IMPROVING ADHERENCE BETWEEN GLASS TILES AND CEMENT MORTAR BY ORGANOSILANES

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ABSTRACT

Adherence between glass tiles and cement mortars is crucial to the stability of tile systems and, based on chemical features, only the weak van der Waals forces and hydrophilic interactions may be expected to develop between glass tiles and Portland cement mortar. In this sense, the major objective of this paper was to use organosilanes as primers to modify glass tile surface properties in order to improve adhesion between glass tiles and cement mortars. The glass tile surface has been treated with several silane derivatives bearing specific functionalities. Contact angle measurements and Fourier Transformed Infrared Spectroscopy were used as characterization techniques for evaluating the character of the modified surface. In order to assess the effect on adhesion properties pull-off tests were conducted. Contact angle results have given reliable evidence that they were altered from hydrophilic to hydrophobic after silane modification. FTIR spectra presented major peaks associated with the organic moieties. Pull-off test results have indicated that surface modification has affected bond strength between cement mortar and glass tile. The adherence results varied in a broad range reflecting the overall balance of silane and cement features including reactive organofunctional group, hydrophobic/hydrophilic features, kind of interactions developed between silane and cement.

1. INTRODUCTION

Adherence between glass tiles and cement mortars is crucial to the stability of tile systems and, based on chemical features, only the weak van der Waals forces and hydrophilic interactions may be expected to develop between glass tiles and Portland cement mortar. On the other hand, surface modification is generally performed to create or modify certain surface properties and silane coupling agents are the standard products used to alter surface features. Besides that, it has been studied the possibility of development of covalent bonds between some organosilanes and cement through covalent bonds. In this sense, the main goal of this paper was to use organosilanes as primers to modify glass tile surface properties in order to improve adhesion between glass tiles and cement mortars.

2. EXPERIMENTAL PROCEDURE

Soda-lime glass tiles were selected to be used to evaluate the effect of silane modification in the adherence between mortar and silane modified glass tile. Glass tile surface has been treated with several silane derivatives bearing specific functionalities. Amino and vinyl groups were chosen as reactive functionalities of silanes for evaluating their compatibility with Portland cement (Table 1). The silanes were supplied by Sigma-Aldrich.

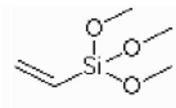
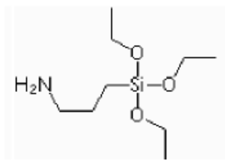
Reagent	Chemical Structure
Vinyl trimethoxysilane	
(3-Aminopropyl) triethoxysilane	

Table 1. Silanes used in this work.

Before silane application, surface impurities were reduced and hydroxyl concentration was increased. Glass tile surface impurities were reduced by immersion in 20% nitric acid for 2 hours, followed by deionized water (D.I.) rinse, and then immersion in deionized water overnight followed by drying.

Hydroxyl functionalization of tile was increased by immersion in a 70:30 mixture of deionized water: 30% hydrogen peroxide for 45 min at about 70ºC followed by dropping of 5 ml of NH_4OH for each 100 ml of the mixture. After cooling, glass tile was rinsed in D.I. water and then dried in methanol.

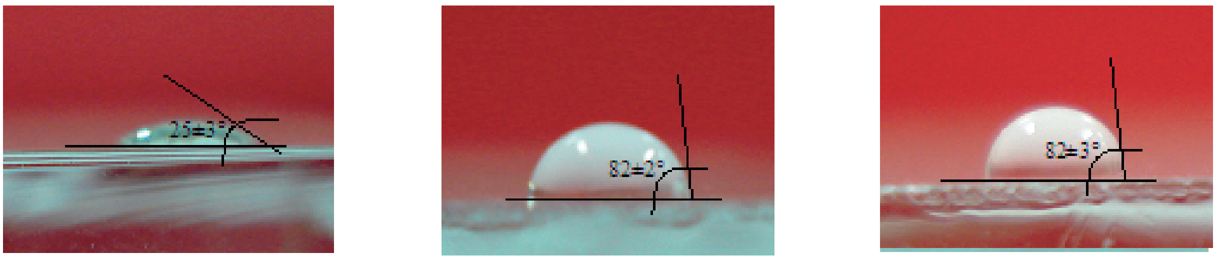
Silane application was conducted by dropping 500 μl of alkoxysilane onto the glass surface followed by spreading out using other glass tile. Silane layer was polycondensed for 2 hours inside a water vapour chamber. After that, they were rinsed free of excess materials by dipping in methanol.

The influence of surface functionalization on the hydrophilic/hydrophobic behaviour of glass substrate was estimated via contact angle measurements. We have evaluated the average contact angle of Milli-Q water (18.0 MΩ) spread over glass tiles. Also FTIR (Fourier Transformed Infrared Spectroscopy) was used to characterize the presence of organic chemical groups in the inorganic glass tiles, reflecting the effectiveness of the developed procedure for functionalization of surface. Transmission technique was used (FTIR Spectrum 1000, Perkin Elmer) within the range between 4000 and 2700 cm^{-1} during 32 scans. This range was chosen because it contains most of the peaks of –CH stretching without overlapping with glass characteristic peaks.

