥ 1 N/mm²	heat ageing
- 7 d sc + 21 d under water + 25 cycles freeze/thaw	≥ 1 N/mm²	freeze thaw cycles
Open time (tensile adhesion strength)	≥ 0,5 N/mm² ≥ 20 min. (waiting time)	

Table 1

BONDING OF TILES WITH EXTREMELY LOW WATER UPTAKE («PORCELANIC» TILES)

The test results obtained for the laying of Spanish "Porcelánico" tiles are given below.

To investigate the influence of cellulose ethers and redispersible powders on the important properties of tile adhesives modified in this way, a formulation comprising 35 % Portland cement was used (Table 2).

Tile adhesive test formulation		
35	w t %	Portland cement CEM 132.5 R
64.6	w t %	Quartz sand (< 0.5 m m)
0.4	w t %	Cellulose ether

Table 2

INFLUENCE OF CELLULOSE ETHERS

Cellulose ethers are used in tile adhesives in order to confer sufficient water retentivity on them. This is necessary to ensure that the mixing water is available to the tile adhesive long enough for most of the adhesive to hydrate. Hydration of the tile adhesive is a prerequisite for strong bonding. High water retentivity also ensures prolonged wetting capability of the adhesive once it has been combed onto the substrate (tilers still refer to this period as the "open time"). Especially at high temperatures or in windy conditions, the reliability of the bond is increased if the adhesive has prolonged wetting power, because once the adhesive starts to form a skin, the surface area which is actually bonded decreases. An additional function of cellulose ethers is to enhance the tile adhesive's non-slump property.

Today, manufacturers of tile adhesives have a broad range of highly different cellulose ether grades to choose from. These differ in their molecular weights (viscosities) and a large number of chemical substitutions (types and degrees of etherification). Modern cellulose ethers generally fall under the group of methylhydroxyethylcellulose ethers (MHEC) or methylhydroxypropylcellulose ethers (MHPC).

In this study, the influence of cellulose ethers modified to different extents was examined (Table 3).

Cellulose ethers used

	Viscosity of the 2% solution	Degree of etherification	Consistency-enhancing modification
A	45,000 (MHEC)	high	+++
B	5,000-6,000 (MHPC)	high	++
C	45,000-52000 (MHEC)	high	+

Table 3

To test the slip resistance (non-slump property) of tile adhesives, the test setup shown in Figure 2 was used in place of the one stipulated in the corresponding Standard [3]. The setup used made it possible to quantify the slip resistance by weighing down the tile. The slip resistance is defined as the weight under which the test tile (on a fine-stoneware substrate) still just resists slipping. The test itself is conducted in accordance with the Standard [3].

The wetting properties were determined on the basis of DIN EN 1347 [4], except that stoneware tiles measuring 5 x 5 cm_ were used, which were weighed down in each case for 30 seconds with a 2-kg weight.

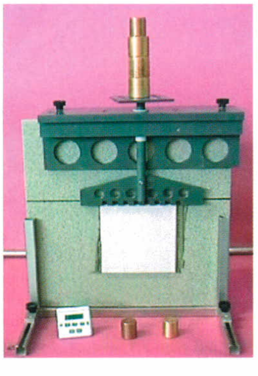

***Figure 2.** Determination of non-slump-properties of Ceramic Tile Adhesives*

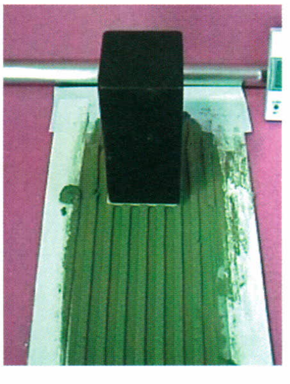

***Figure 3.** Determination of Wetting-properties of Ceramic Tile Adhesives*

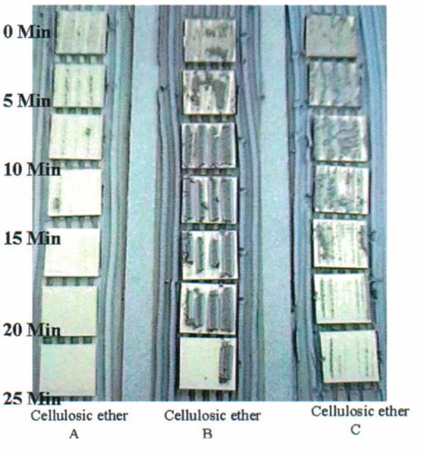

***Figure 4.** Determination of Wetting-properties of Ceramic Tile Adhesives*

[3] DIN EN 1308, *Adhesives for Tiles, Determination of Slip* (March 1997).

