



TECHLINE3

FOR THE CONSTRUCTION INDUSTRY

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Interaction of Cellulose Ethers and Redispersible Polymer Powders in Cement Based Tile Adhesives

ABSTRACT

Cellulose ether products and redispersible polymer powder products are commonly used as additives to improve the performance properties of cement based tile adhesives. Cellulose ethers provide water retention for open time, tile correction time, and proper hydration development of the cement paste. Cellulose ethers also provide rheological properties for enhanced application characteristics. Redispersible polymer powders improve the adhesion and flexibility of the tile adhesive.

Formulators of cement based tile adhesives seek to optimize the formulation cost and performance by selecting the right combination of cellulose ethers and redispersible polymer powders. Therefore, it is important to understand the types of synergies and interactions that might occur between these additives in the tile adhesive formulation. A designed experiment was carried out to screen the impact of cellulose ether and redispersible polymer powder on the performance properties of a tile adhesive formulation. The test formulation parameters included variation of the cellulose ether chemical substitution type, cellulose ether degree of substitution (DS/MS), redispersible polymer powder type, and the redispersible polymer powder content. Definite synergies and interactions were observed with variations in the combinations of cellulose ether/RDP and RDP concentration; however these observations were dependent on the property examined

INTRODUCTION

Redispersible polymer powders (RDP) and cellulose ethers (CE) are used as additives in many construction applications. Cellulose ethers like methyl hydroxypropyl cellulose (HPMC) or methyl hydroxyethyl cellulose (HEMC) help to control fresh mortar properties like workability, sliding resistance, open time and water retention. They have also a retarding effect on the hydration kinetics of cement which reduces the bonding strength of the mortar. High substitution levels of the CE minimize this effect. Redispersible polymer powders are

additional binders to the cement in dry-mix mortar formulations which enhance the strength and the flexibility of the tile adhesive. The increasing acceptance of norms and regulations has contributed to improved quality of tile adhesives, which require higher levels of additives. As both CE and RDP constitute a significant cost factor in CBTA formulations, a detailed knowledge of their interaction will help to identify the right product combinations to optimize dosage and performance.

Before investigating potential interactions of CE and RDP it is important to understand the individual roles of these additives. Many of the performance tests have been run according to European Norm EN 12004. However, our experience shows that the trend of test results with structural parameters of the additives correlates well with ANSI standards. The qualitative results of these studies are therefore also valid for North American conditions.

CELLULOSE ETHERS

Cellulose ethers (HPMC and HEMC) are available in different molecular weights and substitution levels. The following data have been generated based on a range of various commercially available cellulose ethers. Their structural properties were analyzed and they were all tested according to EN 12004 in the following standard tile adhesive formulation:

Portland Cement CEM I 42.5	40.00 pbw
Quartz Sand (0.1 – 0.3 mm)	60.00 pbw
Cellulose Ether	0.35 pbw
Starch Ether	0.08 pbw
RDP	0.50 pbw
Water	24.00 pbw

Table 1: Standard Tile Adhesive Formulation

In order to understand the impact of additives on CBTA formulations, both the raw mortar properties as well as the final adhesive strength properties have to be considered. They are largely interrelated. Some of the raw mortar properties can even be correlated with the rheology of the aqueous solutions of CE.

The predominant factor controlling the viscosity of CE solutions in water is the molecular weight of the linear polymer. The weight-average molecular weight of the cellulose ethers Mw has been determined by gel permeation chromatography. Plotting the molecular weight against the viscosity of a 1.5% aqueous solution gives a linear relationship (see figure 1).