Portland cement type CPII-F 32 according to Brazilian Standard NBR 11578/91 and deionized water were used to prepare the mortars. Cement:sand ratio of 1:1.7 was used to prepare the mortar (in weight basis). The water/cement ratio was 0.6. The mortar was manually mixed for 3 min. After that, mortar was let to rest for 10 minutes covered by a humid cloth, followed by 1 min of mixing before use. A single layer of cement mortar with average thickness of 6 mm was applied on the surface of a concrete substrate. Tiles were positioned on the mortar, followed by the application of a 10 MPa load for 60 seconds to promote mortar spreading and penetration into surface roughness. Concrete substrates were left in a humidity and temperature controlled (T = (25±5)°C and RH > 90%) chamber for 10 days followed by cure at laboratory ambient (T = (25±5)°C and RH= (70±10)%) for 14 days before pull-off tests. Pull-off assays were conducted adapting the recommendations of Brazilian Standard NBR 14084/98 test method.

3. RESULTS AND DISCUSSION

Contact angle measurements indicated a strong difference on the hydrophilic behaviour of the pure glass tile compared to surface-modified tiles. The average values of contact angle (θc) are shown in Figure 1. These values are in agreement with literature.



a) b) c)

Figure 1. Contact angle measurements for control (unmodified) (a), amine (b), and vinyl (c) silane modified glass tiles.

In Figure 2, FTIR spectra of glass tiles modified with alkosilanes are shown. It can be verified the presence of the -CH stretching vibration bands (2850-3000 cm^{-1}) mainly associated with the propyl group introduced by the functionalization of the glass surface.

Figure 3(a) shows the influence of the surface modification on the bond strength of mortars and Figure 3(b) indicates the variation of bond strength due surface modification.

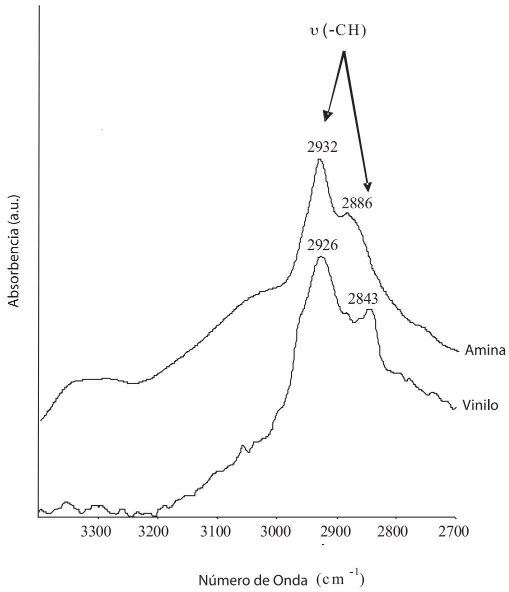


Figure 2. FTIR spectra for the modified glass surface.

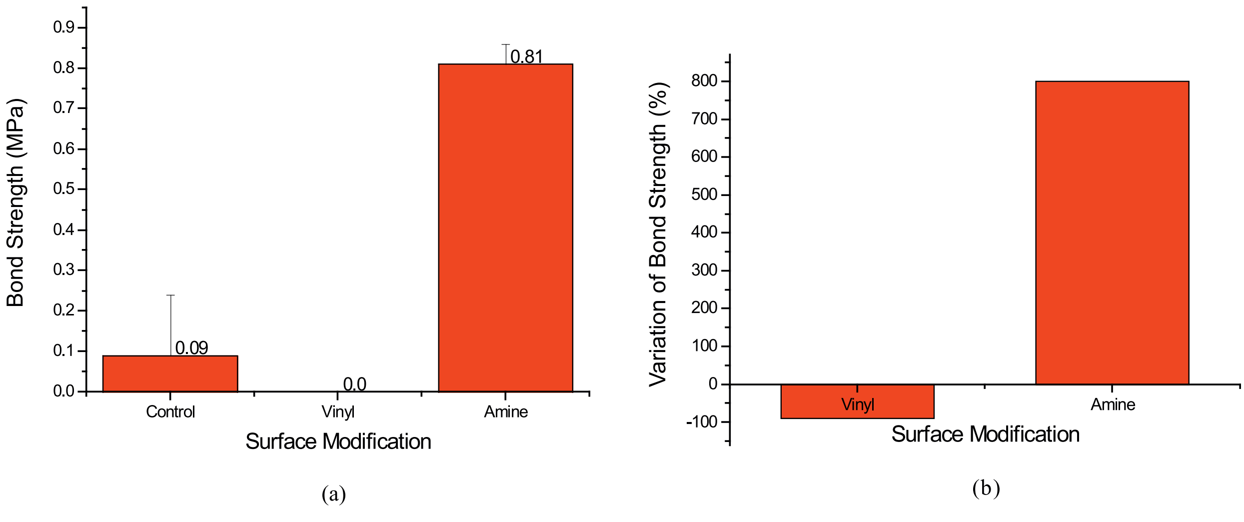


Figure 3. (a) Effect of the surface modification on the bond strength of Portland cement mortars to glass tiles. (b) Variation of bond strength due surface modification.

