USE OF SILICONE RESIN AS INTEGRAL WATER REPELLENT FOR MORTAR AND FIBRE REINFORCED CEMENT BOARDS (FRC)

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ABSTRACT

This paper describes the use of silicone resin as a hydrophobic admixture for cement-based matrix and fiber reinforced cement boards in particular.

The impact of Z-6289 as a hydrophobic admixture in mortar on water uptake, cement setting time and mechanical properties is given.

The positive impact of adding a silicone resin (Z-6289) in a fiber board composition is illustrated. Z-6289 was added to lab-made FRC boards and its effects on the board water uptake, post coating paint adhesion, efflorescence, freeze thaw stability are illustrated.

KEYWORDS:

Silicone resin, hydrophobic admixture, mortar

INTRODUCTION

Reinforcement of cement-based materials with various forms of fibre has been common for a long time [1]. The first modern fibre reinforced construction materials were asbestos-cement boards used as flat or corrugated sheets. Asbestos fibres are mixed with a slurry of cement and water to reach a fibre content around 6-18% of the dry formulation in so- called Fibre Reinforced Cement Boards (FRC).

FRC boards made with asbestos fibres showed good mechanical strength and durability however, the hazards of asbestos fibre to human health have lead to its ban in construction industry in many countries. Alternative fibres such as refined cellulose pulps and synthetic fibres such as polyvinyl alcohol fibres (PVA) or polypropylene fibres (PP) started to be used as replacements in the seventies[2,3].

The successful replacement of asbestos fibres with cellulose fibres however, raised new challenges not only of the way to manufacture the boards but also the properties of the finals boards. For example, cellulose-based boards have a higher tendency to absorb water and are more susceptible to degrade due to water absorption.

Simply stated, cellulose board will generally be more susceptible to problems associated with water absorption than asbestos reinforced boards; and this can be a major problem facing manufacturers as they move to replace asbestos by cellulose fibres. Carbonation is another factor which may negatively impact boards durability [4]. When boards absorb too much moisture, many potential problems can occur, such as:

- Reduced Dimensional Stability
- Reduced Freeze/thaw resistance leading to cracking and warping
- Potential for white efflorescence salts affecting appearance
- Reduced durability

Although asbestos is still used in many countries, there is a major global shift away from using asbestos by replacing it with much safer fibres such as cellulose or wood pulp fibres. Some companies choose to move gradually to asbestos by using asbestos/cellulose blends. Formulations for FRC boards vary greatly throughout the world. The fibre source, the cement source and the type and amount of siliceous extenders vary dramatically from plant to plant. Coupled to this is the fact that manufacturers use many different cure processes and post treatments which all mean that each plant will be different and may require slightly different solutions. Different technical solutions can and are being used to combine aesthetic and the need to reduce water absorption.

When FRC siding panels are manufactured, they will be sold to the end user either 'raw', primed or post finished [5].

- *Raw*: No post treatment in the factory. All coating takes place on site (or not at all).
- *Pre primed*: Many manufacturers prime boards with 'universal' primers (typically acrylic based) before they leave the factory. They are then post coated to the desired colour after fixing on site.
- *Sealed:* Silicone based sealers can be used to protect the boards when raw cement appearance is desired.
- *Primed AND topcoated*: Before painting, some manufacturers preseal with silicon-based penetrants before applying the acrylic prime coat and potentially top coat.
- *Integral water repellent :* A last option consist in integrating the silicon-based hydrophober into the boards formulation to produce an "integral water repellent". Silicon-based hydrophober is then used as a so-called admixture in the board formulation.

The primary purpose for coating panels is for aesthetics. As long as a coating is fully integral, then the water absorption of the board will be reduced. Once the coating fails; so does the protection. Coatings provide little or no protection against ingress of moisture at the edges or the rear surface of the board. By presealing boards with silicone penetrants on the front and rear surfaces as well as the edges prior to coating, then far greater long term protection of the boards can be achieved. If treated in this way; the FRC boards and panels have long term protection against water ingress regardless of the life and/or quality of the paint treatment used. If the silicon-based hydrophobe is used as an admixture, the boards can also be cut on the job site without impairing the protection against water penetration. This last option is raising interest amongst the FRC manufacturers to further reduce risk of ligitation or to eliminate one post treatment step.

