Freeze-thaw resistance Test Report Summary

This report covers the freeze-thaw-protection performance of a number of products including Clear Penetrating Epoxy Sealer [CPES] and the product now known as Permanent Concrete Sealer [PCS], both manufactured by Smith & Co.

In order to easily see the advantages of the Smith & Co. products, please note the following:

1. Figure 3 on page 6/27 of the report [PDF file page 12] shows that our two tested products have about equal water vapor permeability as the reference (untreated concrete specimen). This means the inevitable water that gets into the concrete can most easily escape by evaporation. All of the other tested products showed much worse performance.

2. Figure 6 on page 11/27 [PDF file page 17] and Table 6 on the following page show that after immersion in salt solution and evaporation, the performance is most comparable to the reference, with PCS actually doing better. This is a good thing. Salt in concrete tends to hold moisture in concrete, contributing to both corrosion of the reinforcing steel and to freeze-thaw damage.

This test shows indirectly that PCS resists salt penetration better than any other product tested. In fact, later tests [Figure 21, on the last page] show that ***the salt penetration was actually so low as to not be measurable***.

3. Figure 6 and table 7 again show that the PCS and CPES products were closest in water-evaporation performance to the reference. This is desirable, as it allows water taken up by the concrete to most easily leave by evaporation. Water retained in the concrete causes freeze-thaw damage, which the other specimens exhibited to a much greater degree as will be seen later in the report.

4. Figure 11 [PDF file page 24] shows that the penetration of salt into concrete while it is curing [in the first few months] is almost the same for the reference as for the Smith & Co. products. This shows that porosity continues to develop in the months after casting concrete, and tests done in that time frame are not meaningful.

Interestingly enough, those specimens that scored the lowest on this test did the worst on freeze-thaw damage protection while the Smith & Co. products CPES and PCS did the best.

5. Fig. 13 [page 20/27 of the VTT report, PDF page 26] is the most dramatic evidence that PCS and CPES help concrete resist freeze-thaw damage. Note that all of the other products showed more volume loss than the untreated reference, which means they did more harm than good!

Similar results are seen in Fig. 17 [Page 23/27, PDF page 29]. The CL-WP product looks better in this figure but was actually much worse in the prior Figure 13.

Epilogue

About ten years after this report was prepared, the underlying mechanism of why some products prevent freeze-thaw damage better than others, while some are worse than nothing at all, was finally discovered. Smith & Co has discovered the basic mechanism of why CPES was so successful, and knows now that a third product, the Paving Brick Sealer, a light-stable exterior polyurethane based on our Five Year Clear technology, will also be successful for the most demanding exterior decorative stonework , fine brick and custom masonry applications. For further information, please see http://www.smithandcompany.org/technical.html.

VTT
Valtion teknillinen tutkimuskeskus
Rakennusmateriaalilaboratorio
Building Materials Laboratory

COMPARATIVE STUDY ON CONCRETE IMPREGNATION AGENTS

RESEARCH PROGRAMME

1. Purpose of the study

The purpose of this study is to investigate the ability of various impregnation agents to protect structures from weathering and corrosion when exposed to deicing salts and freezing and thawing as well as to examine the permability and penetration properties of each impregnant. The study is meant to serve as a basis for directions and recommendations concerning impregnation agents by Road Administration in Finland.

2. Impregnation agents to be studied

Representatives of impregnation agents (manufacturers or importers) are free to enter the products they want into the study programme.

3. Tests

3.1 Test concrete and specimens

The proportioning of test concrete is:

cement content	275 kg/m^3
water/cement ratio	0,75
max.grain size	8 mm
air content	6 %

PL 26 (Kemistintie 3)
02151 Espoo
Puh. vaihde (90) 435 61
Telekopio (90) 4356 7004

Betonimiehenkuja 5
02150 Espoo
Puh. vaihde (90) 4561
Telekopio (90) 455 0170

Betonimiehenkuja 3
02150 Espoo
Puh. vaihde (90) 4561
Telekopio (90) 455 0170

Cubes 100 100 x 100 mm^3 are used in all tests.

After casting, the cubes are stored at RH 100 % for 28 d and further at RH 70 %, till they are impregnated at the age of about 56. The cubes are sand blasted before impregnation.

3.2 Water and water vapour permeability

The permeability test is carried out using three cubes per each impregnant. Prior to impregnation, the hardened test cubes are stored in RH 70 % for 28 days. After impregnation the test specimens are kept in the same conditions for further 14 days.

The test is started by immersing the test specimens in 15 % NaCl- solution. The cubes are weighed at the age of 6 hours, 1 day, 2 days and 3 days, and further at intervals of 3 days up to the age of 21 days. The weight is compared with the weight of the unimpregnated specimens.

After the absorption phase, the test cubes are returned to RH 70 % and the weight loss is measured. The test cubes are weighed daily up to the age of 3 days, and further at intervals of 3 days up to the age of 28 days. The specimens are then stored in RH 40 %, and the weight loss is measured. the test cubes are weighed daily up to the age of 3 days, and further at intervals of 3 days until a constant weight has been reached. Evaporation rates are compared between the impregnated and the unimpregnated test cubes. The permeability of water vapour can also be measured by comparing the weight of each cube after the evaporation test (storage in RH 70 %) with the original weight (before absorption) as well as by comparing the amount of evaporated water within the range of RH 70 % - 40 %.

3.3 Penetration of chlorides

After the above described absorption - evaporation cycle of 42 days, the test is continued with 4 further cycles so that one cycle comprises an absorption phase of 7 days and an evaporation phase of 7 days in RH 70 %.

After the cycles, the penetration of chlorides is determined with the cubes mentioned in 3.2. The profile of chloride content at one side of each cube is determined using powder samples obtained by drilling.

3.4 Effect on the rate of carbonation

The effect of an impregnant on the carbonation rate is determined with four impregnated cubes (100 x 100 x 100 mm^3). Prior to coating, the prisms are dried in vacuum (to avoid carbonation taking place). After impregnation, the test cubes are stored in carbon dioxide chamber (CO_2 content 5 %) for 3 months. the RH of the chamber is adjusted to 75 % (saturated NaCl- solution). The carbonation depth is measured by splitting one cube and by applying phenolpthalein indicator at the ages of 0.5, 1, 2 and 3 months. The obtained carbonation depths are compared with those of unimpregnated cubes.

3.5 Frost-salt test

Two sets of specimens are used in the frost salt test. One set (comprising 3 cubes per each impregnant) is stored after casting at conditions T = 20 °C and RH = 100 % for 28 days. Before impregnation the specimens are kept two days at conditions T = 20 °C, RH = 70 %.

Another set of 3 cubes (per each impregnant) is first stored at T = 20 °C, RH = 100 % for 28 days and then at T = 20 °C, RH = 70 % til the age of 6 days. After that the specimens are stored in a CO_2-chamber (CO_2 content 5 %) until impregnation at the age of 102 days.

The frost-salt test for each impregnated set of cubes is initiated subsequent to storing the cubes at RH 70 % for 14 days. Three unimpregnated reference cubes are also included in the test.

One cycle of the frost-salt test includes an 8-hour period in saturated NaCl- solution with a temperature of +20 °C. The test comparises the total of 100 cycles and in the course of the test disintegration of the cubes is observed by measuring the change in volume. After the test the cubes are also photographed.

3.6 Penetration depth of impregnant

The purpose of the test is to find out the ability of an impregnant to penetrate into capillary cavities of concrete under high humidity circumstances. Two cubes are stored at the conditions of T = 20 °C, RH = 100 % and two cubes at the conditions of T = 20 °C, RH = 70 % before impregnation.