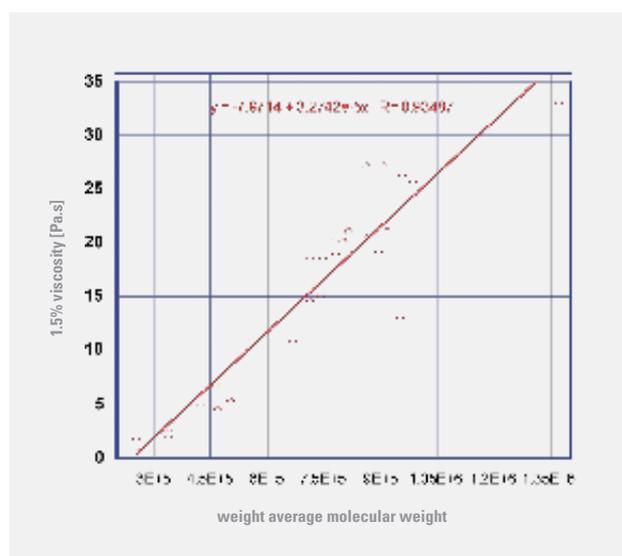


Figure 1: Plot of CE viscosity in Pa s (1.5% aqueous solution at a share rate of 2.0 s-1) against weight-average molecular weight

The aqueous viscosity of the cellulose ether has an important influence on the rheology of the tile adhesive formulation. The rheology of mortars can be reliably measured with a Brookfield RVT rheometer equipped with a T-spindle and Helipath.

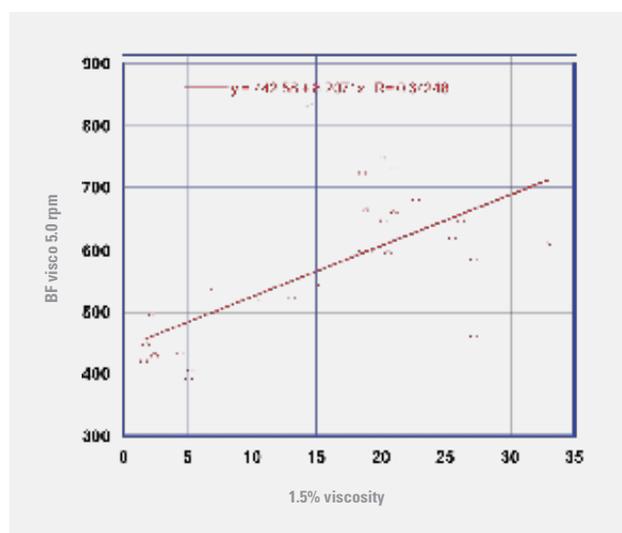


Figure 2: Plot of mortar consistency (as Brookfield viscosity at 5.0 rpm) against aqueous viscosity of the cellulose ether

In order to achieve the same rheology and water retention, high viscous cellulose ethers can be used at a lower addition level; thus saving formulation cost. The viscosity of the cellulose does not only affect the raw mortar properties of the adhesive; it can also have a strong impact on the final strength properties. Reducing the viscosity of the CE has a positive effect on the open time of the tile adhesive as a function of bond strength.

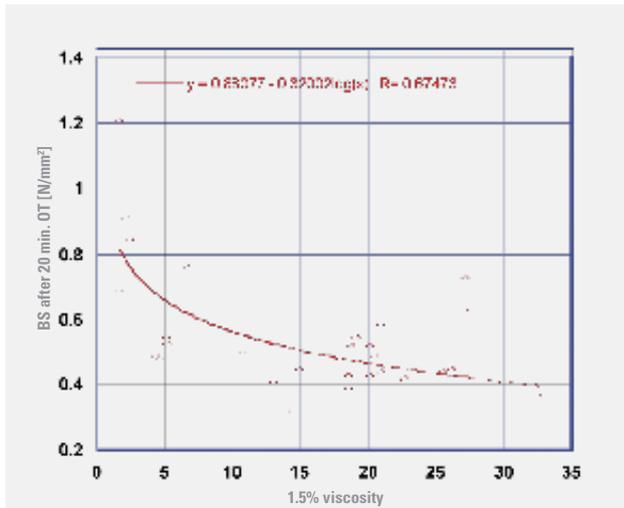


Figure 3: bonding strength after 20min open time as a function of CE viscosity

The reason for this is enhanced wetting capabilities of cellulose ethers with lower molecular weight at the same dosage level. Larger tile surface coverage of the adhesive results in higher bonding strength.

