

Water Uptake and Scaling of Impregnated Pavement Concrete Under Freeze Thaw and De-Icing Agent Attack

A. Frentzel-Schirmacher

F.A. Finger-Institute for Building Materials Sciences
Bauhaus-University Weimar / Germany

Abstract

The paper deals with the problem of protecting young concrete pavement against freeze thaw and de-icing agent attack. Especially pavements cast in autumn may react sensitively to frost attack in combination with de-icing agents during the first winter. The application of hydrophobic agents should be used as a precaution to minimize water uptake and salt transport into the relatively young and immature concrete. The focus of the investigation was to understand the influence of different pre-conditioning storages, age of application and application procedures on the reduction of the water uptake during freeze thaw and de-icing agent attack as well as their influence on the amount of scaling. The freeze thaw and de-icing agent attack were simulated by CDF-test procedure. Independent of the type of the water repellent agent, the application procedure and the pre-conditioning storage only the solution uptake under isothermal conditions could be reduced satisfactorily. With start of freeze thaw attack (FTW) an intense pumping effect during thawing periods leads to an additional solution uptake equal to non-impregnated concrete. Due to this additional solution uptake scaling could not be prevented continual. An initial period of 28 freeze thaw cycles (FTC) with a clear reduction of scaling was followed by a progressive scaling period. Continuing freeze thaw attack led finally to an exceeding of the scaling amount compared to the scaling of an untreated pavement concrete. A secure and continuing improvement of horizontal, coarse pavements against scaling caused by freeze thaw and de-icing agent attack could not

be proved. For vertical walls and other structural constructions that allow a fast run off of the de-icing solutions the observed scaling behaviour should not be relevant in the field.

1 Introduction

Especially concrete which has been cast in autumn may behave sensitively to frost attack in combination with de-icing agents during the first winter. Therefore the German guidelines for the construction of concrete pavements [1] recommend an application of hydrophobic impregnations if de-icing agents have to be applied on the road surface during first winter. The application of hydrophobic agents should be used as a precaution to minimize water uptake and salt transport into the relatively young and immature concrete.

With regard to this special application the following specific qualities of pavement concretes and environmental conditions have to be considered:

Concrete used for pavements is dense with a low w-c ratio between 0.4 and 0.45. Due to 38 % of water related to the dry mass of cement are bound chemically or physically during the hydration process [2], the content of capillary pores as space for transport and chemical reaction of the active substances are very low, in the relatively young concrete as well.

The curing and treatment for this special application takes place under relatively low temperatures. Weather data collected over 5 years at the F.A. Finger-Institute [3] (located in Middle Germany) showed average temperatures during November nights of 3 °C and minimum temperatures of – 6 °C. In December the average night temperature dropped down to 0 °C. The minimum temperature was -13 °C. Several freeze – thaw cycles were observed.

A further and most important difference to the usual application of impregnation substances is the horizontal orientation of the surfaces. Therefore, water and salt solutions remain on the surface for relatively long periods of time.

Truck traffic causes additional pressure along the wheels' paths. According to statistic reports [4] the average practical load at German expressways is about 7200 trucks daily. That is one truck every 12 seconds at a counting point. Trucks normally have a usual weight between 7 - 10 t per axle. That means water on the surface along the wheels paths is pressed with a maximum contact pressure of 0.74 N/mm² [5] by a wheel into the surface layer. In order to understand the influences on the above-mentioned specific qualities, the samples were preconditioned according the described influences. Furthermore, the age of the concrete, the amount of the hydrophobic agent applied and the application technology were varied. The influences caused by traffic loads cannot be presented within this paper.

Table 1: Used concrete composition

Cement content:	359 kg/m ³
Water content:	147 kg/m ³
w-c ratio:	0.41
Artificial air pore content:	5.0 % +/-0,3 %
Compacting factor:	1.30 +/-0,2
Sieve curve of aggregates:	½ AB
Maximum grain size:	22 mm

Table 2: Characteristic of the used water repellent agents

Code	Active substance	Content of active substance [%]	Consistency	Solvent	Coverage [g/m ²]
B	Octyltriethoxysilane	100	liquid	-	200
C	Octyltriethoxysilane	80	cream	water	200

2 Materials

In order to understand the influence of a hydrophobic treatment on a realistic dense concrete with a low capillary pore content, a pavement concrete mixture was used given in table 1.