[4] DIN EN 1347, *Adhesives for Tiles, Determination of Wetting Capability* (March 1997).

The results obtained in these tests are presented in Figure 4 and Table 4. As is clearly evident, good slip resistance frequently comes at the expense of relatively fast skin formation (the tests were conducted without the addition of redispersible powder - using the same amount of water).

The results also show how important it is to choose a cellulose ether which will guarantee the required product quality.

Non-slump property as in Figure 2	
Cellulose ether	**Slip resistance up to**
A	1800 g
B	1000 g
C	800 g

Table 4.

ADDITION OF REDISPERSIBLE POWDERS

Redispersible powders are spray-dried dispersions, which can be added easily to ready-mixed mortars in order to enhance their properties - see [5] and [6]. When the mortar is mixed with water, the dispersion is reconstituted and acts in the same way as the aqueous dispersions that used to be added to mortars. Compared with these earlier dispersions, however, redispersible powders do not have the disadvantages of frost sensitivity, difficult storage, high transport costs, etc. Powder binders have now been available for over 40 years, and thus represent a standard method of modifying dry mortars in the building industry. For the tests described here, three grades of redispersible powder were used, which differ in their glass transition temperature ("softness") (Table 5).

Used Redispersible powders

	type of polymer	glass transition temperature	rheological behaviour Hydrophobic effect
1	V a c /E	- 7 °C	neutral, -
2	VC / E / VL	2 °C	neutral, hidrophobic
3	V A c / E	16 °C	neutral, -

Table 5

[5] J. Schulze, *Redispersionspulver im Zement*, Tonindustriezeitung no. 9, 1985.
[6] K. Adler, *Kunststoff und Zement - eine ideale Verbindung*, Applica 1988, p. 10.

OPEN TIME

It is fairly obvious that tile adhesives characterized by rapid skin formation (eg, grade A in Figure 4) will show inadequate tensile adhesive strength just a short time after being combed onto the substrate. In other words: the tiler must press the tiles very quickly into the fresh bed of adhesive. He can only apply adhesive to a very small area of substrate, otherwise he will be taking too large a risk. This risk can be reduced considerably by the addition of suitable redispersible powders. These improve the capability of the tile adhesive to wet the tiles, and hugely increase the tensile adhesive strength, even if the surface area wetted is small (this will increase the tiler's confidence - who, after all, is judged by the quality of his work - and will enable him to work more quickly). The reason for the improved properties lies in the binder character of the redispersible polymer powder, which substantially increases the tensile strength of the cementitious matrix.

The standards committee for the standardization of tile adhesives recognized the problems involved, and defined the open time on the basis of the adhesive tensile strength after 28 days' conditioning under standard climatic conditions, the tiles having been bedded on a concrete substrate at various intervals (see Figure 3) from the time the adhesive was applied [2].

Figure 5 shows how strongly polymer modification influences the open time.

Here, the open time for the test formulation used (addition of 0.4 % cellulose ether A in each case) was determined as a function of the amount of soft redispersible powder 1 added. The non-modified tile adhesive had an open time of less than ten minutes, which is much too short. High-quality "Porcelanic" tiles, which absorb practically no water, cannot be bonded sufficiently securely without the addition of redispersible powder. To obtain an open time of 20 minutes here, as is prescribed in Standard [1], approximately 5 % powder grade 1 needs to be added.