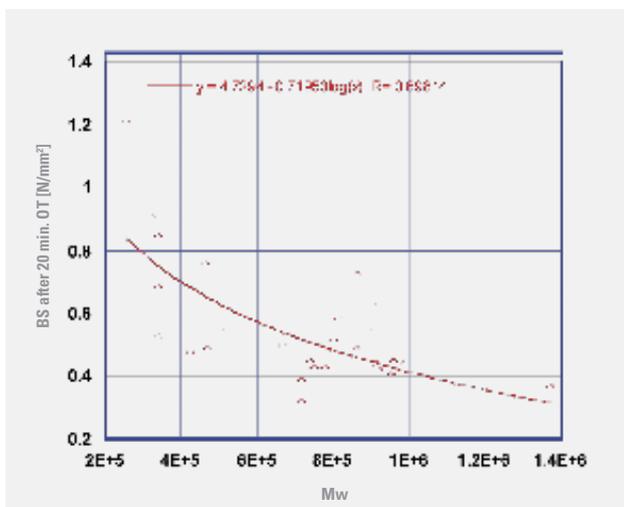


Figure 4: Surface coverage (wetting) as a function of CE weight-average molecular weight

Control of the substitution levels of both substituents in HEMC and HPMC products allows for adjustment of the surface activity of cellulose ethers. The higher the surface activity of the cellulose ether, the more air will be entrapped during the preparation of the cementitious tile adhesive reducing its fresh mortar density.

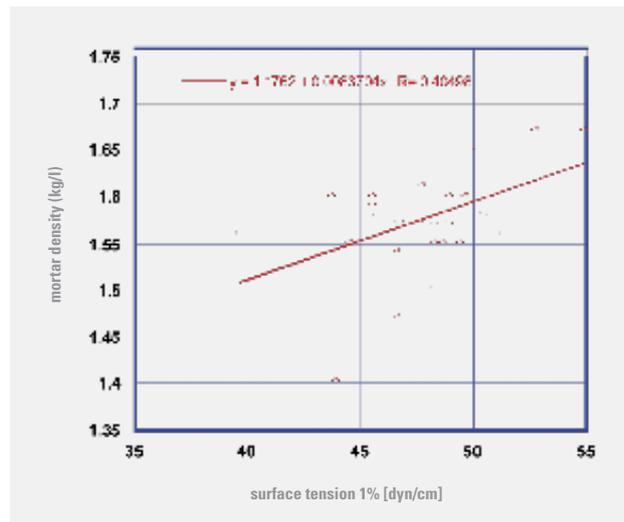


Figure 5: Fresh mortar density as a function of CE surface tension

Low mortar density is beneficial for the workability of the adhesive; thus, requiring less force to spread the mortar. Mortar density has also a strong impact on the strength properties; particularly after the wet aging step. The reason for this relationship will be explained later.