For the tests presented here, two different commercial water repellent agents were used. Table 2 gives an overview about the type and the content of the active substance, the solvent used and an average consumption for dense concretes given by the manufacturer.

3 Test procedures

Two different test procedures were carried out in order to examine the specific investigational aspects. At least three replicate specimens were used for each test method.

3.1 Influence of environmental conditions

In order to examine the environmental aspect a special pre-storage regime, based on weather data collected at the institute, was developed. Table 3 gives an overview about the sequential climatic conditions. To simulate fall placing conditions in the laboratory all concrete components were prechilled at a temperature of 5 °C. After mixing, the fresh concrete had a temperature

Table 3: Pre-storage regime "Autumn - AC"/ "Normal -NC" and main test (CDF)

Climatic conditions	Time	Temperature regime	Moisture regime
Pre-storage "Autumn - AC" (1 - 28 d)			
27 cycles á 24 h	0 - 6 h	+ 10 °C	95 % RH
	6 - 14 h	+ 5 °C	-
	14 - 15 h	+ 5 °C ---> - 10 °C	-
	15 - 17 h	- 10 °C	-
	17 - 18 h	- 10 °C ---> + 10 °C	-
	18 - 24 h	+ 10 °C	95 % RH
Pre-storage "Normal - NC" (0 - 28 d)			
No cycling	0-28 d	20 °C	65 % RH
Pre-storage "Standard" (1 - 28 d)			
Curing	1 - 7 d	20 °C	Water
Storage	7 - 28 d	20 °C	65 % RH
Freeze thaw and de-icing agent attack according CDF-test			
28 - 35 d capillary suction	0 - 7 d	20 °C	Storage up site down in test solu- tion (3 % NaCl)
35 - 63 d	0 - 4 h	20 °C ---> - 20°C	
Freeze thaw attack (FTW)	4 - 7 h	- 20 °C	
56 FTW (2 FTW /d)	7 - 11 h	- 20 °C ---> 20 °C	
	11 - 12 h	20 °C	

of about 10 °C. Specimen with a size of 80 x 80 x 250 mm³ were cast. During the first 24 hours the moulds with the young concrete were stored at 10 °C and 95 % RH and protected against water loss by covering with plastic foils. Afterwards the specimens were de-moulded, the lateral surfaces as well as the bottom of the specimens were sealed with adhesive butyl rubber tape and exposed subsequently to the pre-storage regime “AC-Autumnal climate” as described in table 3. During the next 6 days the surfaces were cured with moist clothes and plastic foil.

A second pre-storage regime with normal casting conditions at 20 °C and constant pre-storage conditions “NC – Normal climate” (20 °C and 65 % RH) as well as a standard pre-storage regime “S - Standard” (6 days water storage and 21 days storage at 20 °C and 65 % RH) were used for comparison. “Normal climate” specimens were cured with moist clothes up to the 7th day like the autumnal stored specimens. Lateral surfaces of “Standard”-specimens were glued with butyl rubber tape at an age of 27 days, one day before CDF-testing [6, 7] started. As a result this specimens got a very good curing and a more intensive drying during the whole period of pre-storage.

The surface treatment materials were applied 14 days after casting with coverage of 200 g/m² for all agents as required by the manufacturer. The coverage was controlled by the weight of the applied surface treatment materials using an electronic balance with an accuracy level of 0.1 g. The average penetration depth was determined at extra specimens on ten points at the broken surface after water spraying. For selected specimens (series with water repellent agent C) penetration profiles by means of FT-IR-spectroscopy [8] could be determined additionally.