In this process of producing "integral water repellent", the silicone will become an integral component of the board as it cures and therefore forms a water barrier throughout the entire board. One advantage of this approach would be that the FRC products would be protected from water absorption from the time they are initially produced and would not depend on a post-treatment or protective coating to provide improved water resistance.

Another useful, and valuable advantage of using an admixture to "build-in" water resistance will be the ability to cut FRC products at the location where they will be installed, and still maintain protection that may be lost when using a post-treatment or coating. Extending this logic a bit, the FRC products will also retain protection even if sealers, coatings or edge sealants like caulking are damaged or not applied perfectly.

In this context, a new silicone resin was developed to be used in FRC process.

This paper describes the efficiency of a specific silicone resin named Z-6289 as hydrophobic admixture for cement matrixes and as admixture for fibre reinforced cement boards in particular.

This study is concentrating on the "hydrophobic performance" of Z-6289 silicone resin used as admixture. In this document, the phrasing "Hydrophobic performance" will describe the extent to which Z-6289 resin used as admixture in a cement matrix (mortar for example) or fibre reinforced cement board formulation can decrease significantly the tendency of final material to absorb water by capillary action.

EXPERIMENTAL

Determination of water uptake upon immersion

Samples of FRC boards or mortar blocks are dried in an oven at 50°C for one day before testing. They are then placed in a vat so that the samples are recovered by three centimetres of water.

At fixed times, samples are removed from water, quickly towelled in order to weight only absorbed water, weighed and replaced in water. The percentage of water uptake is calculated according to this formula:

percentage of water uptake=(Wx-Wi)/Wi $\times$100

With Wx: sample weight after x time in water in grams

Wi : initial weight

Adhesion cross-cut test

Impact of Z-6289 admixture on paint adhesion was tested by applying two coats at 12 hours interval of acrylic paints on one face of the lab-prepared modified boards with a traditional paint brush. The paint is left for drying for 7 days at Room Temperature.

The adhesion cross-cut test was performed according to the ISO2409 standards.

The sample is cross-cut to the substrate with lines at right angles, 1 mm apart using a special cross cut comb. A piece of tape Scotch 2525 was put on the cross (in the same way as it was made) and a slight pressure with the finger was applied. Then it was pulled off in one firm go. Adhesion was evaluated according to the quantity of coating missing on the squares (0 being perfect: with no coating gone and 5 very bad with all the coating removed– see scale).

RESULTS AND DISCUSSION

Impact of Z-6289 addition in a cement matrix was assessed on mortar.

Z- 6289 as admixture in mortar.

Efficiency of Z-6289 as hydrophobic admixture in a cement matrix was assessed by preparing simple mortar made of cement and sand (cement/sand ratio = 1/3) modified by the addition of Z-6289 within the slurry.

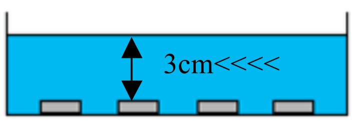
Mortars composed of - 18 g of cement, - 54 g of sand and - 9 g of water were prepared and poured into a 5 * 5 * 2 cm plastic mould.

Mortars were modified with increasing content of Z-6289 : 0, 0.1, 0.5, 0.75 and 1% of Z-6289 vs the dry mortar composition were added into mortar composition. Mortar paste was mixed till homogeneous mixture is obtained. Mortar blocks were left in a climatic chamber at a minimum of 90% Relative humidity. After one day, the samples were demolded and placed back in a climatic chamber at 90% RH. After one week cure, the samples were dried at 50°C for 2 days and then cooled down to Room Temperature.

Resistance to water ingress.

Resistance of modified mortar blocks to water ingress was assessed by immerging the blocks in a plastic basin filled with water. The blocks were placed in such a way to have a height of 3 cm above the blocks (see figure).

The samples were removed after 1, 2, 4, 8 and 24 hours immersion, towelled and weighed.



The increase of weight is reported as % of water uptake vs initial dry weight.