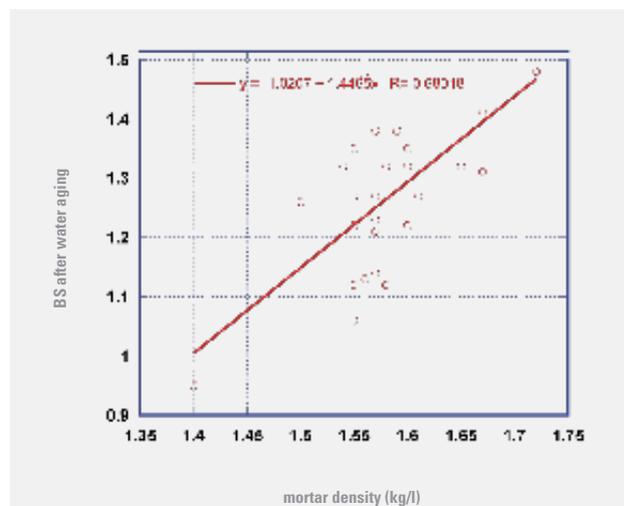


Figure 6: Plot of bonding strength after wet aging against fresh mortar density

Cellulose ether with a weak surface activity will contribute to higher mortar density and improve bond strength. This trend, however, is in conflict with the need for easy workability, which requires low fresh mortar densities. Low fresh mortar density contributes to lower bond strength; particularly after the open time test. In order to find out if the lower mortar densities also result in higher evaporation rates (drying out of the mortar at a faster rate), we have compared the water loss with time of four different CBTA formulations.

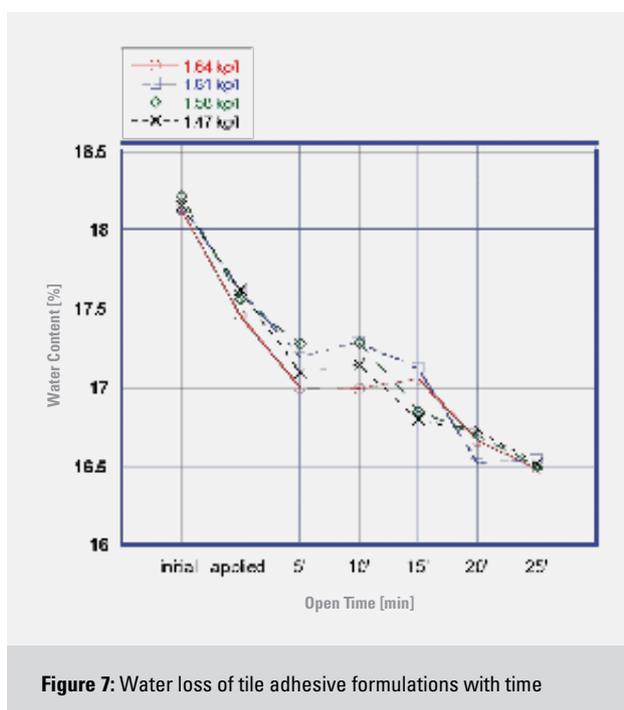


Figure 7: Water loss of tile adhesive formulations with time

Within the accuracy of the experiment, the water loss is independent from the fresh mortar density. The water loss seems to be more pronounced during the first five minutes after the adhesive has been applied. It slows down during the next ten minutes, and starts to become more pronounced again after 15 minutes. This is in line with a mechanism proposed by Jenni et al. . The explanation for this behavior could be that water evaporates quickly on the surface of the CBTA ridges. A thin CE film forms after 5 minutes, preventing further evaporation. More water migrates from the center of the ridges, partially dissolves the initial film, and eventually evaporates. The water flux also transports additional CE to the surface of the tile adhesive where it enriches with time, forming a more solid skin. This skin will not dissolve anymore; thus preventing the adhesive from wetting the tile.

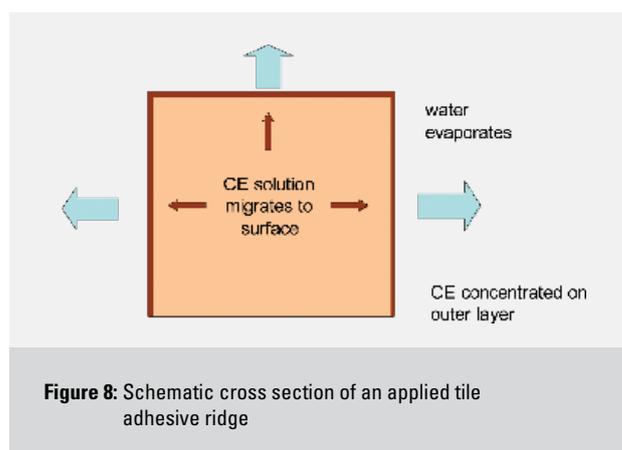


Figure 8: Schematic cross section of an applied tile adhesive ridge

After a period of time, the tile is placed in the mortar bed and a weight is applied. The initial contact of the tile to the adhesive is at the top edge of the ridges. The skin prevents the adhesive from wetting the tile. After the weight is applied, the wet adhesive from the center of the ridges squeezes out at the sides, and wets the tile between the initial ridges.