After impregnation the cubes are dried up in a ventilated oven at the temperature of + 105 °C. After constant weight is reached the cubes are split and the penetration depth is measured. The penetration of an impregnant is signed by a white stripe of salt on the broken surface.

4. Schedule

The tests are carried out during the time interval June 1st ... December 31st 1990. The reports will be ready in January-February 1991.

5. Participation fees

The participation fee for the tests is 9000 FIM per agent. The research is partly sponsored by Road Administration of Finland.

26th April 1990

Erkki Vesikari

Erkki Vesikari
Senior Research Scientist

Requested by : Roads Administration Box 33, 00521 HELSINKI

Order : Tt-27/10/331/90

Object : Comparative study of impregnating agents

1. IMPREGNATING AGENTS

Impregnating agents included in this study and their main effective components are listed in Table 1. The impregnants are also numbered from 1 to 15 in table 1. In addition the agents of the impregnants are given in table 1.

Table 1. Impregnants, their main effective components and their agents.

N:o	Impregnant	Effective comp.	Agent
1	Protectosil 340	silane	Oy Algol Ab
2	Protectosil 820	silane	Oy Algol Ab
3	Degalan LP and Protectosil 300	acrylic resin	Oy Algol Ab
4	Thorosilane	silane	Betton Oy
5	Thoroclear 777	siloxane	Betton Oy
6*	Permanent Concrete Sealer	silicate	Smith & Co.
7	Clear Penetrating Epoxy Seal	epoxy	Smith & Co.
8	Aquapel WR and Hardac Acrylic Sealer	silane and acrylic resin	Hartsitekno
9	Clearseal WP20	acrylic polymer	Hartsitekno
10	LPL K-154	polyol/aluminium composition	Hartsitekno
11	Kostex silta (earlier Wacker 290 S) (*	siloxane	Ins. Tsto Sulin
12	Conservado-30	silane	Sika-Betoni Oy
13	Dynasylan BH N	silane	Oy Suomen Hüls
14	Dynasylan BSM 40N	silane	Oy Suomen Hüls
15	Bufasilan	siloxane	Uretek-Elastomer

(* only the penetration depth was measured

* Permanent Concrete Sealer = PCS

2. PREPARATION OF THE TEST SPECIMENS

Concrete cubes, 100x100x100 mm^3, were used as test specimens in all the tests. Ordinary Portland cement content of the concrete was 275 kg/m^3. The water-cement -ratio of the concrete was 0.75 and the aggregate-cement -ratio was 6,46. Unlike given in the Research Report RAM01983/90 (Instructions for testing of impregnating agents) the maximum grain size was 10 mm. Mischoel air entraining agent was added in the mixture 4,5 % by weight of cement. The air content of the mixture was 6,3 %.

The number of the test cubes per each test is given in table 2.

Table 2. The nuber of the test specimens per each test and impregnating agent and the number of the reference specimens.

Test	Number	
	Impr.agent	Reference
Water and water vapour permeability chloride permeability	3	3
Neutralization test	4	4
Frost-salt test	3	3
Frost-salt test using "aged" test specimens	3	3 (*
Measurement of penetration depth:		
- damp specimens	2	
- dry specimens	2	

(* in addition two cubes were prepared for the determination of the neutralization depth

The cubes were cured in their moulds covered with plastic sheets till the age of 1 d at the age of which they were demoulded. The cubes were cured at 95 % relative humidity at 20 °C till the age of 32 d (not 28 d as given in the Research Report RAM01983/90) at the age of which they were grit blasted.

3. TESTS

The tests listed in table 2 were carried out on the test specimens. The test methods are described in the Research Report RAM01983/90. The properties of the impregnating agents were observed during impregnation.

The time table of the test program is shown in figure 1.

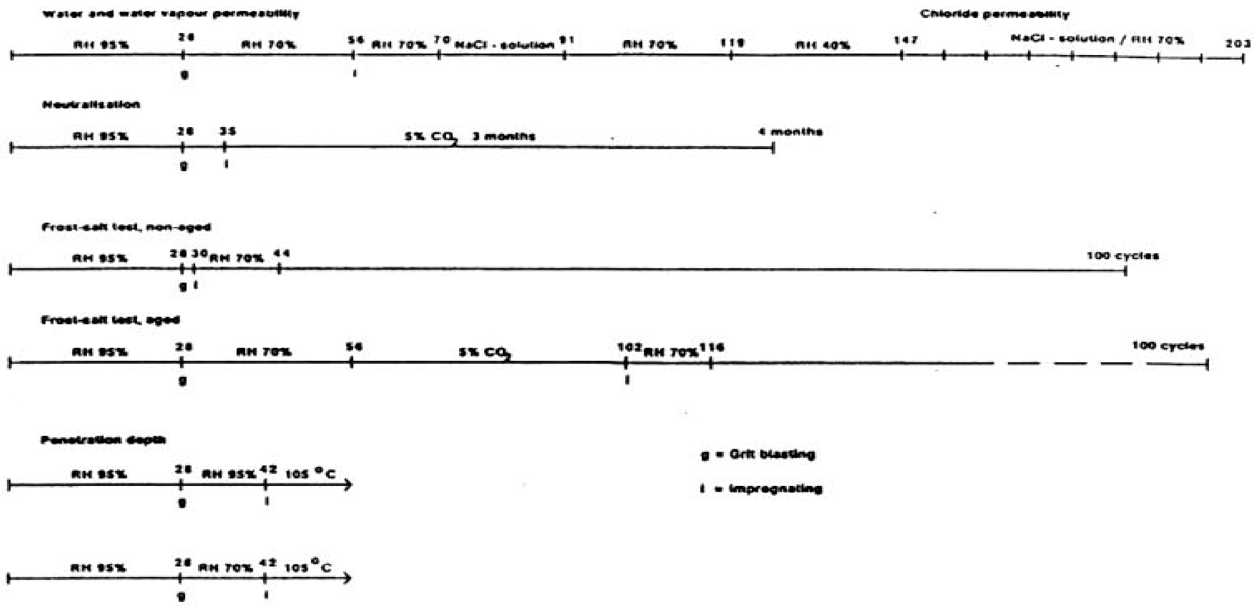

Fig. 1. The time table of the test program.

4. TEST RESULTS

4. Observations of the impregnating agents

All the impregnation agents were easy to apply and they all glided on the vertical surfaces. The impregnants had no colour and they looked like water. The observations made of the impregnation agents are given in table 3.

Table 3. Observations made of the impregnation agents.

Impr ag.	Appl. meth.	Rate g/m^2 (1	Drying time,min(2	Smell	Appearance after drying
1	S	115	30/ 30/ 50	strong	Slightly darkens
2	S	115	30/ 30/ 50	strong	Slightly darkens
3	S	170	90/ 90/150	moderate	Unnoticeable
4	S	185	90/ 90/150	moderate	Slightly darkens
5	S	170	90/ 90/150	moderate	Unnoticeable
6	R (3	195/100	60/ 60/ 90	weak	Unnoticeable
7	R (4	130/60	40/ 40/ 90	moderate	Glazed, greenish
8a	R (5	170	90/ 90/180	moderate	Looks dam
b	R	165	90/120/150	moderate	
9	B	250/215	60/ 90/150 90/ 90/150	moderate	Glazed
10	B	125	60/ 90/180	moderate	Slightly darkens
11	S	160	90/ 90/150	moderate	Unnoticeable
12	R	165/22	120/120/150	moderate	Unnoticeable
13	P	175	90/ 90/150	weak	Unnoticeable
14	P	110	60/ 60/ 90	weak	Unnoticeable
15	S	155	120/120/180	moderate	Unnoticeable

(1 1. layer / 2. layer
(2 sides/upper side/lower side
(3 - application with a roller as much as absorbed by the surface,
- the same amount of water after 3 h,
- rinsing with water after 24 h,
- retreatment after the drying of the surface,

(4 retreatment after 24 h
(5 8a: Aquapel WR
8b: Hardac Acrylic Sealer

In table 3:
- S stands for submerging of the specimens into the impregnation agent as follows:
  - submerging 50 s,
  - drying at room climate on a stand 50 s,
  - submerging 50 s,
  - drying at room climate on a stand 50 s,
  - submerging 50 s,
  - drying at room climate on a stand 50 s,
  - submerging 50 s,
  - drying at room climate on a stand for 12 h before removing into curing conditions or for 24 h before starting the tests.
- R stands for application with a roller.
- B stands for application with a brush.
- P stands for the pouring of the impregnant on the specimens.

