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Effectiveness of Commercial Silicon Based Water Repellents at Different Application Conditions

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Abstract

Research on the application methodologies of silicon-based water repellents on building materials was carried out. From previous studies with model silicon compounds it was known that for volatile compounds the polymerization reaction is mainly influenced by the type of substrate and reaction conditions are not as critical. For non-volatile compounds reactivity increases with conditioning temperature, while the substrate plays a lower role. The present paper focuses on the effectiveness of commercial silicon based water repellents when applied at temperatures between 0 and 55 °C. Due to the complex nature of commercial products and the presence of crosslinking agents, a quantitative evaluation of the polymerization mixture could not be carried out as in the case of the model compounds. The effectiveness of the treatment is based on water absorption measurements carried out before and after artificial ageing. For some applications on limestone, the profile of the water contact angle was also followed during ageing in order to discuss its validity as a measure for the effectiveness of water repellent treatments. Results obtained with water repellent treatments have shown that for the case of brick a trend resulting in lower water repellent characteristics is observed for temperatures between 10 and 40 °C, with a minimum at 30°C. For limestone, the lowest effectiveness is obtained at 35°C. The results confirm the conclusions from previous research carried out with model compounds which showed that applications at a low temperature (0-5°C) did not necessarily result in a poorly performing treatment.

1 Introduction

Reactive silicon compounds are widely used to protect porous building materials against water uptake and damage caused by the constant exposure to moisture. Investigation of their use has shown that a successful performance is possible even after 35 years [1]. Studies of water repellents usually focus on the effectiveness of the treatment in relation to the type of substrate, the nature of the polymer, the concentration of the active compounds, etc [2-7]. Recently, a study on the type of interaction of reactive silicon compounds with brick and stone was carried out revealing the active participation of the substrate in the polymerization reaction [8,9].

However, less emphasis has been given to the influence of application conditions on the effectiveness of a water repellent treatment. In general, application is usually evaluated at standard conditions (20 °C and 50 % relative humidity (R.H.)), although it is well known that a polymerization reaction is influenced by the reaction conditions and that external application circumstances can vary considerably in practice. Hence, the question arises whether the performance of a treatment carried out at 20°C is comparable to one applied at 0º or 55°C.

The paper describes the results of an investigation on the application conditions of silicon-based water repellents on building materials. From previous studies dealing with model silicon compounds [10,11] it was concluded that for non-volatile compounds an increasing reactivity is obtained with increasing conditioning temperature and that the type of substrate has a lower influence. On the other hand, the polymerization of volatile compounds is mainly governed by the evaporation conditions and seems to be strongly influenced by the characteristics of the substrate. From the reactivity of both model compounds, it could be concluded that applications at lower temperatures did not necessarily result in poorly performing treatments. While for non-volatile compounds the lower conversion of the reactive groups increases upon further conditioning, for volatile compounds lower temperatures decrease the evaporation rate and further conditioning also results in an increased conversion.

This research is focused on the effectiveness of treatments with commercial silicon based water repellents conditioned at temperatures between 0 and 55°C. The applications were carried out using mixtures with the recommended concentration of active silicon compounds for an effective treatment. In addition, lower concentration mixtures were used in order to accelerate the ageing effect of the treatment. The influence of the moisture content of bricks on the effectiveness of the treatment is included for some application conditions.

Due to the complex nature of commercial products and the presence of cross-linking agents, a quantitative analysis of the polymerization product was not possible as was done for the model compounds. The evaluation of the effectiveness of

Brand name	Type of reactive group(s)	Type of hydrophobic function(s)	Diluent	Concentration of active compounds (w%)	Dry weight (w%)
Goldschmidt Tegosivin **HL100**	Ethoxy	Methyl	White spirit	10	9.0
				3	2.8
Wacker **290**	Methoxy	Methyl, octyl	White spirit	10	7.3
				3	2.4
Wacker **1311**	Methoxy, ethoxy	Methyl, octyl	Water	10	6.5
				3	1.9
Dynasilan **BSM40**	Ethoxy	Isobutyl	Ethanol	40	1.6
				10	0.5

Table 1: Properties of the commercial water repellents

the treatment is based on water absorption measurements with the Karsten pipe carried out before and after artificial ageing. For applications on brick, additional capillary water absorption measurements were made.