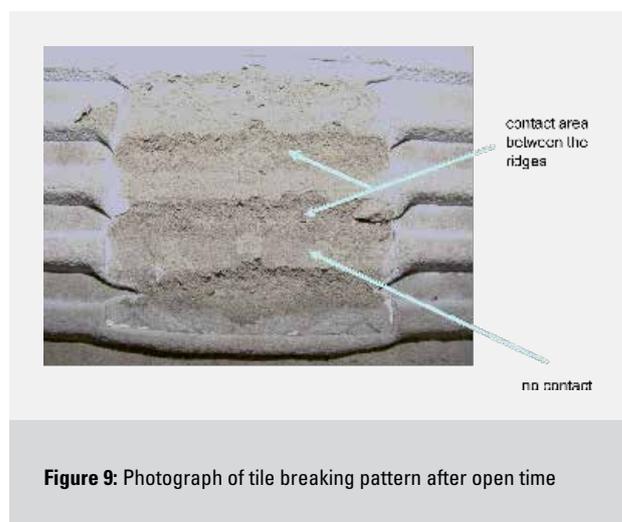


Figure 9: Photograph of tile breaking pattern after open time

The picture above illustrates the breaking pattern after the tile is pulled off after 28 days. There is cohesive failure (breakage within the adhesive) only in the space between the ridges. The mechanism of skin formation is still subject to internal research.

The degree of substitution (DS) of methyl groups has a strong influence on the kinetics of cement setting.

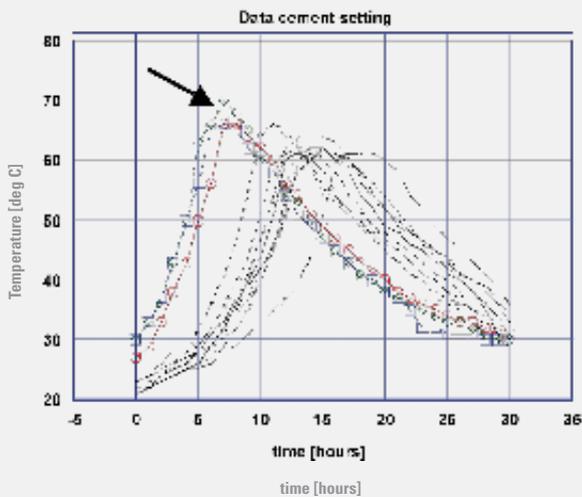


Figure 10: Adiabatic temperature development of cement setting in the presence of various cellulose ethers

Figure 10 shows the temperature development of cement setting in the presence of various types of cellulose ethers. The three curves with temperature peaks at 7 hours (see arrow) represent samples without cellulose ether. Plotting the delay of the cement setting against the degree of methyl substitution DS reveals a linear relationship.

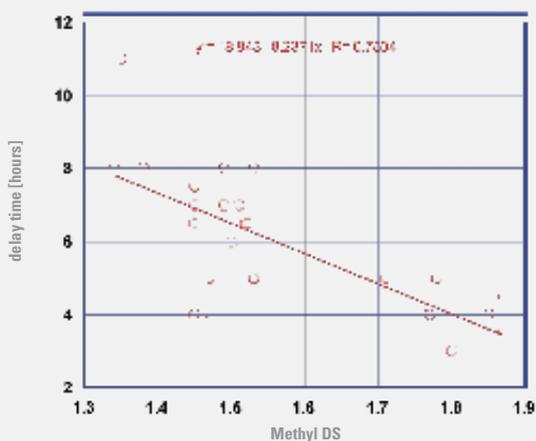


Figure 11: Cement setting delay time plotted against methyl substitution level

It has been shown that sucrose and other glucose derivatives accelerate ettringite formation, but are more effective at retarding C3S (tri-calcium silicate) phase hydration.