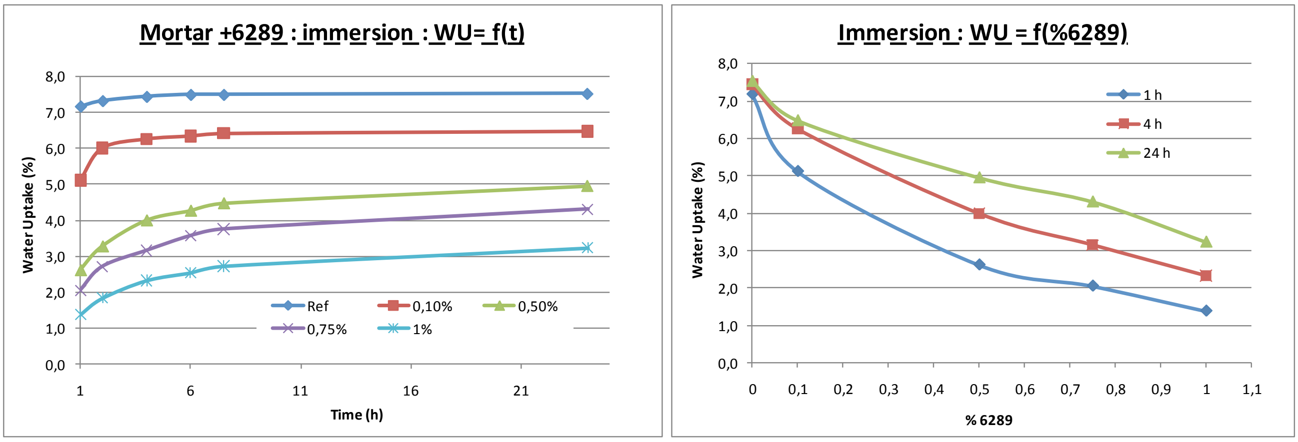


Figure 1 : water uptake of modified mortar as a function of immersion time in water. Plot of water uptake after 1, 4 and 24 hours contact time as a function of Z-6289 content.

The graphs are showing the increase of water uptake as a function of immersion time in water. It is clear that admixing Z-6289 in a simple mortar decreases water absorption.

The decrease of water uptake at given immersion time as a function of Z-6289 addition level into the mortar is illustrated as well.

The impact of Z-6289 is reaching a plateau value as doubling Z-6289 content from 0.5% to 1.0% addition level has only a limited impact on further reduction of water uptake.

Resistance to efflorescence.

Impact of admixing Z-6289 in mortar on resistance against efflorescence was assessed by placing different blocks in a saturated sodium chloride solution.

Transfer of water containing dissolved salts through the interconnected pores system leads to transfer of saturated salt solution at the surface of the block. Evaporation of water leads to precipitation of sodium chloride as illustrated in Figure 4. Positive impact of Z-6289 hydrophobic admixture against efflorescence can be easily illustrated by observing the drastic reduction of sodium chloride precipitation at the surface of the mortar blocks. This clearly illustrates the strong reduction of capillary absorption of water through the interconnect pores systems of modified mortar blocks.

Impact of Z-6289 on mortar mechanical properties.

Mechanical properties of reference mortar and mortar modified with 0.25% of Z-6289 were assessed. Mortar were prepared and tested according to guidelines given in norm EN 196-1. CEMII B-M (S-V) 32.5 N cement was used. Mechanical properties were measured after 28 days cure. Values for the compressive strengths were 48.3 MPa for the unmodified mortar and 44.3 MPa for the modified mortar which correspond to a drop of compressive strength of less than 10%.

Impact of Z-6289 on cement setting time.

Setting time of cement was measured according to the plunger method (according to EN 480-2 norm). No significant different was found between setting time of reference mortar and mortar modified with 0.25% of Z-6289.

Use of Z-6289 as admixture in Fibre Reinforced Cement boards.

Fibre reinforced cement boards were prepared in the lab to assess the efficiency of Z-6289 as hydrophobic admixture. A simple FRC formulations made of 80% of Ordinary Portland cement, 10% of cellulose fibres, 10% of fine sand was selected.