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4.2 Water, water vapour and chloride permeability

The results of the water and water vapour permeability measurements are shown graphically as percentual weight change in figures 2...4.

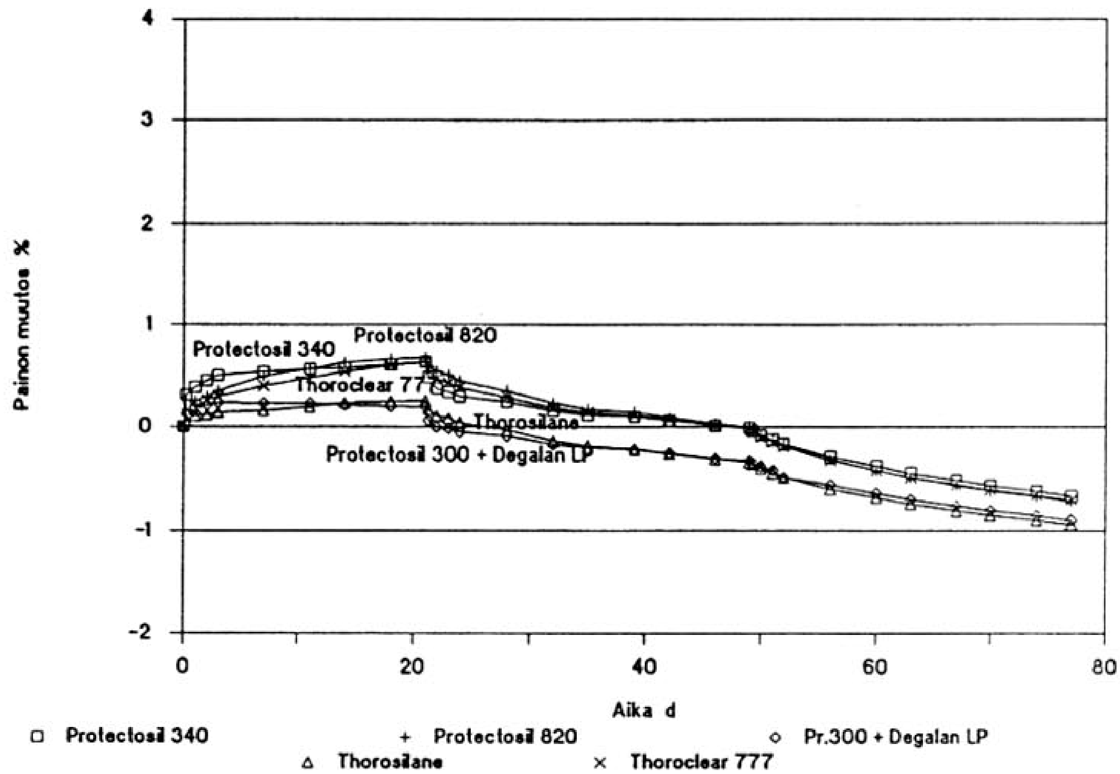


Figure 2. Water and water vapour permeability. Percentual weight change. Impregnating agents 1...5.

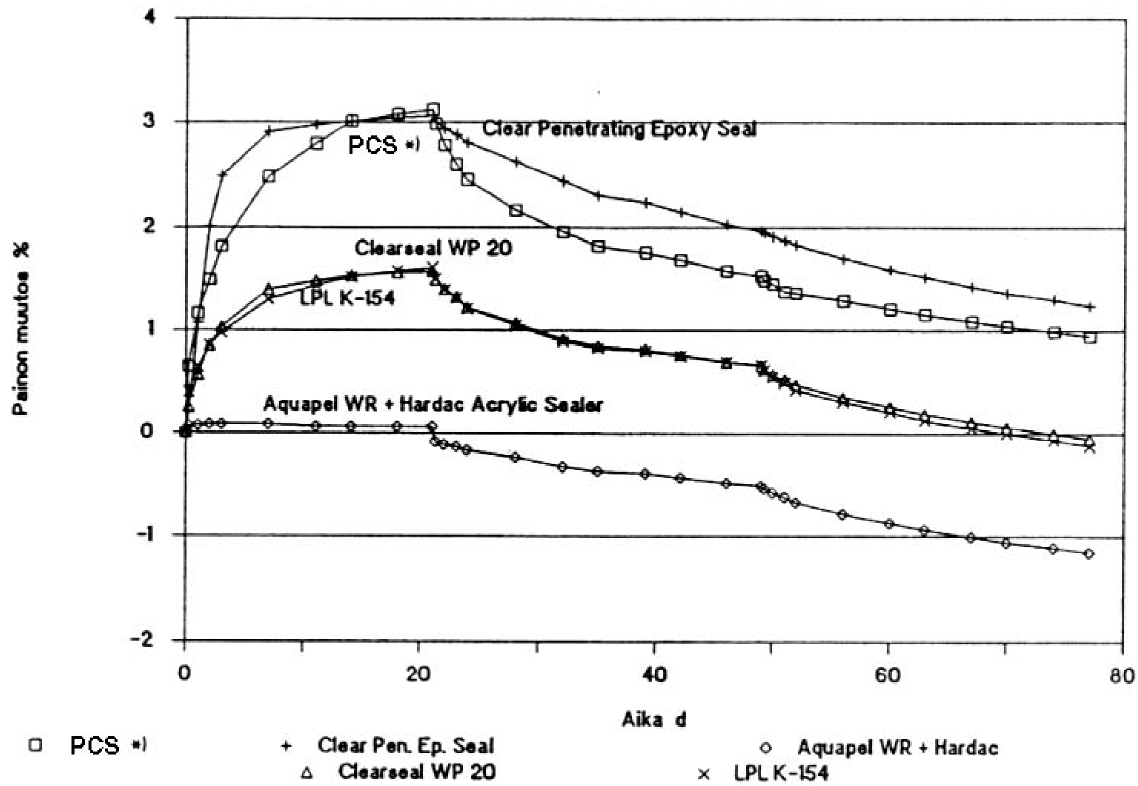


Figure 3. Water and water vapour permeability. Percentual weight change. Impregnating agents 6...10.
*) curing time is half of that recommended