During isothermal capillary suction and freeze thaw attack by CDF-test the NaCl-solution (3 %) uptake of the specimens was observed continuously by determining their changes in weight. During freeze thaw testing the surface scaling versus the number of freeze thaw cycles was determined. At the end of test the specimens were re-conditioned for 4 weeks at 20 °C and 65 % RH. Finally the remaining resistance against isothermal capillary water suction versus unstressed specimens of same age and same pre-conditioning was determined.

3.2 Influence of application procedure

The same procedure for the investigation after “normal climate” storage was generally also used for the examination of the application procedures with the exception of the time schedule. The only exception was the curing and sealing of the reference series “7 days wet cured”. For this test the curing as

well as the sealing was carried out like for the treated specimens in order to realise a comparable microstructure of the surface near zone due to equal drying conditions.

In order to examine the influence of the application procedure, the procedure and the time schedule were varied as follows: single application with 200 g/m² and double application with a second application of 200 g/m² 7 days after first application. First application for both variants was carried out 6 weeks after casting. The CDF-test with 56 freeze thaw cycles started 4 weeks later. All carried out observations during CDF-testing were equal to the aforementioned ones. Drying of specimens was controlled by determining the loss of weight during a 4 weeks lasting pre-conditioning period at 20 °C and 65 % RH. Afterwards the remaining resistance against capillary isothermal water uptake was determined. A second CDF-test with 28 freeze thaw cycles and a further observation of the resulting drying behaviour was carried out after a further re-conditioning period of 4 weeks. Penetration profiles by means of IR-spectroscopy could be derived for all series. The determination of the penetration profiles was carried out at the Forschungszentrum Karlsruhe. The author wants to thank Professor Gerdes for his support of this work.

4 Results and discussion

4.1 Influence of environmental conditions

The test results with respect to the environmental aspect are shown in figures 1 to 3. As can be seen in figure 1 the low temperatures during pre-storage and application did not affect the capillary suction of the isothermal pre-saturation negatively; all treated specimens showed an equally good decrease of the solution uptake.

However, within the first freeze thaw cycle the effectiveness of all agents disappeared and test solution was sucked through the impregnated surface layer in the same manner as through the non-treated surface layers of the standard specimens. The water repellence disappears as long as freezing and thawing continues.

In spite of the clearly lower total-solution uptake the surface scaling could not be reduced permanently as can be seen in figure 2. A reduction of the surface scaling could only be reached up to the 28th freeze thaw cycle. After the 28th freeze thaw cycle surface scaling increased progressively and exceeded the amount of scaled material of the non-treated specimens at the test end. The surface did not scale homogenously over the whole surface but in form of pop-outs of the matrix and partially of the aggregates.

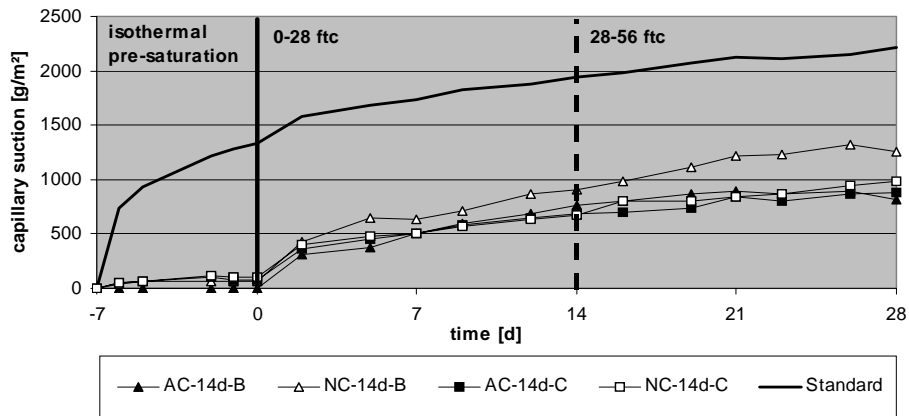


Figure 1: Capillary suction during isothermal pre-saturation and freezing and thawing according to CDF-procedure (variation of pre-conditioning)