The solid content of the slurry was set at 20% in order to ease the filtration process at the lab scale.

10 g of virgin cellulose fibres sheets were cut into smaller pieces and placed in a 500 ml beaker. 250 ml of water was added and mixed with a lab mixer for 2 minutes at 700 RPM to obtain a homogenous slurry.

250 ml of water as well as 10 g of sand were added and mixed for 1 min at 500 RPM.

Still while stirring, 80 g of cement were added. Stirring was maintained for 3 additional minutes.

Z-6289 resin was then added and the slurry was further mixed for 2 minutes.

The slurry was then filtered on a büchner funnel (filtering under vacuum generated with a venturi water pump in order to mimic Hatscheck process).

The boards were removed from the Büchner funnel, placed between two metallic plates and pressed under a pressure of 1.8 tons for 1 minute.

The boards were placed in a plastic bag stored at room temperature for 7 days in order to cure the boards in an atmosphere at 100 % relative humidity.

After 7 days, the boards were, still in the plastic bag, placed in a oven at 50°C for 4 days.

The boards were then removed from the bags and dried in an oven at 50°C for one day.

Testing of FRC boards modified with Z-6289.

Addition level of Z-6289 was increased from 0% up to 1% vs the dry formulation of the FRC boards.

Resistance to water ingress.

First test consisted in immersing the modified and dried boards in water and measuring the water absorption upon immersion time (see experimental section).

The graph of the water uptake of FRC boards as a function of immersion time is illustrated hereafter.

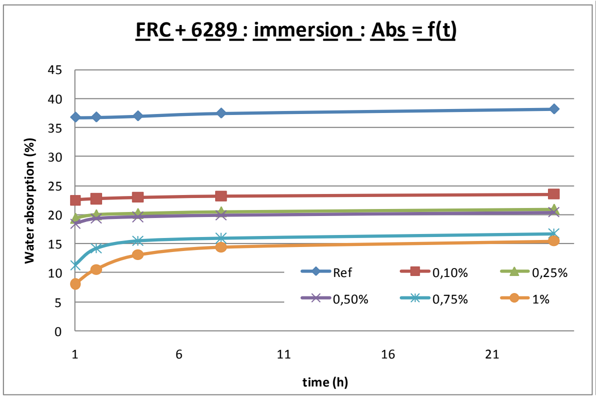


Figure 2 = FRC modified with increasing content of Z-6289. Plot of % of water uptake as a function of immersion time.

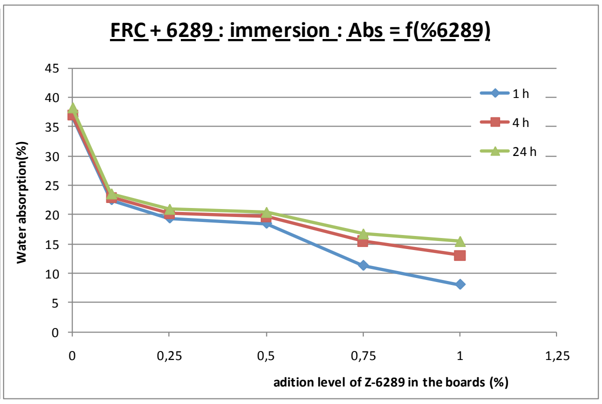


Figure 3 : FRC modified with increasing content of Z-6289. Plot of % of water uptake after 1, 4 and 24 hours as a function of Z-6289 in the modified FRC boards

The graph clearly shows that addition of Z-6289 into the FRC board formulation leads to a strong reduction of water absorption by the boards. Figure 3 shows the plot of water absorption of the modified boards after immersion time of 1, 4 and 24 hours as a function of the Z-6289 content in the boards.

As observed in mortar, the water uptake tends to level off at higher Z-6289 content.

This means that addition level above 0.3-0.5% will have relatively limited benefit, at least in this specific board formulation, and board preparation process.

Resistance to efflorescence.

Impact of admixing Z-6289 on efflorescence in FRC boards was assessed by placing modified and reference boards for a couple of days in a saturated sodium chloride solution.

Migration of water containing dissolved salts through the boards followed by evaporation leads to crystallisation of salts crystals at the surface of the boards (see picture hereafter).