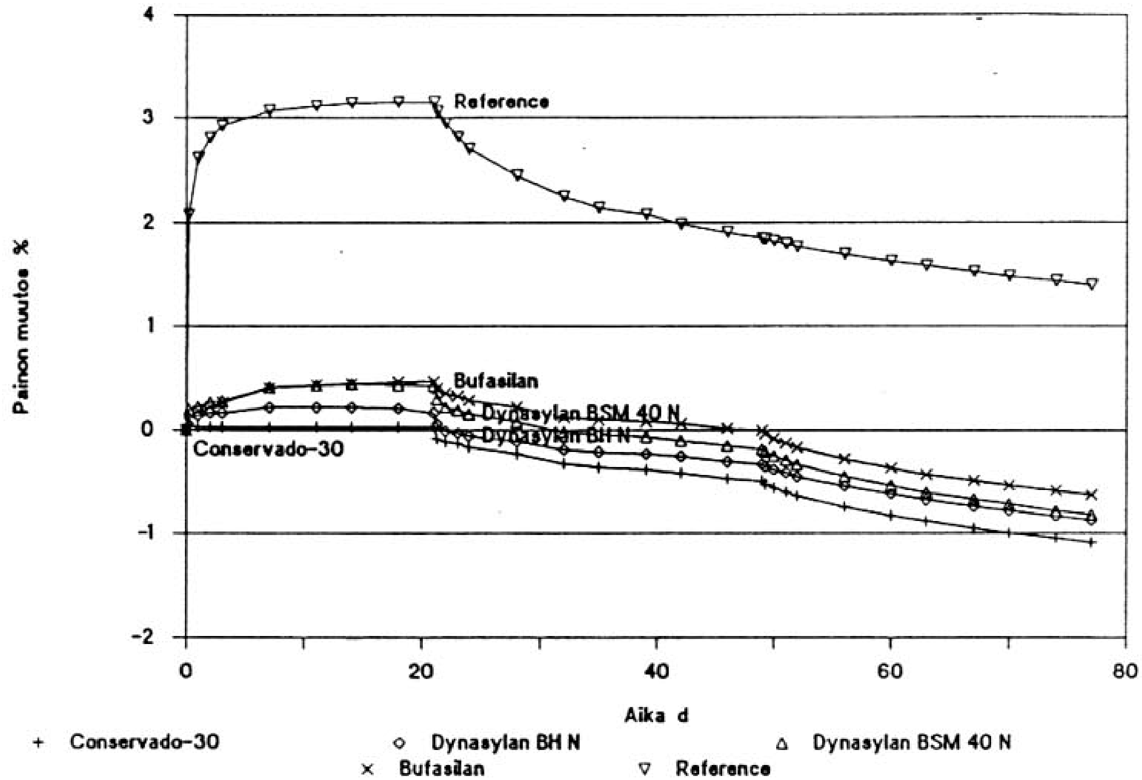


Figure 4. Water and water vapour permeability. Percentual weight change. Impregnating agents 11...14 and reference.

The percentual weight changes (% of the initial weight) of the test specimens at the end of each period are shown in table 4 and figure 5.

Table 4. Percentual weight changes of the test specimens at the end of each period.

Impr.ag.	NaCl-solut.	RH 70 %	RH 40 %	NaCl-immersions
1	0.64	-0.01	-0.65	-0.29
2	0.68	-0.002	-0.71	0.52
3	0.19	-0.31	-0.89	-0.81
4	0.25	-0.33	-0.94	-0.63
5	0.63	-0.01	-0.70	0.52
6 (*	3.12	1.53	0.94	2.25
7	3.06	1.96	1.24	2.89
8	0.06	-0.51	-1.16	-1.04
9	1.57	0.66	-0.05	2.03
10	1.61	0.66	-0.12	1.59
12	0.03	-0.49	-1.09	-0.95
13	0.17	-0.32	-0.88	-0.81
14	0.43	-0.18	-0.82	-0.44
15	0.47	-0.002	-0.62	-0.57
Ref.	3.16	1.85	1.40	2.67

(* curing time was half of that recommended

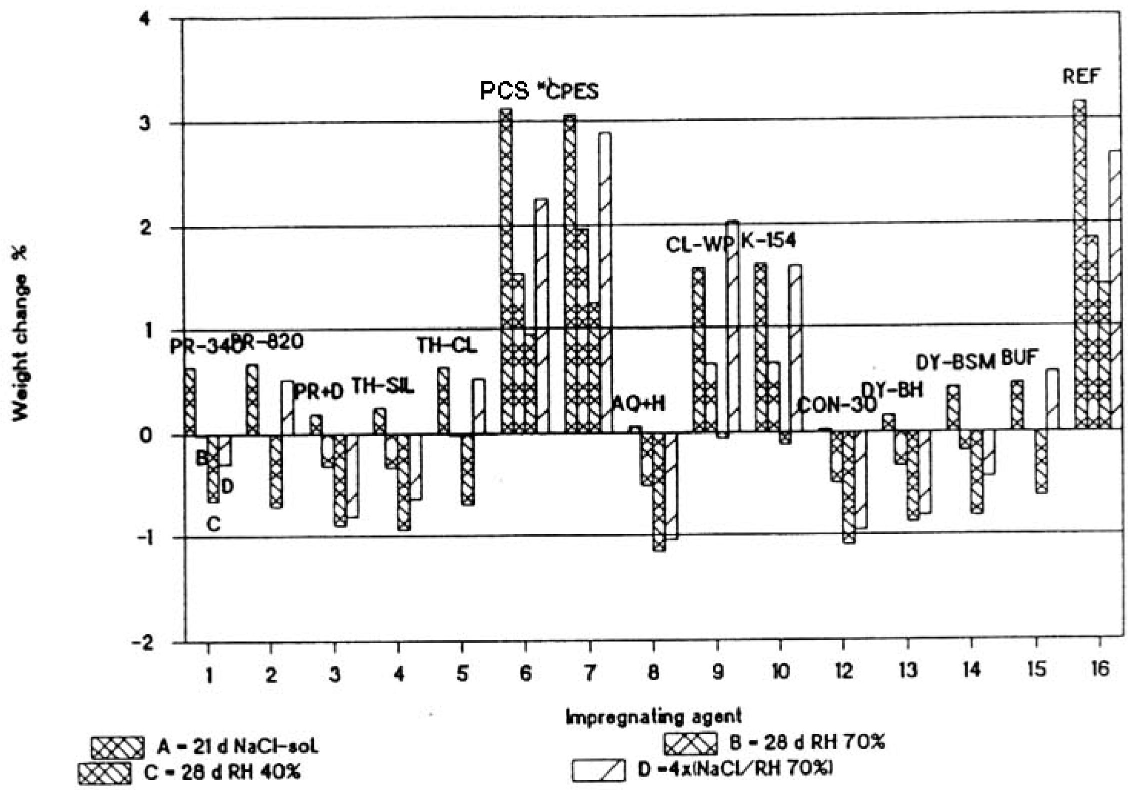


Fig. 5. Percentual weight changes of the test specimens at the end of each period.
*) curing time was half of that recommended

The relation of the percentual weight changes of the test specimens at the end of each period to those of the reference specimens are shown in table 5 and figure 6.

Table 5. The relation of the percentual weight changes of the test specimens at the end of each period to those of the reference specimens.