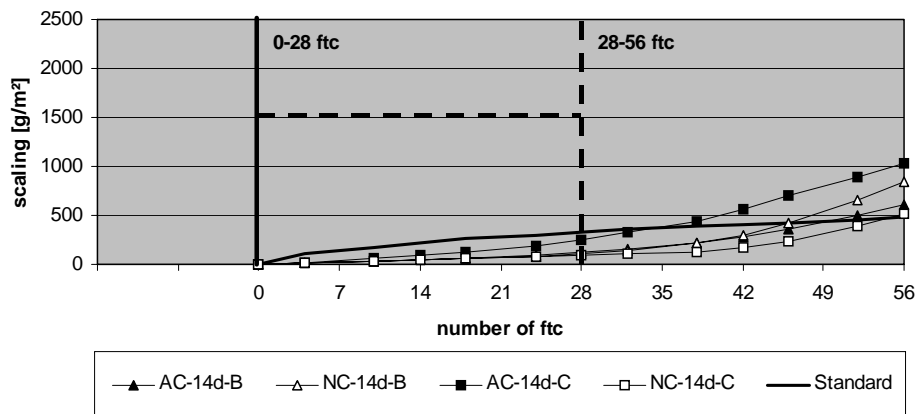


Figure 2: Scaling during CDF-testing after different pre-conditionings

That's why a calculation of an average scaling depth and loss of thickness of the treated surface zone was not possible.

The visually determined average penetration depths at the broken surfaces shown in table 4 illustrate additionally the independent effectiveness of a hydrophobic treatment from the given pre-storage conditions. In all cases the penetration depth of the "autumnal" stored series exceeded slightly the penetration depth of the "normal" stored series. Because of the high density

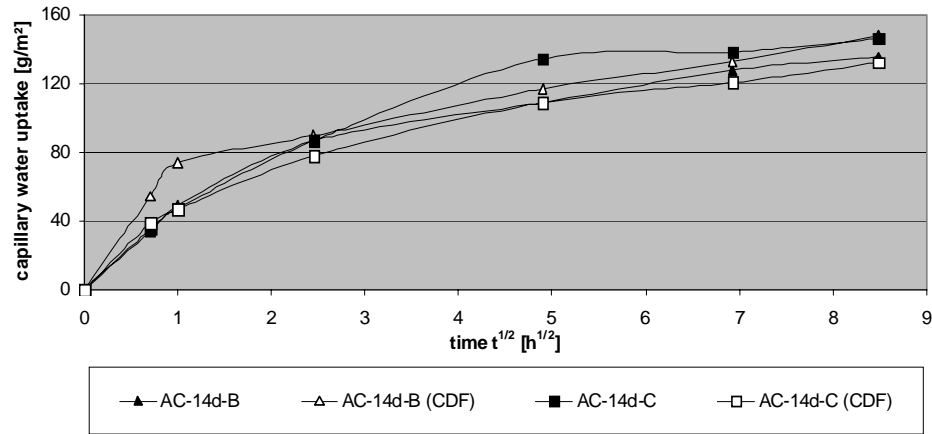


Figure 3: Comparison of capillary water suction of specimens after CDF-testing and specimens without freeze thaw attack (variation of pre-conditioning)

Table 4: Visual average penetration depth before and after CDF-test

	Visual penetration depth [mm]			
	Before CDF-test		After CDF-Test	
	NC	AC	NC	AC
B	6	9	4	6
C	3	5	2	3

of the concrete, the achieved depth was relatively low. The determination of the penetration profiles by means of IR-spectroscopy, which were carried out for example for series C showed even a relevant active substance content only within the first millimetre for both pre-storage conditions.

Despite the unsatisfactory penetration depths, the full effectiveness of both water repellent agents and both pre-conditioning methods was regained after reconditioning the test samples after CDF-test. A comparison of the capillary water uptake of specimens without and after exposure freeze thaw attack respectively is given in figure 3. It can be concluded that the effectiveness of a water repellent treatment after an intensive freeze thaw attack connected with medium-high scaling between 500 and 1000 g/m² is not diminished. If the concrete gets the possibility to dry out, the water repellence under isothermal conditions returns.