The beading effect after spraying water on a treated surface can be quantified by means of the contact angle, which is related to the solid-water interfacial tension by Young's equation [12]. Contact angle values are often used as performance [13] or as effectiveness criterion [7] for water repellent treatments. In this paper, the validity of the contact angle as a measure for the durability of water repellent treatments is discussed.

2 Experimental

2.1 Products

The properties of the commercial water repellents are presented in Table 1.

2.2 Substrates

The properties of the bricks and the limestones are presented in Table 2.

2.3 Preparation, treatment and conditioning

2.3.1 Bricks

The brick samples were cut cubes (5x5x5 cm^3) and the influence of their moisture content, at the time the water repellent was applied, on the performance of the treatment was investigated. For this purpose, two conditions were investigated: 10 °C/70 % RH and 30 °C/40 % RH. The moisture content for all treatments was cho-

Substrate	Real density (kg.m^{-3})	Porosity accessible to water (vol %)
Red brick	1853	21
Soft mud brick	1550	42
Euville	2233	16
Maastrichter	1400	49

Table 2: Real density and porosity accessible to water of bricks and limestones

sen below the critical moisture content to avoid application of the product onto wet surfaces. The critical moisture content is related to the endpoint of the first drying phase of saturated samples which proceeds by liquid water transport to the surface followed by evaporation [14]. For the calculation of the critical moisture content, a numerical evaluation of the drying curve was carried out [15,16].

A second set of brick samples was prepared where the specimens after drying were conditioned till constant weight—at 0º and 5 °C, both at 70 % RH, and 40º and 50 °C both at 15 % RH—before the water repellent was applied by capillary absorption during 10 seconds. This was followed by 1-week conditioning at the same temperature and RH.

2.3.2 Limestones

Samples of 5 x 5 x 2 cm^3 were dried at 60 °C to constant weight. The treatment was carried out by capillary absorption during 10 seconds, followed by a conditioning for 1 week at following temperatures: 5º, 10º, 20º, 35º and 55°C all at 65% RH.

2.4 Artificial ageing

The ageing program used for 20 years in the laboratory to study the effectiveness of water repellents is in accordance to SAE J 1960 [6]. The total artificial ageing procedure takes 1008 hours. Research on the performance of treatments in practice has shown that this artificial ageing is equivalent to 20 or 30 years of natural ageing.

2.5 Effectiveness measurements

The waterabsorption measurements with the Karsten pipe are in accordance with the RILEM Recommendations, as noted in the 25-PEM-document of RILEM. The results are expressed as $_{15-5}$, being the difference in water level of the pipe after 15 and 5 minutes, or as percentage effectiveness according to the equation (1):

$$\text{Effectiveness (\%)} = (\Delta_{15\text{-}5,\ \text{untreated}} - \Delta_{15\text{-}5,\text{treated}}) \times 100 / \Delta_{15\text{-}5,\text{untreated}} \quad (1)$$

The capillary water absorption coefficient (CWA) has been measured on bricks according to RILEM-UNESCO 1978, II.6. The measurements are carried out on the same test surface as used for the measurements with the Karsten pipe.

2.6 Contact angle measurements

Contact angle measurements are carried out with a contact angle goniometer (Ramé-Hart 100-00). On each test surface, the average value of 10 measurements is obtained.

3 Results and Discussion.

3.1 Results on brick

3.1.1 Consumption

The consumption of the water repellent products for bricks with a different moisture content is presented in table 3

Generally, the red brick absorbs more water repellent than the soft mud brick. Silicon compounds diluted in organic solvents show higher consumption than the aqueous emulsion (W 1311).

In general, an increase in moisture content of the bricks causes a decrease in the uptake of organic solutions. The same tendency, but more marked is observed for the W1311 treatment on the soft mud brick. For the red brick, the consumption of W1311 increases with increasing moisture content.

3.1.2 Influence of the moisture content on the effectiveness of the treatment

With the exception of 2 treatments carried out on dry soft mud brick, all treatments showed initially a $\Delta_{15\text{-}5}$ lower than 0.5 ml. Taking into account that the $\Delta_{15\text{-}5}$ of untreated samples is 8.6 ml, this corresponds to an effectiveness of at least 94 %. Artificial ageing results in an increase of the $\Delta_{15\text{-}5}$ for some samples. Figure 1 shows the plot of the initial CWA and the $\Delta_{15\text{-}5}$ after ageing as a function of the moisture content of the tested bricks.