Addition of Z-6289 as admixture leads to a strong reduction of the efflorescence as limited salt crystallisation is observed even at the lowest Z-6289 addition level. As soon as the Z-6289 addition level reaches 0.25%, almost no crystal formation can be visually observed.

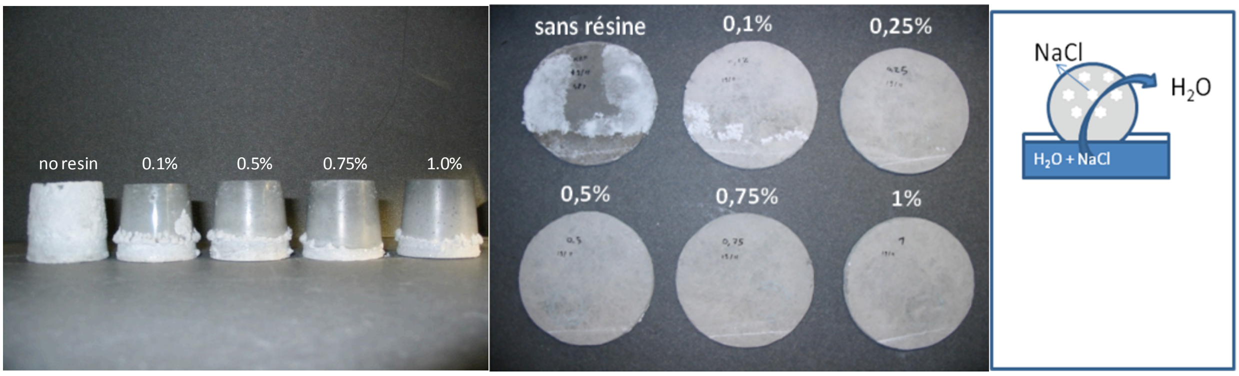


Figure 4 : Picture of A: mortar blocks and B : FRC boards modified with increasing content of Z-6289 placed in saturated chloride solution for several days. Decreased precipitation of salts at the block surface illustrates the resistance against efflorescence.

Resistance to freeze-thaw cycle.

Untreated and modified FRC boards were submitted to a set of freeze-thaw cycles.

One cycle was as followed.

1. The boards were immersed for one hour to insure saturation with water.
2. Boards were then placed for 24 hours in a refrigerator (-15°C).
3. Boards were then removed from the refrigerator and placed at Room Temperature for 24 hours. Boards were placed in individual plastic bags to avoid contamination and drying of the boards.

Figure 5 : picture of untreated and modified FRC boards (with 0.5% Z-6289) after 10 freeze thaw cycles.

The following picture shows two boards from their side (one untreated reference and one board modified with 0.5% Z-6289).

The pictures clearly show that the reference board starts to bend due to the sequence of 10 freeze thaw cycles while the modified board is not impacted.

It was also observed that the edges of the untreated boards started to be damaged (scaling) after the freeze thaw cycles.

Although not quantitative, this comparison clearly shows that admixing Z-6289 in FRC boards improves freeze thaw stability.

Resistance to QUV.

In order to assess the impact of UV radiation on the "hydrophobic performance" of Z-6289, reference and modified boards were placed in a QUV chamber for 500 hours.

A set of FRC boards modified with increasing content of Z-6289 (0, 0.1%, 0.25%, 0.5%, 0.75% and 1%) were compared.

Water absorption of the different boards was measured before and after QUV.

Results show that water uptake for a given immersion time does slightly decrease after QUV (graph given for an immersion time of 24 hours). This does demonstrate that the efficiency of the hydrophobic treatment is not modified by irradiation with UV light (no loss of performance is observed).