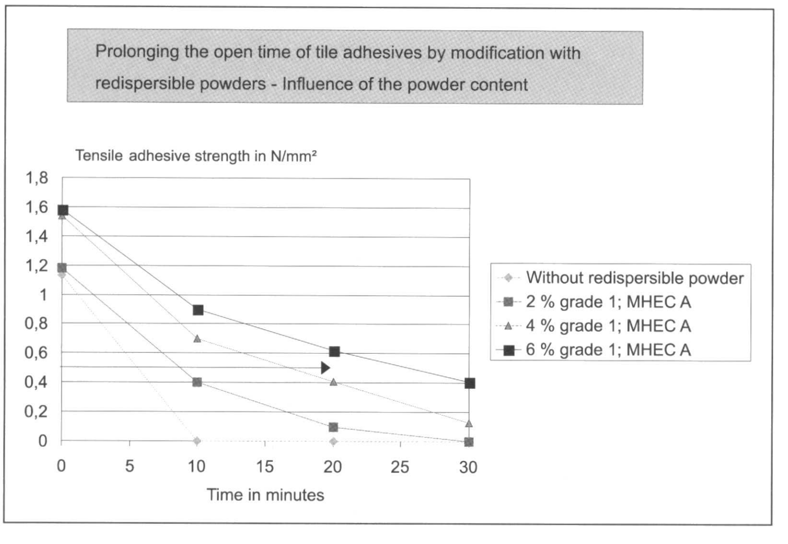

Figure 5

The open time determined in this way can, of course, be influenced by using different grades of redispersible powder.

[1] prEN 12004, *Adhesives for Tiles, Definitions and Specifications* (November 1995).
[2] DIN EN 1346, *Adhesives for Tiles, Determination of Open Time* (March 1997).

In Figure 6, 6 % of the redispersible powder grades 1 to 3 was used in each case, together with 0.4 % cellulose ether B. With the soft, grade 1 powder, the minimum required tensile adhesive strength of 0.5 N/mm_ was not reached after a bedding interval of 30 minutes. With the grade 3 powder, this value was just reached. With the hard, grade 2 powder, the open time is clearly over 30 minutes.

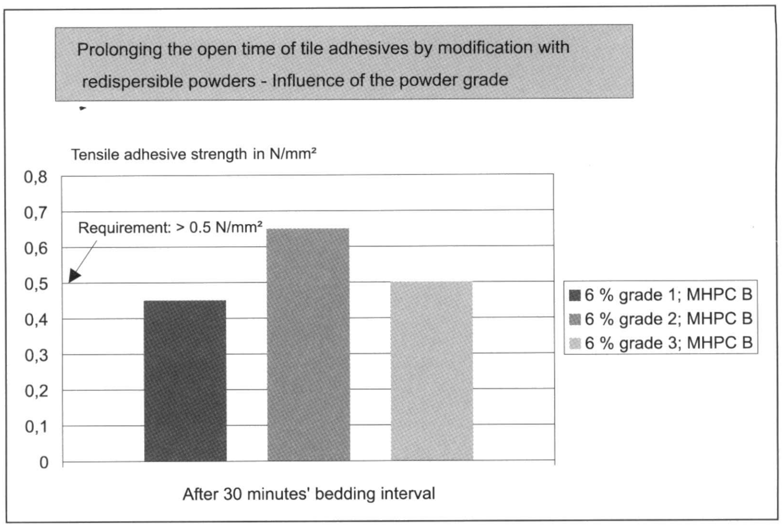

Figure 6

TENSILE ADHESIVE STRENGTH AFTER HEAT CONDITIONING

The properties of a tile adhesive after immersion in water and exposure to alternating freeze-thaw conditions will depend predominantly on the properties and amount of the cement used. Under these "wet" conditions the cement is generally able to hydrate sufficiently and develop the required strength. The addition of redispersible powders has only a minor influence in this case - see [7].

High temperatures, by contrast, such as are encountered in warmer climatic regions and on the exterior of facades and on balconies and patios in summer, have a crucial effect on the tensile adhesive strength of tile adhesives. The CEN standard takes this into account by prescribing the determination of the tensile adhesive strength after heat conditioning at 70 °C. High temperatures are critical because they cause both the tile adhesive and the substrate to dry out excessively. There is increased shrinkage of the substrate, in particular, requiring the tile adhesive to show greater flexibility. An additional problem, of course, is that the mineral cement binder is not able to hydrate to the same extent that it would under standard climatic conditions. The organic redispersible powder has to take over the cement's role of binder.