No clear influence of the moisture content of bricks at the time of application could be established for the water absorbing properties of the treated samples. For the tested range of moisture content, some samples show a high initial CWA, whereas in all cases almost no water is absorbed during the test with the Karsten pipe. Generally, the samples showing a $\Delta_{15\text{-}5}$ of at least 0.8 ml after ageing are characterised by a high initial CWA.

The lack of a clear influence of the moisture content of bricks on the effectiveness of the treatments made us decide to restrict the investigation program of limestone to applications on dry samples only.

Substrate	Moisture content (w%)	Consumption (g.m^{-2})			
		G HL100	W 290	W 1311	D BSM40
Red brick	0–1	685	734	188	654
	1-5	545	584	247	421
Soft mud brick	0-1	513	402	207	475
	1-5	494	391	98	333
	5-8	340	327		342

Table 3: Consumption of commercial water repellents on bricks containing a different moisture content.

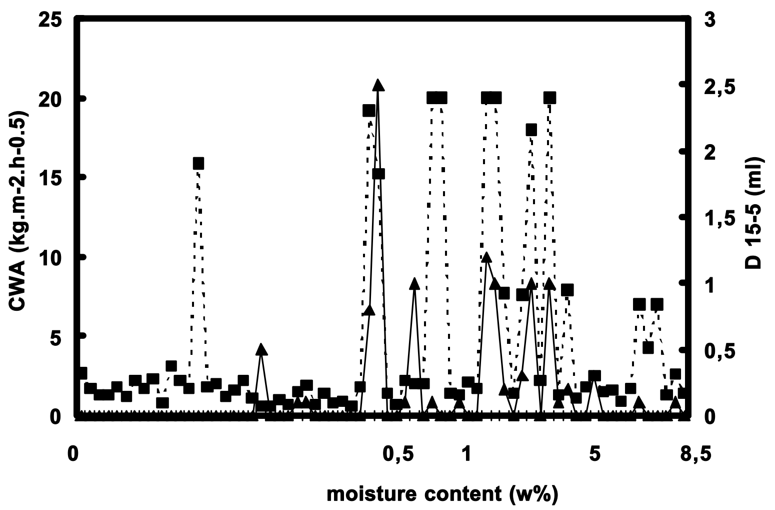

Figure 1: plot of the initial CWA (- -) and the D $_{15\text{-}5}$ after ageing (--) as function of the moisture content of bricks.

3.1.3 Influence of the conditioning temperature on the effectiveness of the treatment.

Figure 2 presents the plot of the average initial CWA and the average $\Delta_{15\text{-}5}$ after artificial ageing of treated bricks as function of the conditioning temperature.

Both measurements reveal higher water absorptions for conditioning temperatures between 10º and 40°C with a maximum at 30 °C.

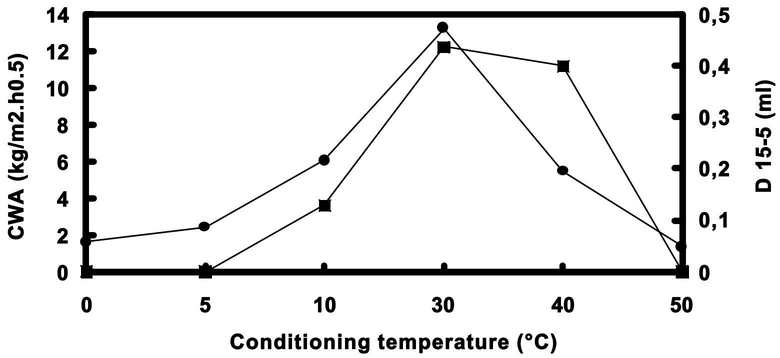

Figure 2: plot of the average initial CWA (●) and the average $D_{15\text{-}5}$ after ageing (■) of treated bricks conditioned at different temperatures.

Substrate	Consumption (g.m^{-2})			
	G HL100	W 290	W 1311	D BSM40
Maastrichter	3247	3070	4164	3377
Euville	375	380	410	394

Table 4: Consumption of water repellents on Maastrichter and Euville.

3.2 Results on limestone

3.2.1 Consumption

The consumption of the water repellents on Maastrichter and Euville limestones is presented in Table 4.

The consumption of the water repellents is about 10 times higher on Maastrichter than on Euville and for both limestones, the highest consumption is obtained for W 1311.

3.2.2 Influence of the conditioning temperature on the effectiveness of the treatment

With one exception, all treatments carried out on Maastrichter show excellent water repellent characteristics after ageing.

The average effectiveness of water repellent treatments on Euville as function of the conditioning temperature is presented in Figure 3.