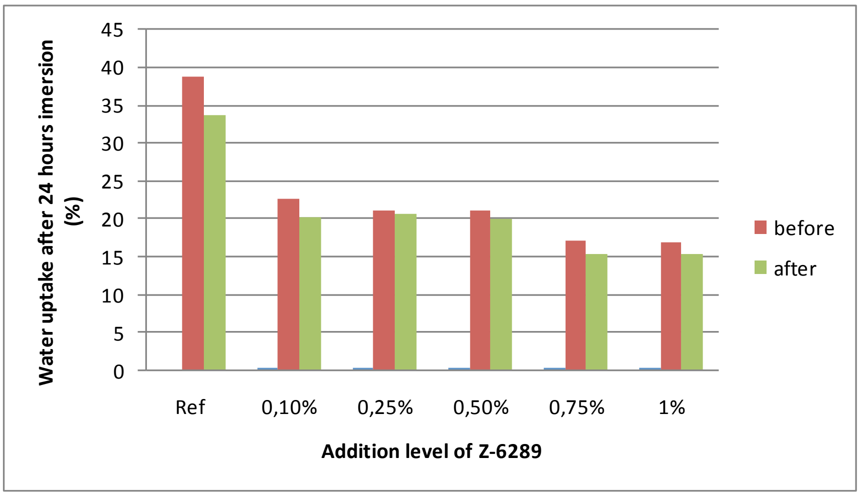


Figure 6 : water uptake of reference and modified boards after 24 hours BEFORE and AFTER 500 hours QUV at different Z-6289 addition level.

Impact on paint adhesion.

Adhesion of paint on reference and modified FRC boards was assessed using ISO 2409 test method (see experimental, rating from one to 5, the lower, the better)

Two different paints were used (named "Laque" and "Lasure").

The table gives the rating of the paint adhesion on the different boards (reference boards and modified boards with different Z-6289 addition content).

The table shows clearly the absence of impact of Z-6289 addition on paint adhesion.

Table 1 : rating of paint adhesion according to ISO 2409 test method on reference and modified FRC boards.

FRC (% of Z-6289 addition level)	Laque Rating	Lasure rating	Total of ratings
reference	2	2	4
0,10%	2	1	3
0,25%	1	2	3
0,50%	2	1	3
0,75%	2	1	3
1%	2	1	3

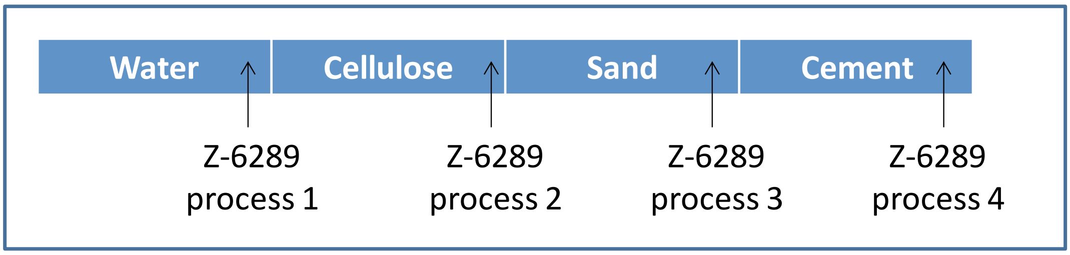
Impact of Z-6289 addition stage.

So far in this text, the Z-6289 was added at the latest stage of the sequence of components addition used for the making of the FRC boards.

Z-6289 was added at different stage of the FRC boards preparation. Addition level of 0.5% Z-6289 was used throughout this study on addition stage modification.

The boards were prepared according to the different processes (addition of Z-6289 at different stages as illustrated) then filtered, pressed and cured as described earlier.

Absorption of water by modified boards was assessed by water immersion test.