It was confirmed that sucrose acts via nucleation poisoning/surface adsorption. The same effect was demonstrated on the polymer as well. H.J. Weyer et al. have monitored the cement hydration with the help of synchrotron radiation in-situ in the presence of cellulose ethers. They found out that the level of methylation has a significant effect on the hydrolysis of the C2S/C3S phases. With increasing methyl-substitution, the anhydroglucose unit of the cellulose ether becomes more hydrophobic, adsorbs less on the clinker phase surface, and consequently has less retardation effect (see figure 11).

A strong retardation effect on the cement setting enables the water to evaporate and dry out the adhesive before the cement can completely hydrate; thus, we observe lower strength properties of the adhesive (figure 12) with low methyl DS.

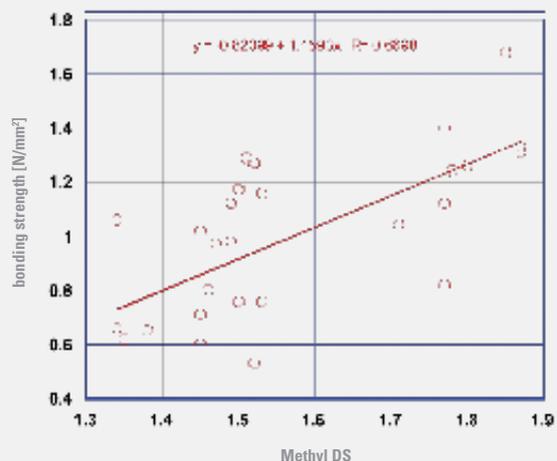


Figure 12: CBTA bonding strength as a function of methyl degree of substitution

The cement setting will resume once the adhesive contacts water. The bonding strength after the water aging step is at a higher level, and almost independent from the methyl DS, as can be seen in figure 13. This effect is more pronounced in formulations containing CE with low methyl DS.

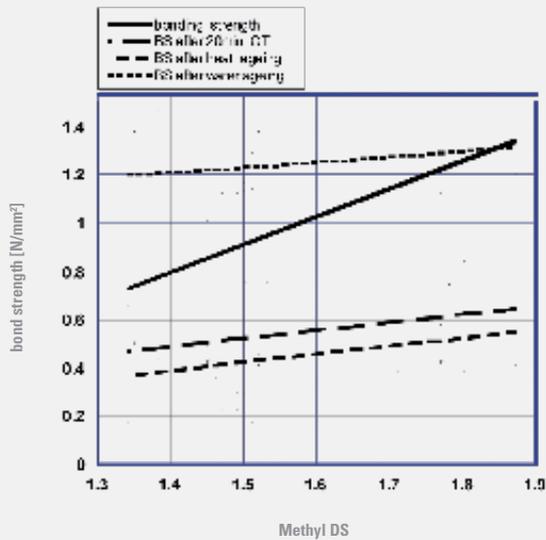


Figure 13: CBTA bonding strength after various aging steps as a function of methyl substitution level

The bond strength after open time is usually weaker and follows a different mechanism as described earlier. Upon heat aging at 70°C, the adhesive is subject to thermal stress at the tile/adhesive interface. This reduces the bond strength significantly.

The addition of RDP helps to make the adhesive more flexible, and improves the bond strength. Depending on the dosage level of the polymer powder, the cohesive strength is improved because the interface between tile and adhesive gets stronger. Adding more RDP to the tile adhesive formulation causes the adhesive to break within the cement matrix and not at the interface as illustrated in figure 14. The breaking pattern of the tile after the heat aging test is shown at 1%, 3% and 5% addition level of a redispersible polymer powder.