Impr.ag.	NaCl-solut.	RH 70 %	RH 40 %	NaCl-immersions
1	20.13	-0.67	-46.73	-10.81
2	21.43	-0.13	-50.75	19.47
3	6.12	-16.94	-63.57	-30.46
4	7.92	-17.88	-67.27	-23.72
5	19.83	-0.75	-49.75	19.60
6 (*	98.82	82.57	67.61	84.42
7	96.91	106.21	88.78	108.17
8	1.99	-27.69	-82.65	-38.99
9	49.87	35.63	-3.57	75.97
10	51.07	35.92	-8.35	59.53
12	0.97	-26.61	-77.94	-35.60
13	5.25	-17.44	-62.59	-30.26
14	13.50	-9.65	-58.62	-16.43
15	14.77	-0.12	-44.42	21.37

(* curing time was half of that recommended

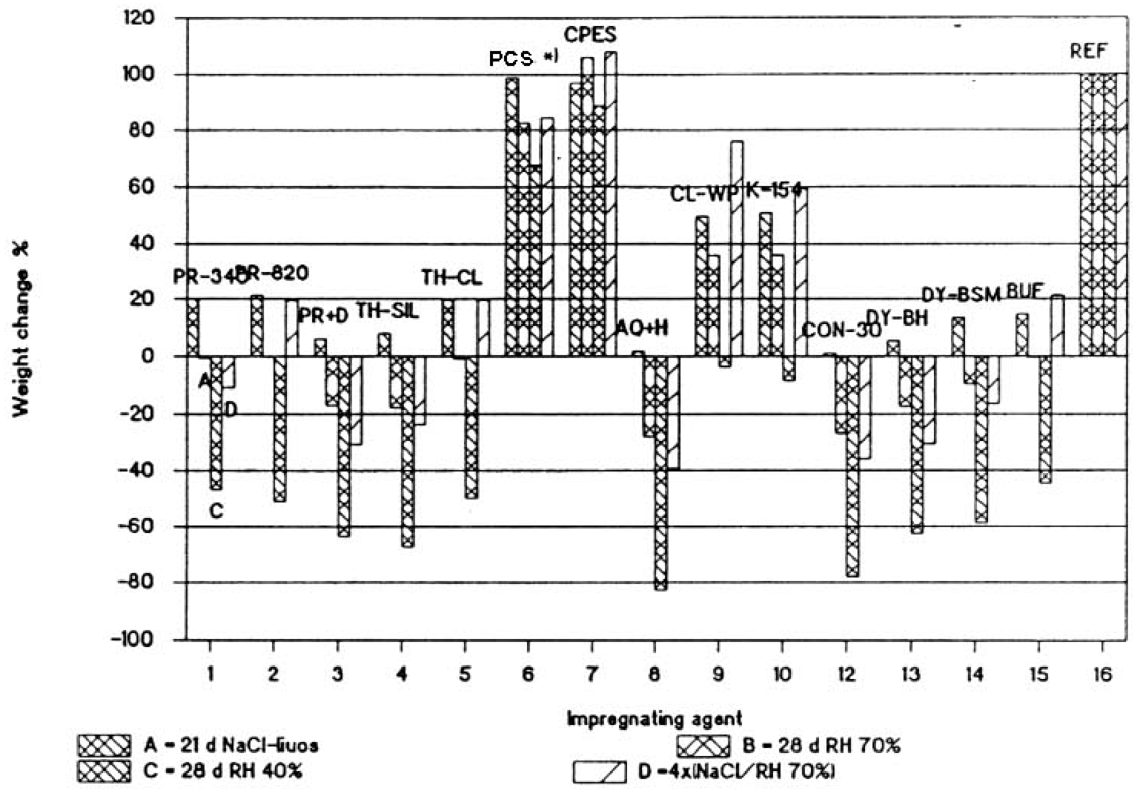


Fig. 6. The relation of the percentual weight changes of the test specimens at the end of each period to those of the reference specimens. *) curing time was half of that recommended

The amounts of water evaporated from the specimens till the end of the curings at 70 % and 40 % relative humidities as the percentage of the amount of solution penetrated into the specimens during the immersion in NaCl- solution in the beginning of the test are shown in table 6 and figure 7. The relation of the amount of water evaporated from the specimens during the curing at 40 % relative humidity to that of the reference specimens is shown in table 6 and figure 8.

Table 6. The amounts of water evaporated from the specimens as the percentage of the amount of solution penetrated into the specimens in the in NaCl- solution immersion and the relation of the amount of water evaporated from the specimens during the curing at 40 % relative humidity to that of the reference specimens.

Impr. agent	Water evaporated as percentage of the solution penetrated in NaCl-immersion, %		Relation of the amount of water evaporated at RH 40 % to that evaporated from the reference specimens
	RH 70 %	RH 40 %	
1	101.95	202.85	73.28
2	100.36	204.91	78.81
3	262.19	559.95	61.53
4	232.30	476.11	67.71
5	102.22	211.13	75.14
6 *	51.02	69.70	123.63
7	35.75	59.42	103.38
8	916.52	1941.67	69.29
9	58.11	103.17	92.35
10	58.78	107.24	98.32
12	1712.25	3667.57	63.71
13	294.74	627.93	59.19
14	141.88	292.31	70.85
15	100.48	233.24	61.83
Ref.	41.38	55.71	100.00

* curing time was half of that recommended

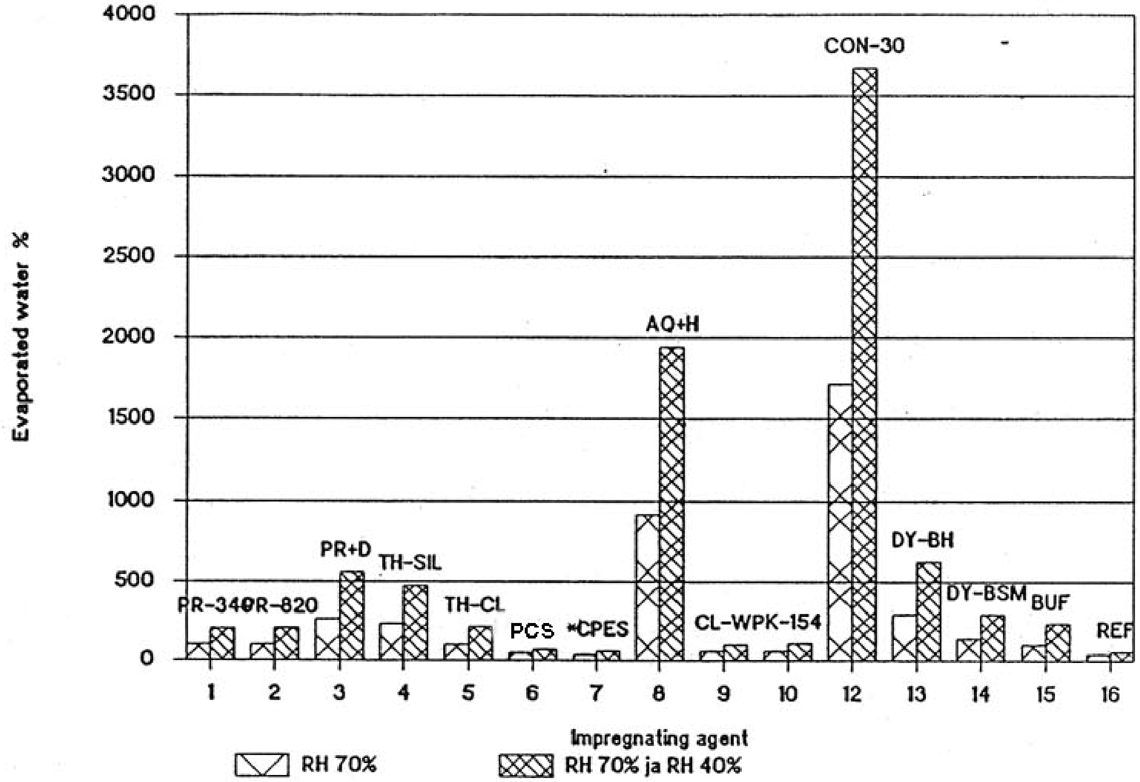


Fig. 7. The amounts of water evaporated from the specimens till the ends of the curings at 70 % and 40 % RH as the percentage of the amount of solution penetrated into the specimens during the immersion in NaCl-solution in the beginning of the test. (* curing time was half of that recommended