In accordance with previous investigations [3-6], these results show that lowering the concentration of active compounds decreases the effectiveness of the treatment. Treatments carried out using water repellents with a dry weight between 6.5 and 9.0 % w/w perform perfectly before ageing, regardless of the conditioning temperature. When lower concentration solutions were used some samples show water absorption, especially when conditioned at 20°C. The influence of the active com-

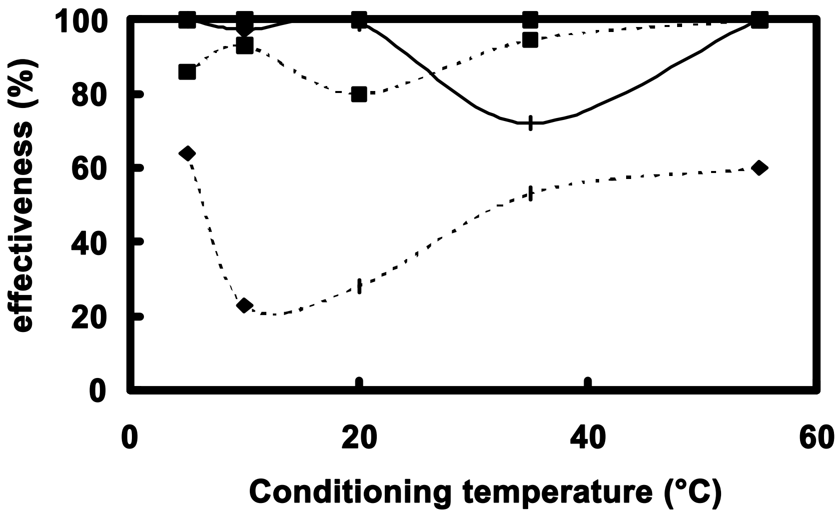

Figure 3: Average effectiveness of water repellent treatments on Euville before (■) and after (♦) artificial ageing as function of the temperature of conditioning.

— : water repellents with a dry weight between 6.5 and 9.0 w%

--- : water repellents with a dry weight between 1.6 and 2.8 w%

pounds concentration on the effectiveness of the treatment is more important after ageing. For water repellents whose dry weight ranges between 6.5 and 9.0 % w/w, the effectiveness varies between 100 and 74 %, the latter corresponding to a conditioning temperature of 35 °C. The effectiveness of treatment with the same water repellents but having a dry weight below 3 % decreases for all conditions tested. The lowest results were obtained for conditioning at 10º or 20°C. As a consequence, the influence of the concentration of active compounds on the effectiveness of the treatment is more important at 10 °C than at 35°C.

3.3 Contact angle measurements

As an example, Figure 4 presents a plot of the contact angle and of the effectiveness of a treatment with G HL100 on Euville, conditioned at 55 °C, during artificial ageing.

The contact angle of the Euville treated with G HL100 diluted to 10% decreases during ageing from 127º to 102°. No water is absorbed when evaluating effectiveness with the Karsten pipe before, during and after artificial ageing. When a 3% solution of G HL100 is applied, the plot of the contact angle as function of ageing is quite similar, showing that the treatment appears hydrophobic. However, the effectiveness of the treatment drops from 100 to 3 % when evaluating it with the

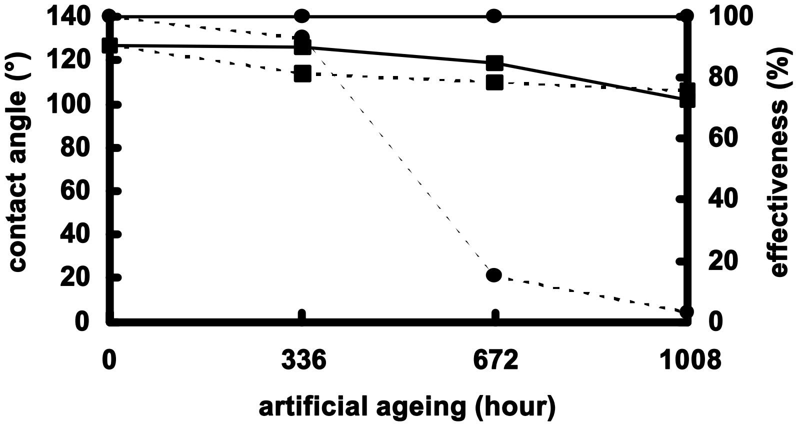

Figure 4: contact angle (■) and effectiveness (●) of a treatment with a 10 % (—) or a 3 % (---) solution of G HL100 on Euville during artificial ageing (conditioning temperature: 55 °C).