4.2 Influence of application procedure and age of concrete

The distribution of the active substance, shown in figure 4, could be improved in comparison to a 14 days application by an application at a good dried surface after a 42 days lasting storage at 20 °C. A further improvement of the active substance content within the first 4-5 mm could be reached by a second application of further 200 g/m² one week after first application. Furthermore the decrease of the active substance content after a double application started about 1 mm deeper than for the single application. The visibility of the water repellent treatment to the naked eye reached an active substance content of less than 0.1 wt-% in all cases.

The capillary NaCl-solution uptake during pre-saturation of the CDF-test under isothermal conditions, shown in table 5, could be reduced about 70 % in which the liquid water repellent agent reached a slightly higher reduction. Despite the improved penetration depth and active substance content in the surface zone additional water uptake with the start of freezing and thawing could neither be prevented (see figure 5 in comparison with figure 1) nor reduced. As for the series after different pre-conditionings NaCl-solution was sucked through the impregnated concrete zone in the same manner as an untreated concrete zone. Thus the penetration depth as well as the type and the content of the active substance do not affect the suction of the test solution under freezing and thawing conditions.

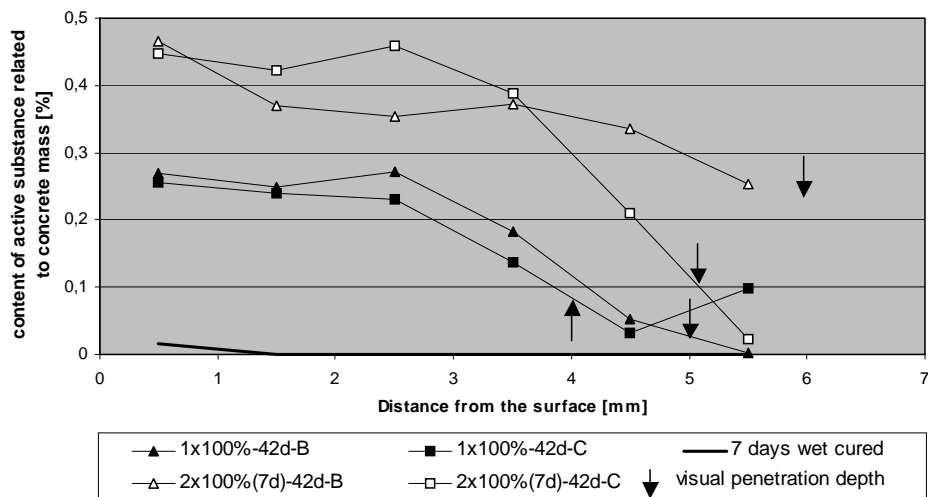


Figure 4: Comparison of penetration profiles measured by means of FT-IR spectroscopy and visual determined penetration depths at broken surfaces

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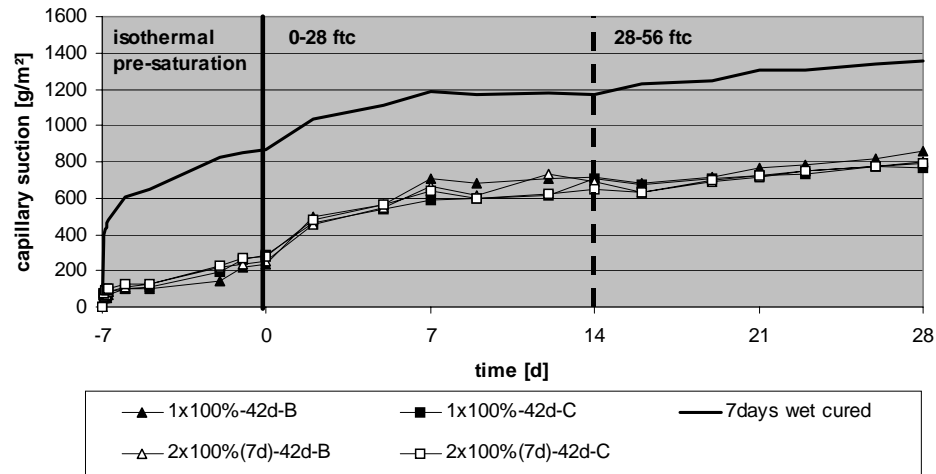