As shown in Figure 7, a non-modified tile adhesive is totally unable to bond the high-quality tiles used in the study following heat conditioning at 70 °C. Only after the addition of redispersible powder is it possible to bond such tiles securely. Here too, a harder powder (powder grade 2) generally produces higher tensile adhesive strengths than does a softer powder (powder grade 1).

[7] H. HEROLD, *Modification of Ceramic Tile adhesives with Redispersible Polymer* Powders, Qualicer 98, Castellon/Spain 8 / 11 March 1998, Vol. II, p. 191.

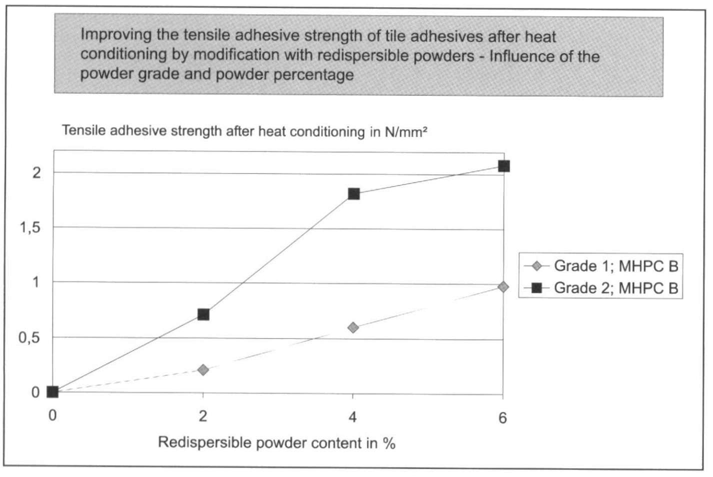

Figure 7

Figure 8

FLEXIBILITY

The importance of a tile adhesive's flexibility, or deformability, has already been pointed out in the above section ("Tensile adhesive strength after heat conditioning").

There are various methods of determining the flexibility of this kind of adhesive mortar:

According to the CEN Standard [9], flexibility is determined by means of the so-called strip bending test, which is performed on a 3-mm-thick strip of tile adhesive (see Figure 8).

The degree of deflection undergone by a mortar strip prior to fracture in this experiment is a measure of its flexibility. Normally, the amount of deflection is only measured at 23 °C, after the strips have been conditioned for 14 days at standard temperature and > 95 % relative humidity, followed by 14 days in a standard climate. However, we consider it equally important for a tile adhesive to retain sufficient flexibility at low temperatures, too.

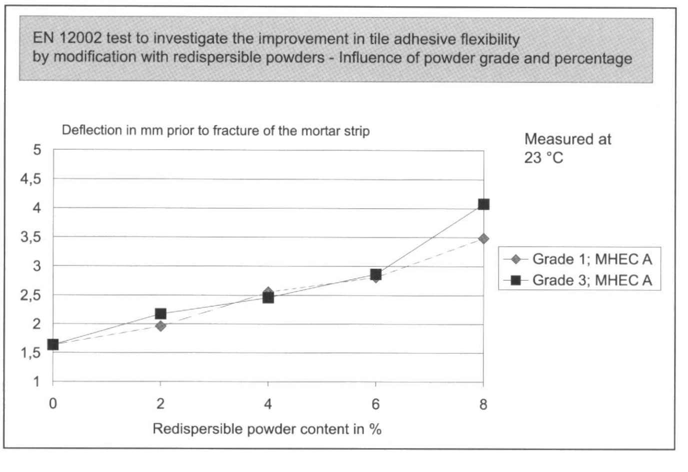

Figure 9

[9] DIN EN 12002, *Adhesives for Tiles, Determination of Deformability* (August 1997).

As Figure 9 shows, the flexibility of tile adhesives can be improved significantly by modifying them with redispersible powders. When measurements are made at room temperature, the flexibility is comparable for hard and soft powder grades, somewhat higher values being obtained for the harder grade 3 powder.

This is not the case when measurements are performed at -10 °C. (Figure 10). The advantages of soft, flexible redispersible powder grades now become clearly apparent. The soft powder A confers much greater flexibility on the tile adhesive.