1% RDP



3% RDP



5% RDP

Figure 14: Breaking pattern of tiles after the heat aging test with various amounts of RDP

REDISPERSIBLE POLYMER POWDER

Liquid polymer dispersions can be spray-dried and used as a powder component in dry-mix mortar formulations. When mixed with water, the powder redisperses and forms films in the cement matrix, which further enhance the strength properties of cementitious mortars. Vinylacetate/Ethylene copolymers are the preferred polymers in the market for producing redispersible polymer powders. We have compared two RDP products in a CBTA formulation at two different dosage levels.

	Composition	T _g [°C]	MFFT [°C]
Polymer 1	Vinylacetate/Ethylene	6	0
Polymer 2	Vinylacetate/Ethylene	17	3

Table 2: CBTA Formulation

Polymer 2 has a higher glass transition temperature compared to Polymer 1. The adhesives have been tested according to some of the shear bond specifications of ANSI A118.4 and A118.11 in the following formulations:

Component	Unit	Formulation 1	Formulation 2
Cement	pbw	40.0	40.0
Sand	pbw	56.7	54.7
RDP	pbw	2.0	4.0
HPMC	pbw	0.3	0.3
Water	pbw	24.0	24.0

Table 3: ANSI A118.4 / A118.11 Formulations

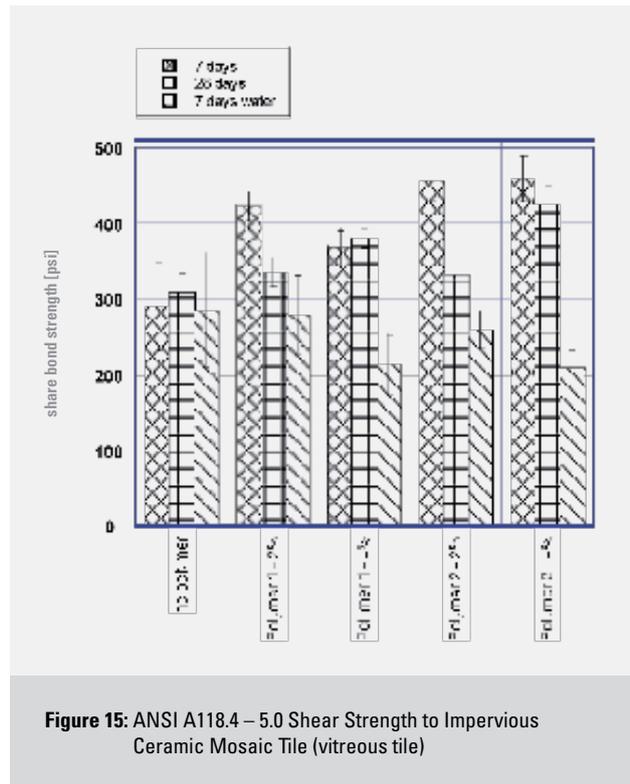


Figure 15: ANSI A118.4 – 5.0 Shear Strength to Impervious Ceramic Mosaic Tile (vitreous tile)

The shear bond strength of two impervious ceramic mosaic tiles (low absorption vitreous tiles) after various aging periods is illustrated in figure 15. The shear bond strength after 7 days is the case of 2% RDP dosage level, specific to this evaluation, higher compared to the 28 days. The reason for this is still unknown to us. When comparing the shear bond strength after 28 days for the five formulations, it becomes obvious that that the use of RDP in the formulation improves the strength properties of the adhesive. The harder polymer (Polymer 2) provides higher strength.

After the test specimens have been immersed in water for 7 days, the shear bond strengths decrease to the level observed for the mortar without polymer powder. In formulations with 4% RDP the drop in strength is even more pronounced. The water immersion step seems to completely reduce the contribution of the RDP to the mortar strength.