Figure 5: Capillary suction during isothermal pre-saturation and freezing and thawing during first CDF-test (variation of application procedures)

Table 5: Comparison of capillary suction (g/m²) during isothermal pre-saturation of CDF-Tests and re-saturation between both freezing and thawing tests (results in brackets illustrate the reduction of solution uptake in %)

	Solution	Age of specimens [d]	7days wet cured	1x100%-42d-B	1x100%-42d-C	2x100%(7d)-42d-B	2x100%(7d)-42d-C
Isothermal pre-saturation of 1. CDF	NaCl (3%)	71	867 (0)	233 (73)	283 (67)	250 (71)	275 (68)
Re-saturation after first drying period	Water	140	602 (0)	203 (66)	205 (66)	208 (65)	190 (68)
Isothermal pre-saturation of 2. CDF	NaCl (3%)	176	617 (0)	233 (62)	233 (62)	242 (61)	217 (65)

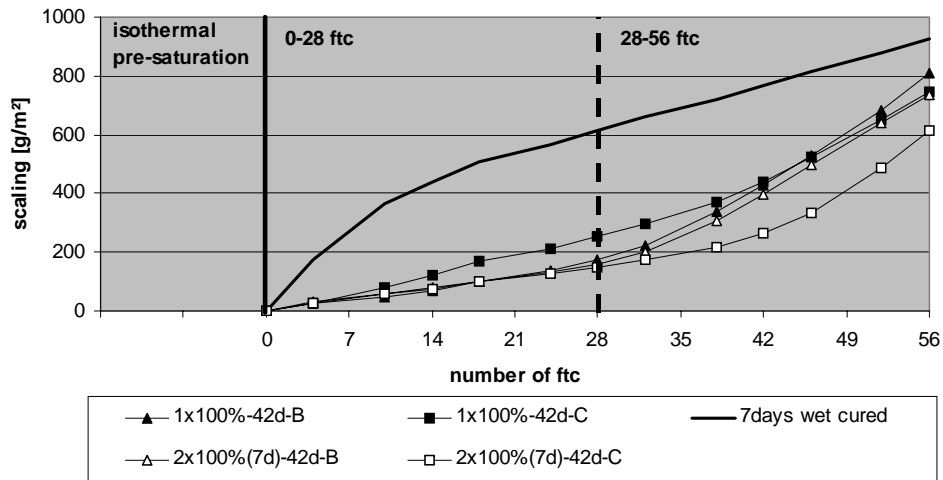


Figure 6: Scaling during first CDF-test (variation of application procedures)

Figure 6 illustrates the scaling resulting from the solution uptake during freeze thaw attack. As before mentioned the amount of scaling was significantly reduced up to the 28th freeze thaw cycle. After 28th freeze thaw cycle the scaling behaviour changed and a progressive scaling at the surface of all treated series started as observed before. The amount of scaling did not exceed the amount of scaling of the untreated series after 56 freeze thaw cycles. This is not attributed to an improvement of the scaling behaviour, which is nearly equal, but to the higher scaling of the untreated series during first freeze thaws. The obtained higher scaling is caused by the less effective curing by wet clothes in comparison to a standard curing as well as by the better drying conditions due to the unsealed lateral surfaces. After scaling of the outermost weak surface zone, which was directly influenced by the curing, the scaling was noticeably reduced. After 14th freeze thaw cycle the scaling curve proceeded linear as it does usual for good cured Portland cement concrete. So it must be concluded that the curve progression of solution uptake as well as of the scaling could not be influenced by an improved impregnation nor was it dependent on the type of the water repellent agent.