Statistic analysis of bond strength results indicate a significant increase of adherence, with 95% of confidence, for glass tile modified with (3-Aminopropyl) triethoxysilane. For this functional group we have also verified an important change of rupture mode from mostly interfacial failure to a mixed mode interfacial-cohesive of the mortar (Figure 4). However, for vinylsilane, a decrease of the bond strength was verified.

The increasing bond strength and cohesive rupture of mortar is believed to be associated with covalent bonds between alkoxy-derived and calcium silicate hydrates (C-S-H) (Figure 5). Minet *et al*. (2006) and Franceschini *et al*. (2007) have shown the incorporation of organic groups from alkoxysilanes in calcium silicate hydrates in alkaline media at room temperature without disrupting the C-S-H inorganic framework. These results were obtained for very small inorganic groups, like amino. For larger-sized or for highly hydrophobic organic groups, like vinyl, phase separation has occurred, in agreement with the pull-off tests results.

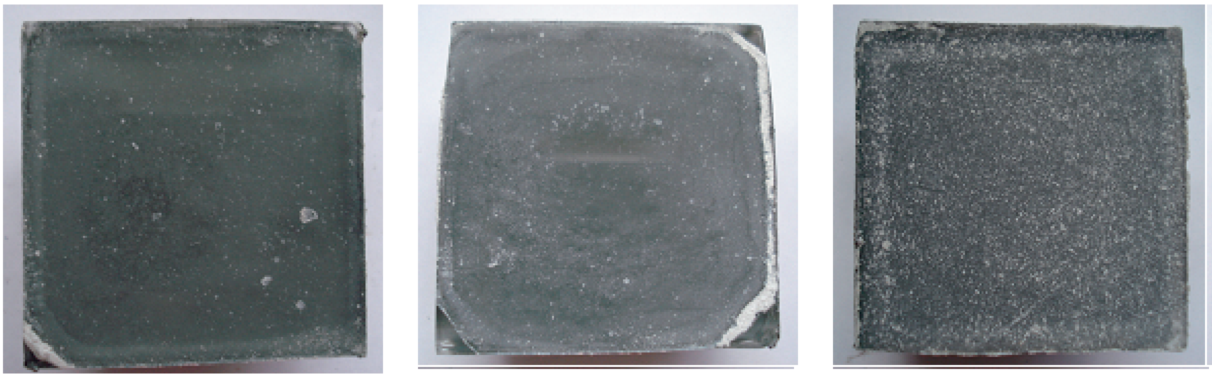


Figure 4. (a) Interfacial mode of rupture for control glass tile and (b) for vinylsilane modified surface. (c) Mixed mode interfacial-cohesive failure of the mortar due aminosilane glass tile modification.

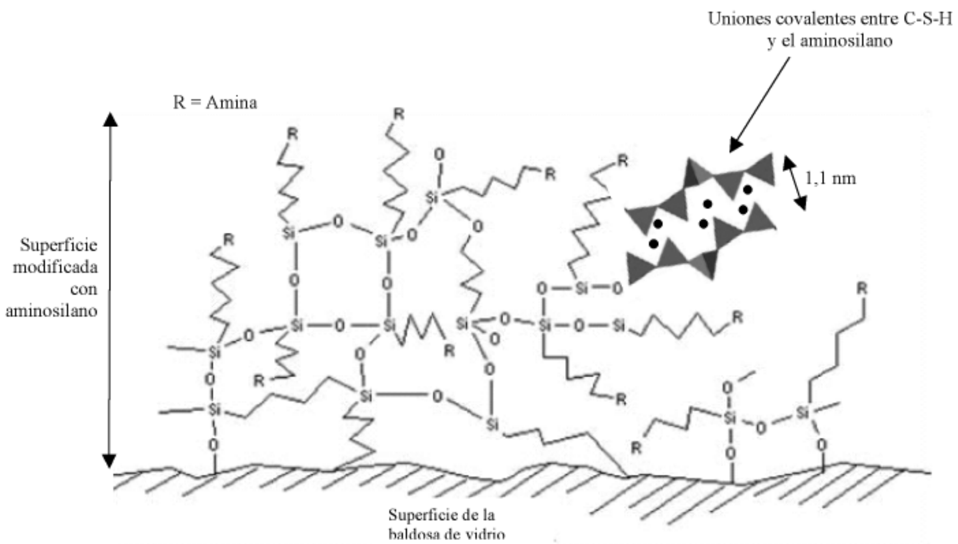


Figure 5. Possible interactions between aminosilanes and Portland cement.

4. CONCLUSIONS

In this work we have shown through contact angle and FTIR measurements that the proposed method of silane application and reaction was efficient to functionalize glass tile surface. Also, the effects of surface modification on bond strength between Portland cement mortar and glass tile were verified. The adherence results varied in a broad range reflecting the overall balance of silane and cement features including reactive organofunctional group, hydrophobic/hydrophilic features, kind of interactions developed between silane and cement. Based on the results aminosilanes were effective on improving adherence between Portland cement mortar and glass tile materials.

5. ACKNOWLEDGMENTS

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