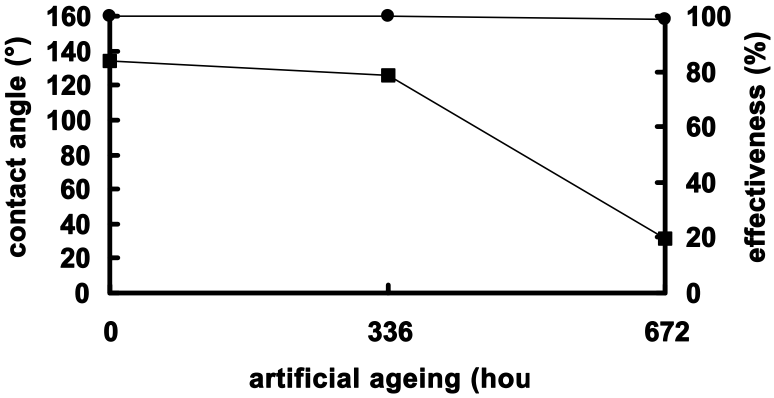

Figure 5: contact angle (■) and effectiveness (●) of a treatment with D BSM40 on Maastrichter during artificial ageing (conditioning temperature: 35 °C).

Karsten pipe. After ageing the absorbed amount of water approaches the value obtained before treatment although the corresponding contact angle reveals water repellency.

The plot of the contact angle and of the effectiveness of a treatment with D BSM40 on the Maastrichter limestone conditioned at 35 °C, is presented in Figure 5.

The phenomenon shown in Figure 5 is the opposite of what was illustrated in Figure 4. The contact angle drops from 134 to 31° during ageing, while the effectiveness of the treatment, as measured with the Karsten pipe, remains 100%.

4 Conclusion

The influence of application conditions of commercial silicon-based water repellents on brick and limestone has been investigated. The treatments were carried out using mixtures at the recommended concentration for good performance as well as at 70% lower concentrations in order to accelerate the ageing procedure. The influence of the moisture content of the bricks, at the time the product was applied, on the effectiveness of the treatment has also been studied. In all cases, the moisture content when the treatment was applied was lower than the critical moisture content to avoid applications onto a wet surface.

Although some treated bricks show an initial high CWA, evaluation with the Karsten pipe showed no water absorption, sometimes even after ageing. This indicates that a high initial CWA does not necessarily indicate a poor durability of the treatment. Both the initial CWA as well as the measurements with the Karsten pipe after ageing reveal a lower effectiveness for application temperatures between 10 and 40°C for some treatments. All treatments carried out at low (0-5°C) or high temperature (50°C) show an excellent water repellent behaviour.

There is no clear influence of the moisture content of bricks, at the time of treatment application, on the water absorbing properties of the treated samples. For the range of moisture content tested, some samples show high water absorbing characteristics.

Since the Maastrichter limestone shows a high consumption of water repellents, almost all treatments show excellent hydrophobic characteristics. Contrariwise, the results for the Euville limestone show a decrease in effectiveness with decreasing dry weight of the water repellent product. The decreased effectiveness is even more manifested after artificial ageing. For products with a dry weight between 6.5 and 9.0 % w/w, the lowest effectiveness after artificial ageing is obtained at 35 °C. For water repellents having a dry weight below 3 % w/w, the lowest results are obtained at 10 or 20°C. Consequently, the influence of the concentration of active compounds on the effectiveness of the treatment is more important at 10 °C than at 35°C.

The results obtained from this investigation confirm the conclusions from the research carried out with model compounds: treatments carried out at a low temperature do not necessarily result in a poor performance. In addition, the results confirmed good performance for conditioning up to 55°C.

The effectiveness of treatments carried out at a low temperature can be explained by the low evaporation rate of the active and volatile compounds. Lower results obtained between 10º and 40°C can be explained by an increase in the evaporation rate of volatile compounds which might not be compensated by a similar increase in reactivity of the reactive groups. Higher application temperatures (50º – 55°C) cause an increase in the polymerisation rate resulting in an increased reduction of the evaporation rate of volatile compounds.

The validity of the contact angle for the evaluation of the effectiveness of the treatment has been investigated by comparison with water absorption measurements using the Karsten pipe during artificial ageing.

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