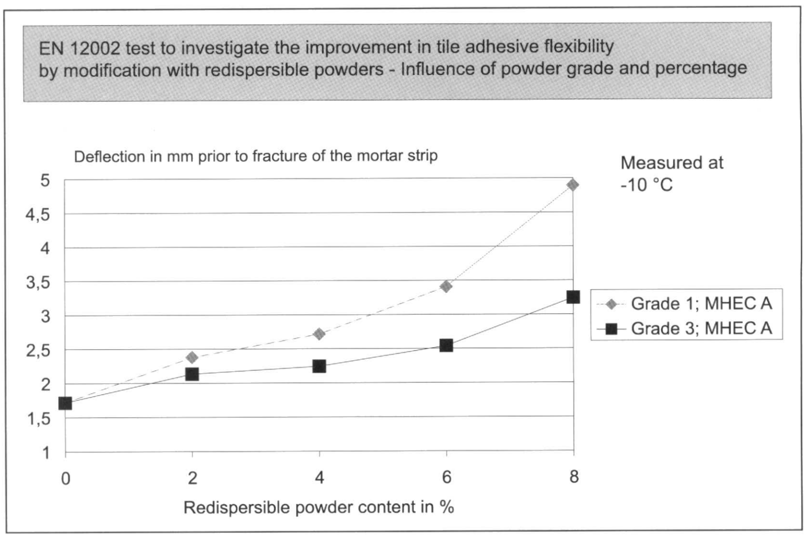

Figure 10

Another method of testing the flexibility of tile adhesives is the so-called shear test as described in DIN Standard 53265 [10] (Figure 11).

As specified in the Standard, two tiles are bonded back-to-back, with an overlap, using the tile adhesive under test. After 28 days' conditioning in a standard climate, they are subjected to a shear test. The deformation that takes place until the shear body fails is measured with distance sensors. Flexibility is found to increase with the proportion of redispersible powder. Once again, the increase is comparable for soft and hard powders when measurements are performed at rom temperature (Figure 12).

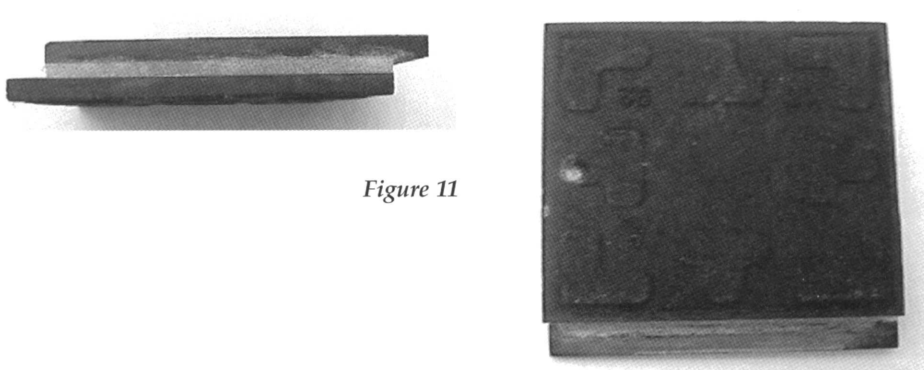

Figure 11

[10] DIN 53265, *Prüfung der Verformbarkeit* (March 1988).

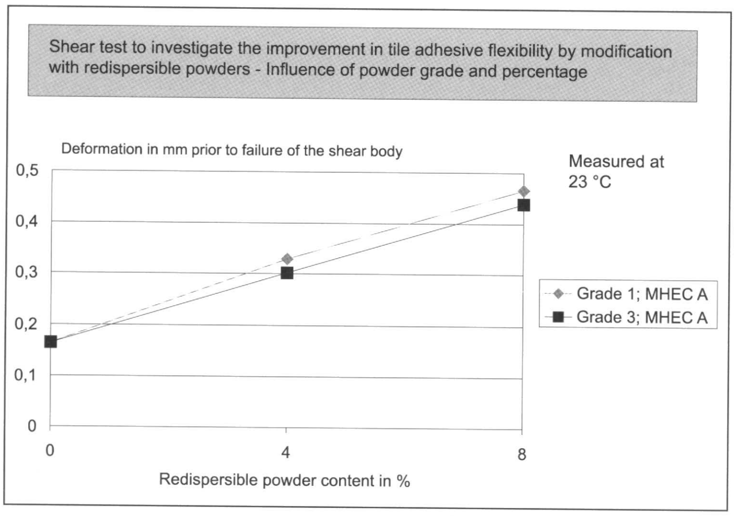

Figure 12

HOW REDISPERSIBLE POWDERS WORK

A number of studies have been carried out on the interaction of cement and redispersible powder in modified mortar systems, eg, [5, 6, 7, 8].