After a 4 weeks long re-conditioning the remaining effectiveness of all series were tested. Table 5 shows for all series a slight reduction of the water uptake. Thus it appears that the effectiveness of all treatments despite scaling amounts between 600 and 800 g/m² was still completely given. The reduced water uptake of the untreated series has to be traced of the scaling

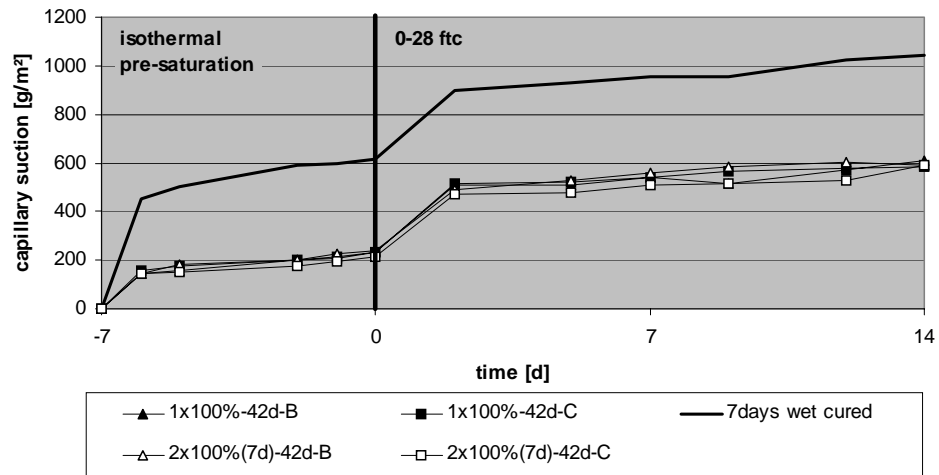


Figure 7: Capillary suction during isothermal pre-saturation and freezing and thawing during second CDF-test (variation of application procedures)

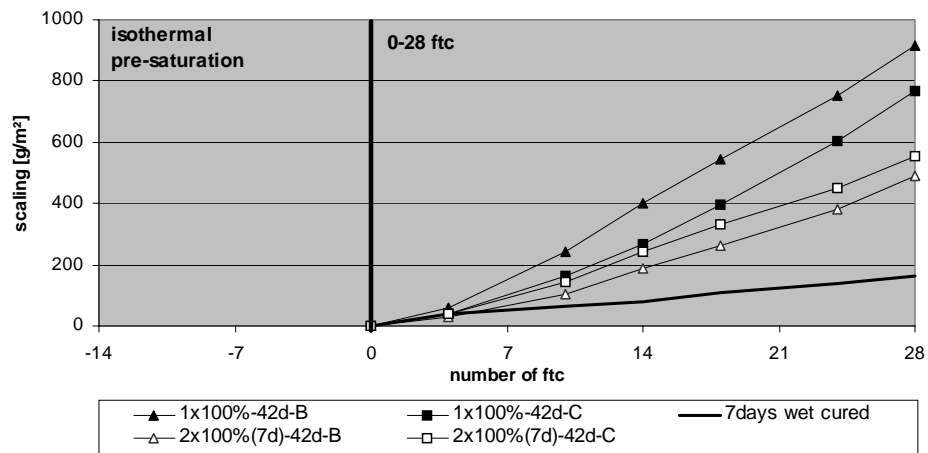


Figure 8: Scaling during second CDF-test (variation of application procedures)

of the suboptimal cured outermost surface zone during first 14 freeze thaw cycles.

After a further re-conditioning a second CDF-test with 28 freeze thaw cycles was carried in order to investigate the scaling behaviour after drying periods. Curves of solution uptake and scaling are shown in figures 7 and 8.

No changes could be observed with regard to the solution uptake, even though a significantly different scaling behaviour was observed. While the untreated series scaled linearly and very slowly, all specimens of the treated series showed right from the start an intense progressive scaling. In which the progress of the double treated series was noticeable less intense than the single treated series. No influence of the type of the active substance could be observed.

It must be also assumed that after further drying periods this intense scaling continues as long as active substance remains in the concrete.

5 Discussion

The influence of different pre-conditioning storages, age of application and application procedures at dense pavement concrete on NaCl-solution (3 %) uptake and scaling under freeze thaw attack has been investigated.