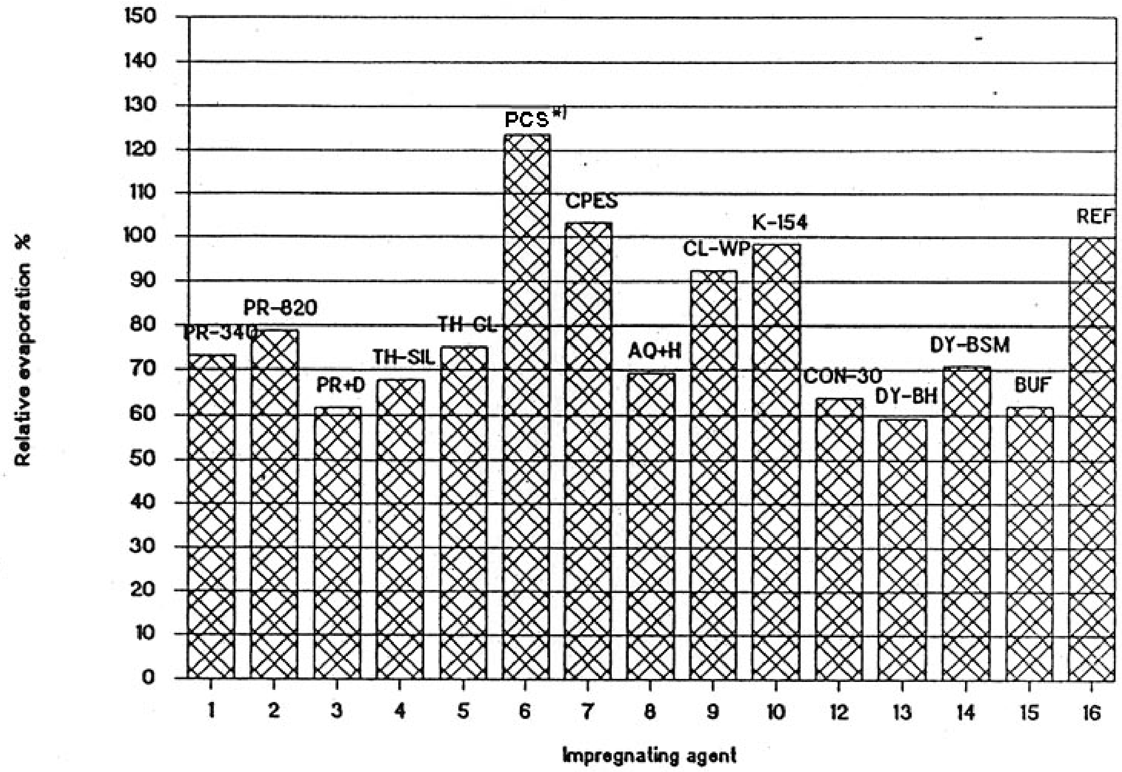


Fig. 8. The relation of the amount of water evaporated from the specimens during the curing at 40 % RH to that of the reference specimens

According to the agent of PCS the 14 days curing time used in the testsis half of that recommended. This may have influenced the test results.

4.3 Chloride permeability

The chloride contents of the specimens at the depths of 0...20 mm and 20...50 mm and the percentual change of the weight of the specimens at the end of the test (compared to the initial weight) and the relation of this weight change to that of the reference specimens are shown in table 7. The percentual weight change of the specimens at the end of the test is shown in figure 9 and the relation of their weight change to that of the reference specimens is shown in figure 10.

Table 7. The chloride contents at the depths of 0...20 mm and 20...50 mm and the percentual change of the weight of the specimens at the end of the test and the relation of this weight change to that of the reference specimens.

Impr. agent	Chloride content % 0-20 mm	Chloride content % 20-50 mm	Weight change in the end %	Relation of the weight change to that of the reference specimens, %
1	0.26	0.13	-0.29	-10.81
2	0.27	0.19	0.52	19.47
3	0.15	0.1	-0.81	-30.46
4	0.09	0.08	-0.63	-23.72
5	0.63	0.19	0.52	19.60
6 *	1.25	0.69	2.25	84.42
7	1.08	0.77	2.89	108.17
8	0.17	0.08	-1.04	-38.99
9	1.27	0.55	2.03	75.97
10	1.49	0.7	1.59	59.53
12	0.44	0.15	-0.95	-35.60
13	0.25	0.15	-0.81	-30.26
14	0.34	0.16	-0.44	-16.43
15	0.43	0.14	0.57	21.37
Ref.	1.3	0.89	2.67	100.00

* curing time was half of that recommended

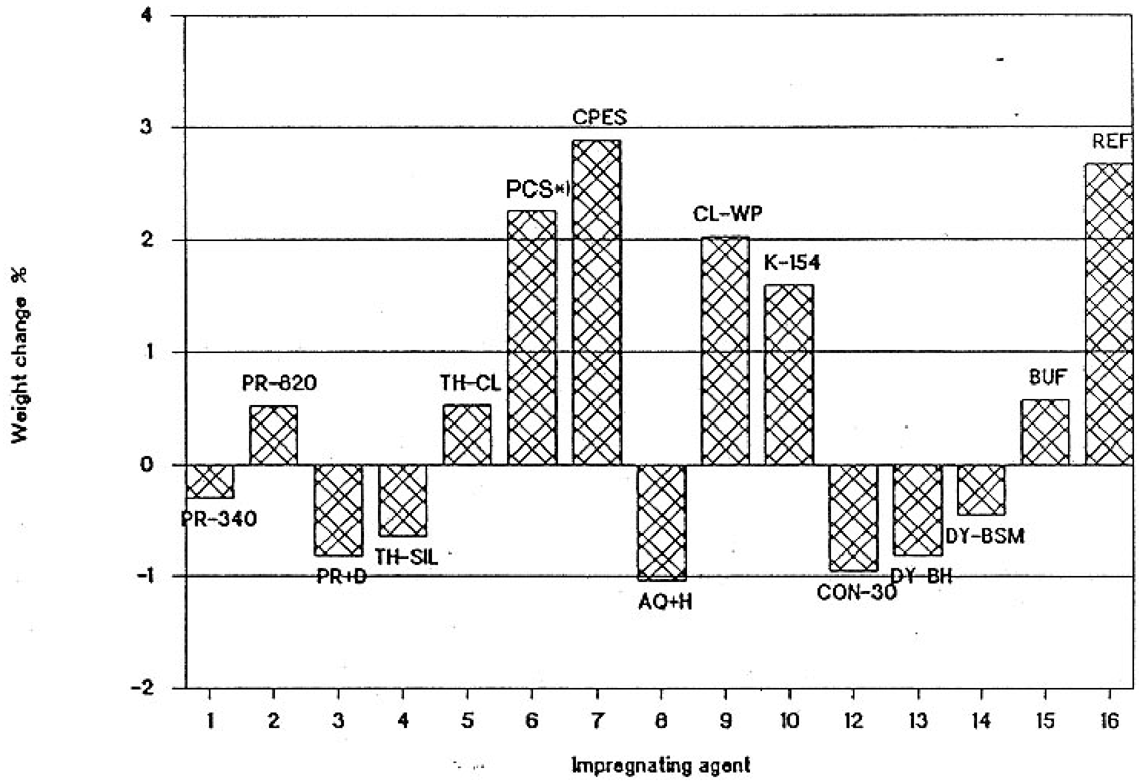


Fig. 9. The percentual weight change of the specimens at the end of the test.
*) curing time was half of that recommended