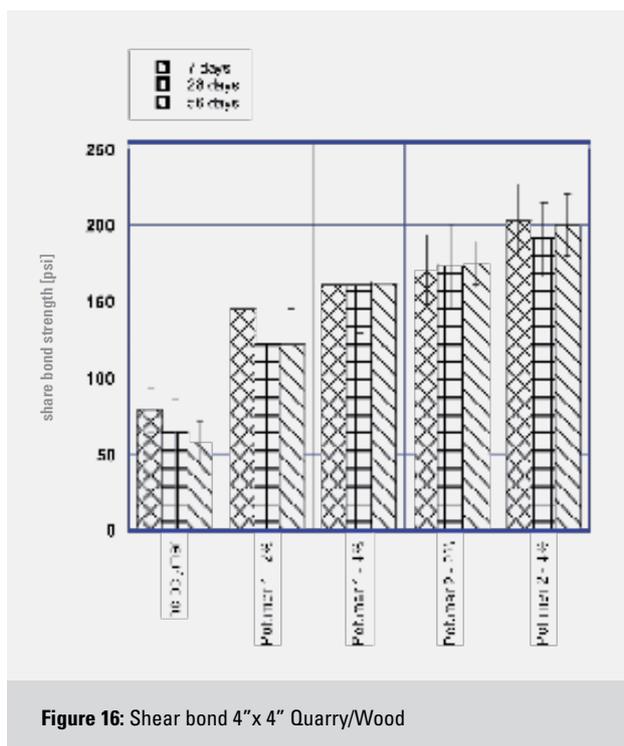


Figure 16: Shear bond 4" x 4" Quarry/Wood

When quarry tile / plywood composites are tested for shear bond strength, the impact of the polymer becomes more pronounced, as illustrated in figure 16. Adding 2% of polymer 1 to the formulation doubles the strength of the adhesive. With 4% of polymer 2 the strength is almost four times the value of the adhesive without polymer powder. An increase in bond strength is also observed between formulations containing polymer 1 and polymer 2; most likely due to the higher glass transition temperature of polymer 2.

INTERACTION OF CELLULOSE ETHER AND REDISPERSIBLE POLYMER POWDER

Formulators of cement based tile adhesive try to optimize the cost/performance ratio by selecting the right combination of cellulose ethers and redispersible polymer powders. The understanding of synergistic effects or incompatibilities between these additives is critical. We have used an experimental design to screen the impact of CE and RDP in a CBTA application and to identify potential interactions.

The base formulation was:

Quartz Sand (0.1 – 0.3 mm)	60.00 pbw
Portland Cement CEM I 42.5	40.00 pbw
Cellulose Ether	0.35 pbw
Water	26.00 pbw

Three independent variables:

Variable	Parameters		
CE Chemistry	HEMC		HPMC
RDP dosage	1%	3%	5%
RDP grade	RDP1	RDP2	RDP3

Both cellulose ethers have an aqueous viscosity of 35,000 mPa*s (2% aqueous solution, at 20 rpm Brookfield RVT, spindle 4, 23°C)

Variable	Parameters	
CE Chemistry	HEMC	HPMC
DS MeO	1.6	1.8
MS HPO/HEO	0.27	0.15

The RDP grades represent:

RDP1	Vinylacetate/ethylene copolymer with neutral rheology
RDP2	Vinylacetate/ethylene/Veova* terpolymer with neutral rheology
RDP3	Vinylacetate/Veova* copolymer with thickening effect

Fresh mortar density is an important parameter affecting the strength properties of cement based tile adhesives. Both the type of CE and the type and dosage of the RDP play an important role for the density of the mortar. Figure 17 illustrates the possible combinations and their effect on mortar density.

* Veova = vinyl ester of versatic acid (a synthetic, highly branched-C10 tertiary carboxylic acid)