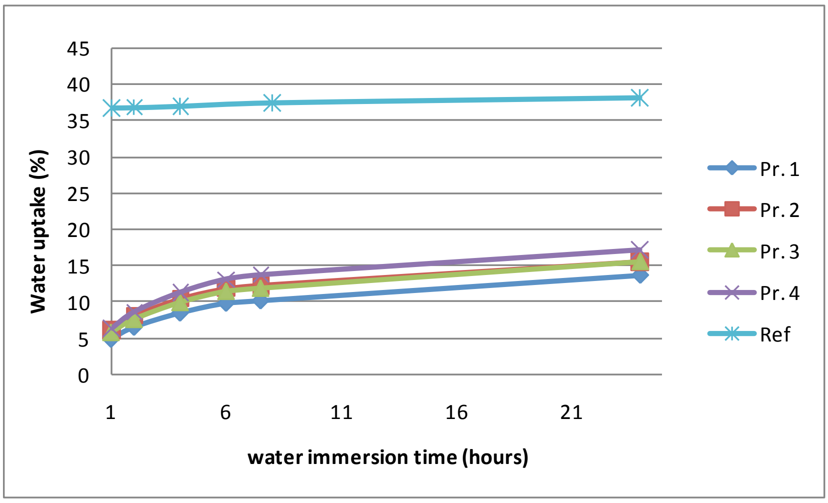


Figure 7 : water absorption as a function of immersion time of boards modified with 0.5% of Z-6289 added at different stages.

Results show that water absorption (and then the hydrophobic performance) is not very much impacted by Z-6289 addition stage. It can be however observed that water uptake might tend to decrease when Z-6289 is added at earlier stage of board preparation, suggesting mixing time of Z-6289 with board components needs to be long enough to insure transfer of hydrophobic active material on cement or cellulose.

Measurement of Z-6289 adsorption/reaction on FRC boards components

Results presented so far indicate that addition of Z-6289 silicone resin in a cement-cellulose slurry leads to a good hydrophobic treatment of the cement matrix. This means that a fair amount of the resin has adsorbed and reacted at the surface of cement particles or cellulose fibres.

An extensive set of testing was carried out to evidence if all Z-6289 was captured by the board components or if some could be flushed away with water during the filtration process.

At first, it was attempted to extract the silicone resin from the filtrated water and to quantify it by different analytical method. After several attempts, it was realised that some resin was trapped within the paper filter used in the Büchner funnel. This method was then considered as being inadequate for the purpose.

Finally, centrifugation of the slurry was used to separate solid and liquid phase of the slurry. Slurry made of 16g of CEM II cement, 2g of fine sand, 5g of wood pulp, 100g of water and different content of Z -6289 (from 0.05g up to 0.2g, corresponding to addition level vs dry FRC composition ranging from 0.25% up to 1.0%) were prepared as described above. Liquid and solid phase were extracted separately with hexane. Both hexane phases were collected and evaporated. Un-reacted Z-6289 was quantified by gravimetric measurements. The experimental method is illustrated in Figure 8.

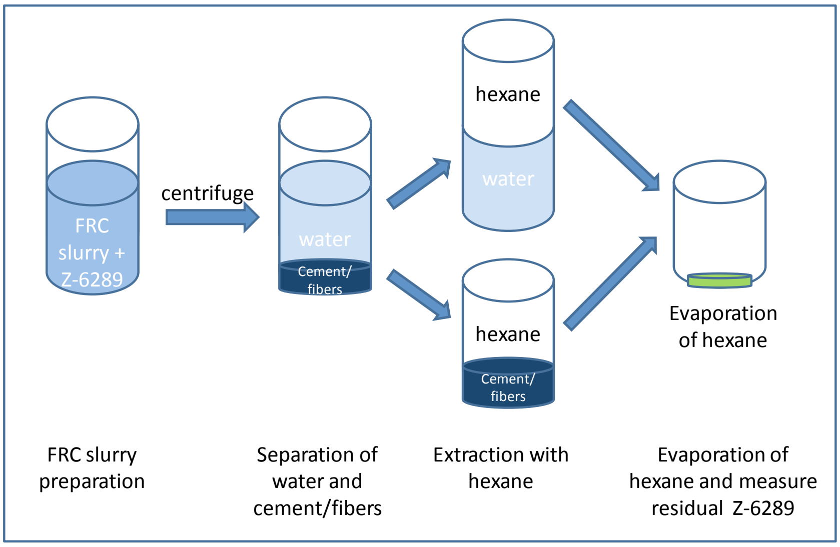


Figure 8 : schematic illustration of the extraction method used to quantify unreacted Z-6289 during preparation of modified FRC boards/

Detailed description of the experimental detail will be described elsewhere but it was evidenced that below addition level of 0.5% of Z-6289 vs the mass of the dry composition, virtually no Z-6289 could be detected in the liquid phase after centrifugation.