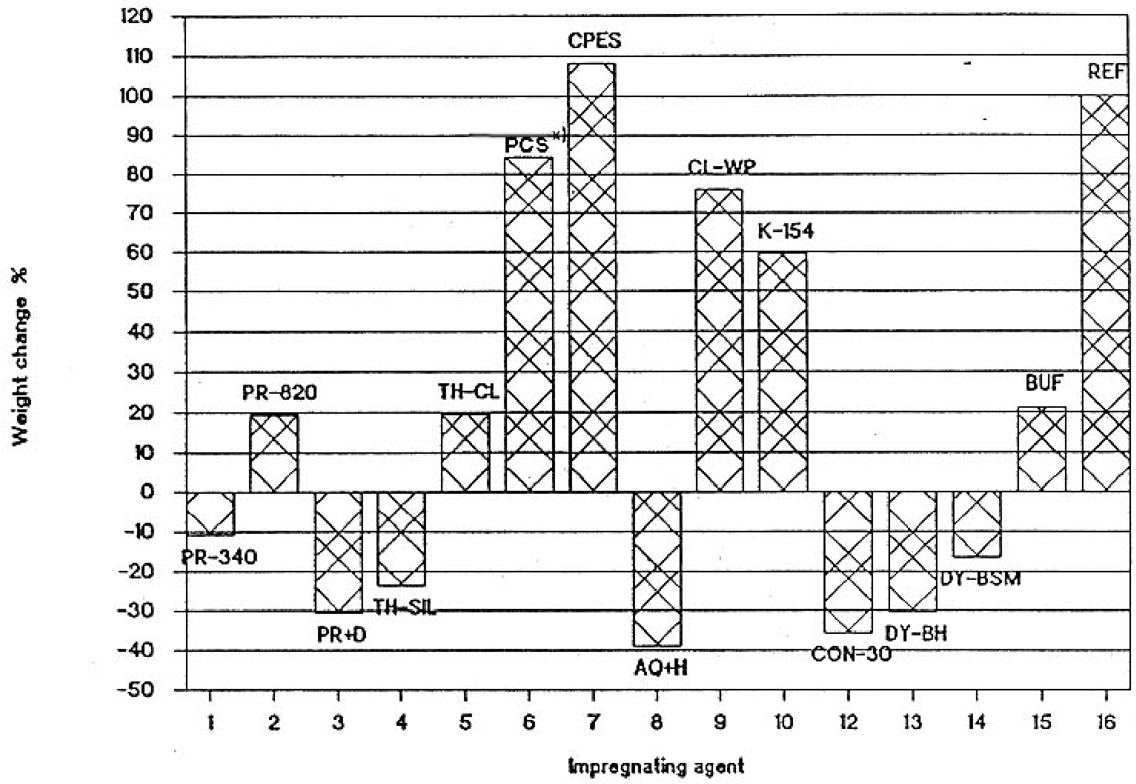


Fig. 10. The relation of the percentual weight change to that of the reference specimens.
*) curing time was half of that recommended

The relation of the chloride contents and the amount of solution penetrated into the specimens during the immersion in the NaCl-solution is shown graphically in figure 11.

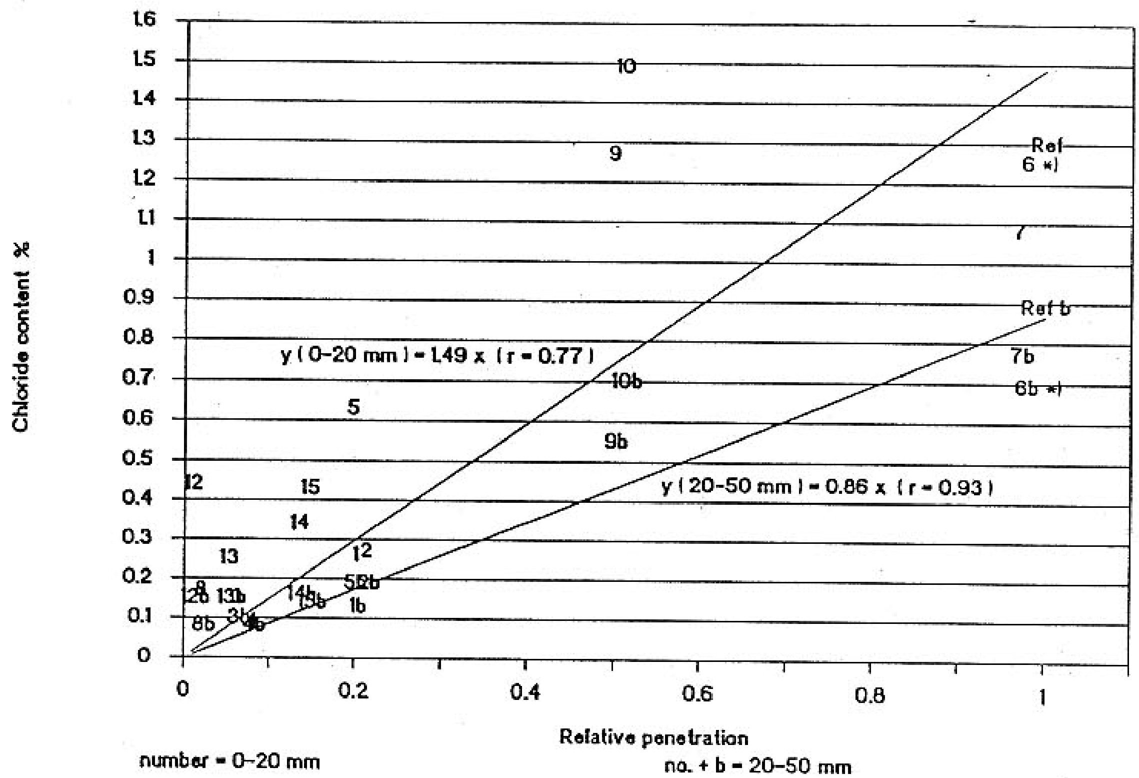


Fig. 11. The relation of the chloride contents and the amount of solution penetrated into the specimens during the curing in the NaCl-solution.
*) curing time was half of that recommended

According to the agent of PCS the 14 days curing time used in the test is half of that recommended. This may have resulted the test results.

4.4 Effect of impregnating agents on the neutralization rate

Figure 13 shows graphically the mean neutralisation depths at different points of time.