The main reason for the huge improvements in properties obtained by modifying tile adhesives with redispersible powders (improved processing, longer open times, enhanced adhesion and greater flexibility) is the formation of polymer films (so-called "resin domains") in the hardened cement matrix. These result in increased inner cohesion of the tile adhesive due to additional "bonding" of the hardened cement and the fillers (mostly quartz sand) by means of the polymer, which has a higher tensile strength than cement.

The formation of such polymer films between the hardened tile adhesive matrix and the substrate, and between the tile adhesive matrix and the tile, is also of crucial importance for increasing the adhesive tensile adhesive strength and the open time.

In the case of traditional stoneware tiles, which have a high absorption capacity, the wet cement is able to penetrate to a certain extent into the tile and thus to anchor it mechanically. The mineral binder and the stoneware tile literally fuse together, with the cement crystals growing into the open pores of the tile (Figures 13,14).

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[5] J. SCHULZE, *Redispersionspulver im Zement,* Tonindustriezeitung no. 9, 1985.

[6] K. ADLER, *Kunststoff und Zement - eine ideale Verbindung,* Applica 1988, p. 10.

[7] H. HEROLD, *Modification of Ceramic Tile adhesives with Redispersible Polymer* Powders, Qualicer 98, Castellon/Spain 8 / 11 March 1998, Vol. II, p. 191.

[8] J. SCHULZE, *Thermoplastische Polymere,* Beton 5/1991.