Test series under different pre-conditioning storages showed the well known importance of a good dried surface in order to achieve a distinct water repellent lined surface zone. Two weeks of pre-storage has to be assessed as not sufficient. Distinct penetration depths of the active substance could be obtained no more than after 42 days of pre-storage. Further improvement of the penetration depth could be obtained by a double impregnation.

As also observed with brick and limestone by De Clercq and De Witte [9] low application temperatures do not reduce the effectiveness of a water repellent treatment necessarily. These observations could be confirmed for concrete. The application of the water repellents at 10 °C with subsequent changing temperatures between -10 °C and 10 °C did not affect the effectiveness of the treatment in comparison to impregnated specimens, which were stored at 20 °C.

The achieved test results showed the wide applicability of the water repellent agents also under harsh application conditions as well as the comparable effectiveness of the used active agents. However, the results illustrated also the problem of impregnation of relative dense concretes, which have a low content of capillary pores as space for chemical reactions of the active agents with cement paste.

Independent of penetration depths, active substance distribution and pre-storage conditions comparable reductions of solution uptake under isothermal conditions could be obtained. However, with the start of the freeze thaw cycles, additional solution was pumped into the concrete. This pumping effect, described by Setzer [10] could not be prevented by any of the investigated variations of water repellent treatments. Setzer describes frost

action as a very effective pump. Drop in temperature under 0 °C leads to an increasing difference of pressure between unfrozen water and ice of 1.22 mPa/K. This effect causes a contraction of the cement matrix during freezing because of water transport from the matrix to existing macroscopic ice formations. During thawing the decreasing difference of pressure leads to an expansion of the cement matrix. Due to the macroscopic ice in concrete pores is still frozen; liquid water is sucked by concrete from outside. The observed suction during thawing was completely independent from the type of active substance, the conditions during pre-conditioning as well as from the penetration depth and distribution of the active substance.

From investigations by Gislason [11] it is known that a hydrophobic treatment doesn't resist higher hydrostatic pressures. He showed that once too high hydrostatic pressures had exceeded a threshold value, water could flow through the cracks without further high pressure. The water repellence disappears as long as the crack can't dry at its opening. In case of suction of test solution during thawing the threshold value of hydrostatic pressure was definitely exceeded. Therefore it should be examined, if the water repellence of treated specimens after frost action also disappears as long as water exists on the concrete surface and drying of the impregnated pore openings are prevented.

After drying periods of 4 weeks the water repellence of all investigated series reappeared completely despite partial medium high scaling amounts. Scaling behaviour was not consistent with the obtained overall solution uptake. Despite a clear reduction of the overall solution uptake during the whole freeze thaw test surface scaling could be reduced only up to the 28th freeze thaw cycle independent of type of active substance its distribution and penetration depth. Between 28th and 56th freeze thaw cycle the scaling amount increased progressively versus number of freeze thaw cycles also as before mentioned independent from the type of active substance its distribution and penetration depth. Similar observations had been made by Wittmann [12], who found a start of an increased scaling of normal concrete (w-c ratio 0.5 and 0.58) without artificial air pores under freeze thaw and de-icing agent attack after 60 freeze thaw cycles.

A repeated frost action after a 4 weeks long drying period did not start anymore with a reduction of the scaling amount. With the start of freeze thaw cycles a progressive behaviour of the scaling process was observed. It must be assumed that if once the progressive section of scaling is reached the intense scaling does not stop until the water repellent lined surface zone is completely scaled.

Under common application conditions like impregnation of vertical walls and structural members that allow a fast run off of the de-icing solutions the observed scaling behaviour should not be relevant, because only very small amounts of solution are available on the concrete surface that can be sucked during thawing period through the water repellent surface zone. However, for pavement concretes with a relatively coarse surface that prevents the run down of the de-icing solution and which are situated in regions with a high number of freeze thaw cycles combined with a high attack of de-icing solutions a water repellent impregnation of the surface zone can lead to a suddenly increased scaling which cannot be stopped and may exceed the scaling of an untreated surface remarkably.

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