Once addition levels of Z-6289 above 0.5% vs dry composition were used, some residual Z-6289 was extracted from the liquid phase as illustrated in the following graphs.

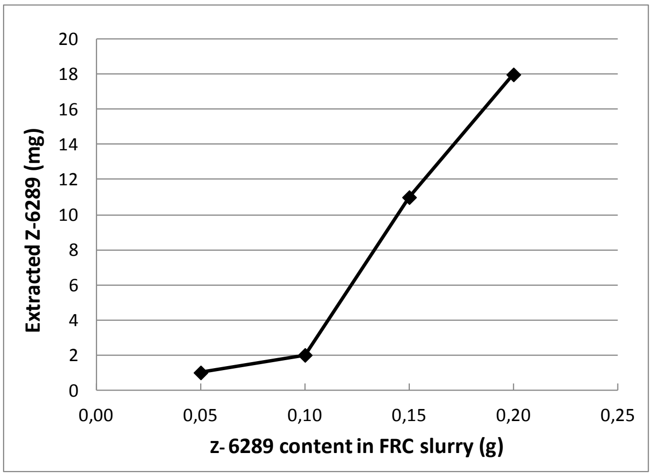


Figure 9: amount of Z6289 extracted with hexane as a function of content of Z6289 added in the FRC slurry.

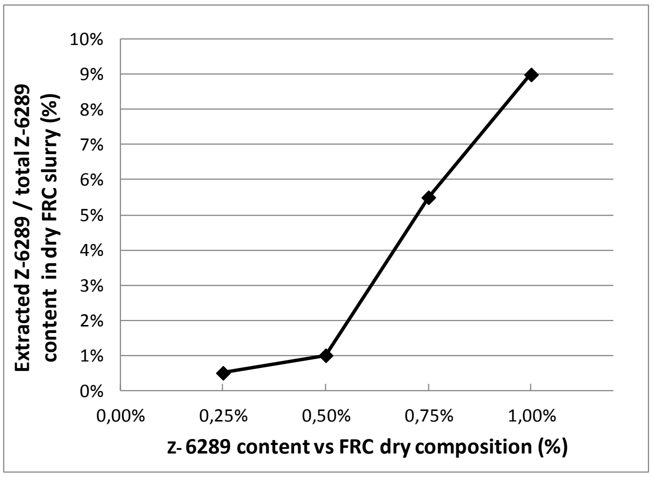


Figure 10 : percentage of extracted Z-6289 vs overall content of Z-6289 added in the FRC slurry as a function of Z-6289 content in FRC dry composition.

Figure 9 shows the amount of Z-6289 extracted with hexane as a function of the quantity of Z-6289 added in the slurry. Figure 10 shows the % of "lost" or "flushed" Z-6289 (vs content of Z -6289 added in the slurry) as a function of increasing content of Z-6289 added in the FRC dry composition.

Both figures clearly shows the important increase of unreacted Z-6289 once addition level vs dry FRC composition is above a threshold value of 0.5%. Below 0.5% of Z-6289, residual (or unreacted) Z-6289 is virtually zero, or at least, impossible to clearly measure with the method used (some transfer of cement particles in the hexane phase makes detection of very low content of Z-6289 very problematic).

This set of testing shows that addition level of Z-6289 vs dry FRC composition below 0.5% insures adsorption and reaction of Z-6289 on FRC components.

CONCLUSION

This work illustrates the strong positive impact of adding a silicone resin (Z-6289 in this case) as hydrophobic admixture in a cement matrix and more specifically in a fibre reinforced cement board.

This set of experiments clearly demonstrates that adding Z-6289 in a cement matrix decreases the tendency of the matrix to absorb water by capillarity.

The positive impact of admixing Z-6289 in FRC was clearly evidenced by properties such as resistance against freeze-thaw damage, resistance to QUV, lack of impact on paint adhesion. The study suggests as well that proper mixing time between Z-6289 and the FRC slurry should be insured to enable adsorption and reaction of the resin with the FRC components.

Extraction studies demonstrates that below addition level of 0.5 % vs FRC dry composition, all Z-6289 is adsorbed and reacted to FRC components.

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