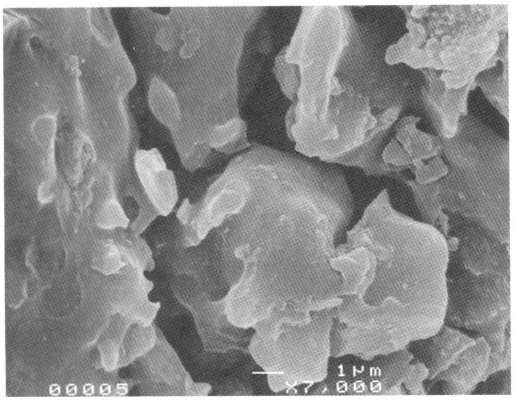

Figure 13. *Earthenware Tile (Electron Microscope Picture.*

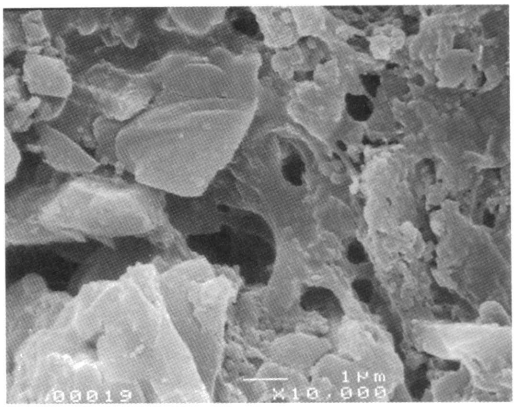

Figure 14. *Earthenware Tile - Tile Adhesive Interface*

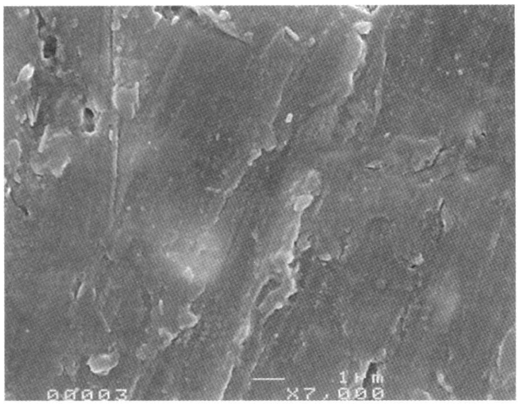

Figure 15. *Stoneware Tile "porcelanato" (Electron Microscope Picture)*

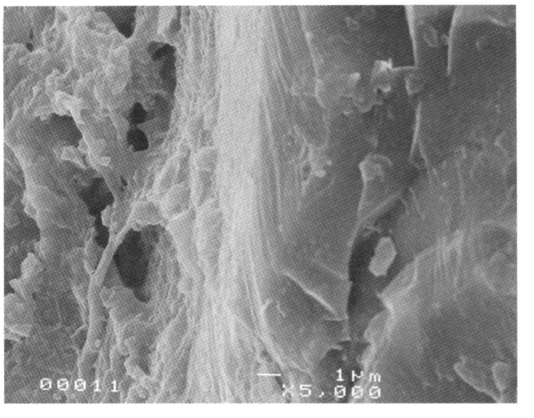

Figure 16. *Tile Ahesive - Stoneware Tile "porcelanato" Interface*

With modern high-quality tiles, by contrast, which show little or zero absorption, this mechanism is no longer effective. Here, it is alone the physical anchoring effect of the polymer by means of its surface contact with the tile that must ensure sufficient tensile adhesive strength. (Figures 15, 16)

It is easy to appreciate that the flexibility of the otherwise rather brittle hardened cement matrix will increase as the proportion of "soft" polymer films increases. The polymer films form "hinges" at which, under the influence of a load, a deformation occurs to accommodate the shear stresses.

CONCLUSIONS REGARDING THE FORMULATION OF TILE ADHESIVES IN ACCORDANCE WITH THE EUROPEAN STANDARD.

Here we should differentiate between standard tile adhesives (C1) and adhesives which fulfil more stringent requirements (C2).

C1 adhesives must exhibit a tensile adhesive strength > 0.5 N/mm^2 after the prescribed conditioning. Flexibility is less important here. As our study has shown, hard polymers, in particular, provide for a rapid increase in tensile adhesive strengths following conditioning under standard climatic conditions and at elevated temperatures. Table 6 shows an example of a C1 adhesive modified with the hydrophobic redispersible powder 2.

Example for C1-tile adhesive

Formulation according to table 2 with cellulose ether B and 2 % Redispersible Powder 2

Tensile adhesion strength:	
28 days (d) standard conditions (sc):	1,28 N/mm²
7 d sc + 21 d immersion in water:	0,91 N/mm²
14 d sc + 14 d 70°C + 1 d sc:	0,71 N/mm²
25 Frost-Tau-Wechsel:	0,55 N/mm²
Open time:	
Tensile adhesion strength (after20 min. waiting time)	0,53 N/mm²

Table 6

C2 tile adhesives are required to exhibit a tensile adhesive strength > 1 N / mm^2 after the conditioning prescribed in the Standard. The higher tensile adhesive strength should ideally be combined with maximized flexibility. For most experts, C2-quality tile adhesives correspond to the as yet non-standardized "flexible" tile adhesives. For maximum flexibility also at low temperatures, soft polymers have for decades been the ideal solution. Table 7 shows an example with 4 % soft powder grade 1.

Example for C2-tile adhesive

Formulation according to table 2 with cellulose ether C and 4 % Redispersible Powder 1

Tensile adhesion strength:	
28 days (d) standard conditions (sc):	1,39 N/mm²
7 d sc + 21 d immersion in water:	1,05 N/mm²
14 d sc + 14 d 70°C + 1 d sc:	1,14 N/mm²
25 Frost-Tau-Wechsel:	1,06 N/mm²
Open time:	
Tensile adhesion strength (after20 min. waiting time)	0,51 N/mm²

Table 7

CONCLUSION

Modern high-quality tiles are characterized by a very dense and thus highly abrasion- resistant ceramic matrix of extremely high mechanical strength. As a result, the tiles are often larger and thinner, but their porosity and water absorption capacity is more or less zero. This has the advantage of conferring very high resistance to freeze-thaw cycling, but the bonding of such "Porcelanic" tiles involves the problems already described.

Since the wet cement is not able to crystallize in the pores of the ceramic tile body, there is no mechanical adhesive bond. These tiles can only be bonded securely by means of modern thin-bed tile adhesives modified with redispersible powder. It is the physical adhesive bond formed within the substrate - tile adhesive - tile system that provides for a sufficiently high tensile adhesive strength and flexibility of the bond.