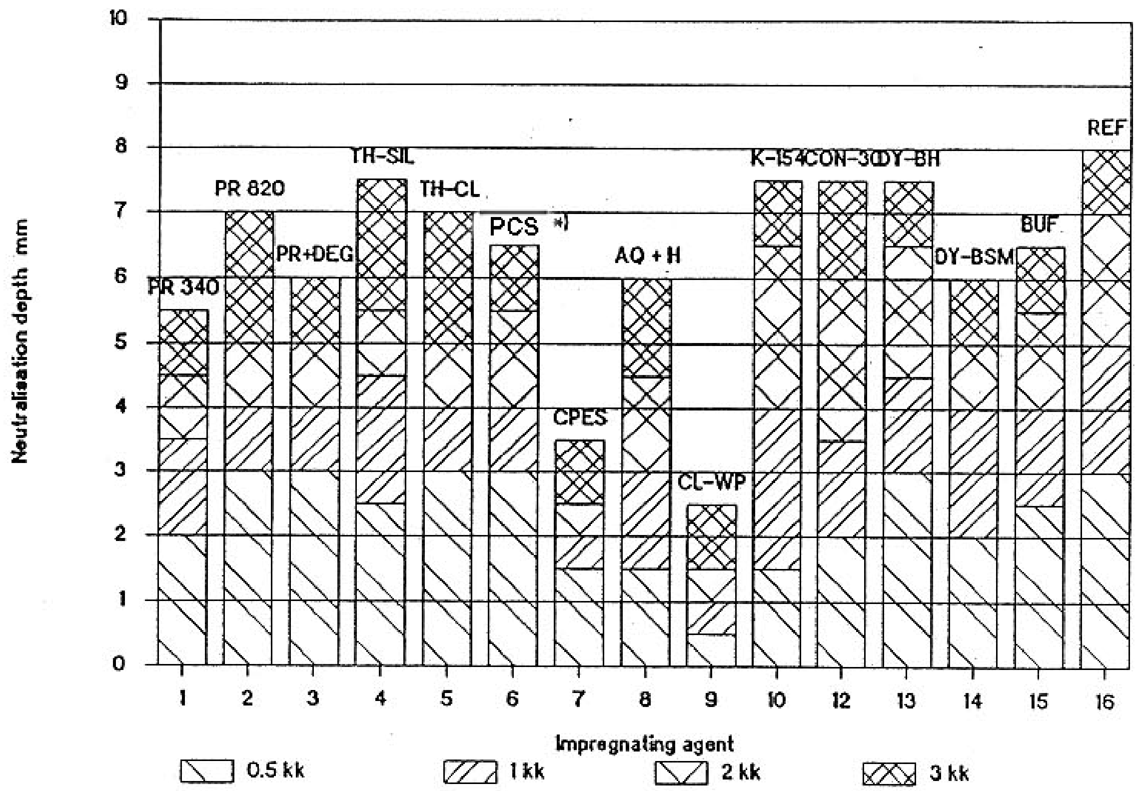


Fig. 12. The mean neutralisation depth of the test specimens at different points of time.
*) no curing

According to the agent of PCS the few hours curing used in the test has not made it possible for the impregnant to penetrate into concrete. Penetrating takes some 4 weeks. It is possible that longer curing time would have resulted in better results in neutralisation test.

4.5 Frost-salt test

Figure 13 illustrates the mean volume change of the test specimens after 10, 25, 50 and 100 freeze-thaw cycles in the frost-salt test.

Photographs of the test specimens after 100 freeze-thaw cycles are shown in figures 14...16.

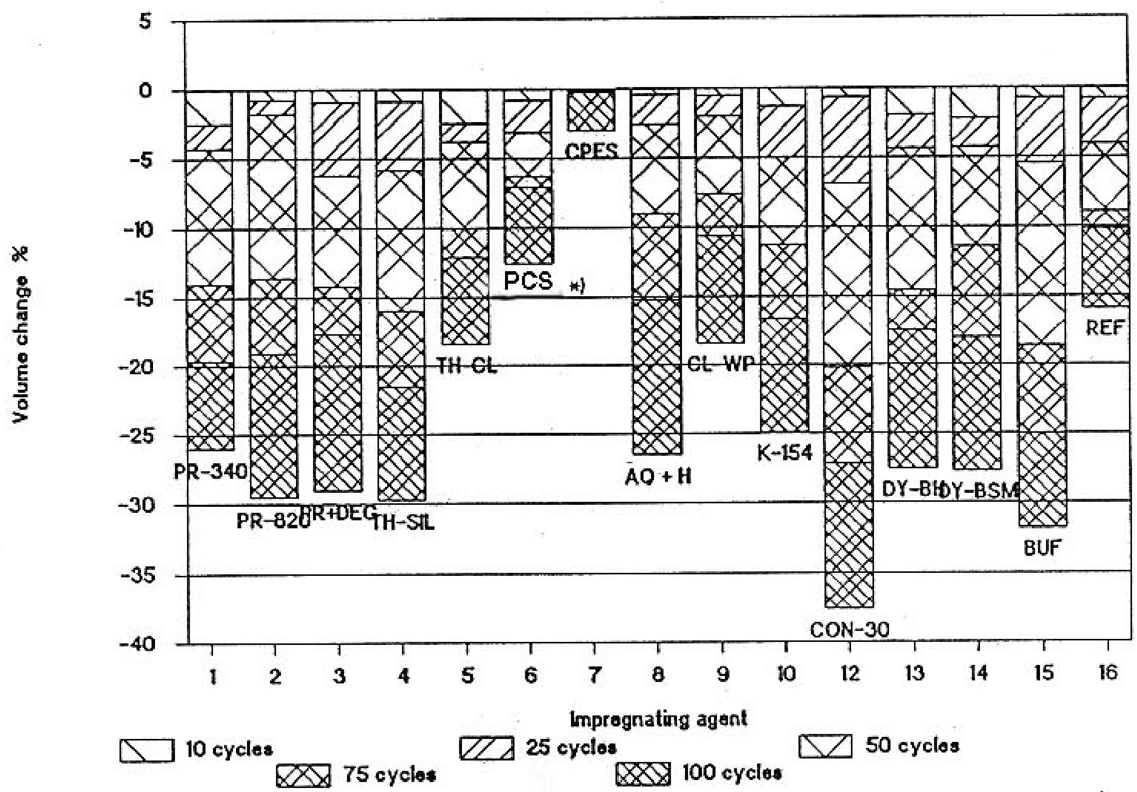
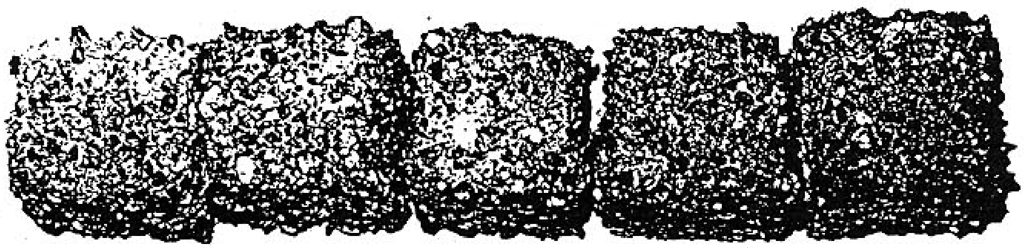


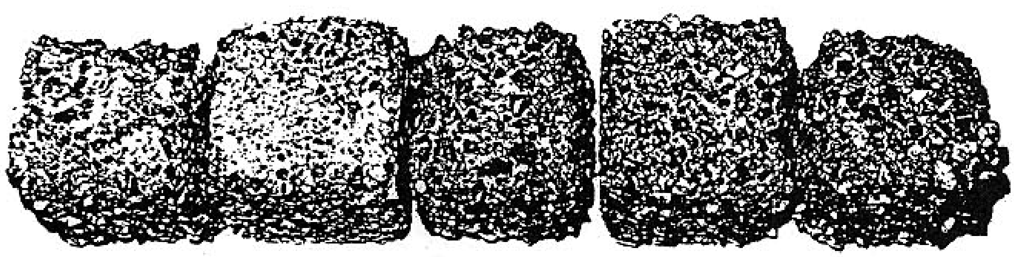
Fig. 13. The mean volume change of the test specimens after 10, 25, 50 and 100 freeze-thaw cycles. (* curing time was half of that recommended.



Protectosil 340 | Protectosil 820 | Protectosil 300 + Degalan LP | Thorosilane | Thoroclear 777

Non-aged | 100 cycles

Fig. 14. Test specimens after 100 freeze-thaw cycles. Impregnating agents 1...5.



PCS *) | Clear Penetrating Epoxy Seal | Aquapel WR + Hardac Acrylic Sealer | Clearseal WP 20 | LPL K-154

Non-aged | 100 cycles

Fig. 15. Test specimens after 100 freeze-thaw cycles. Impregnating agents 6...10.
(* curing time was half of that recommended.

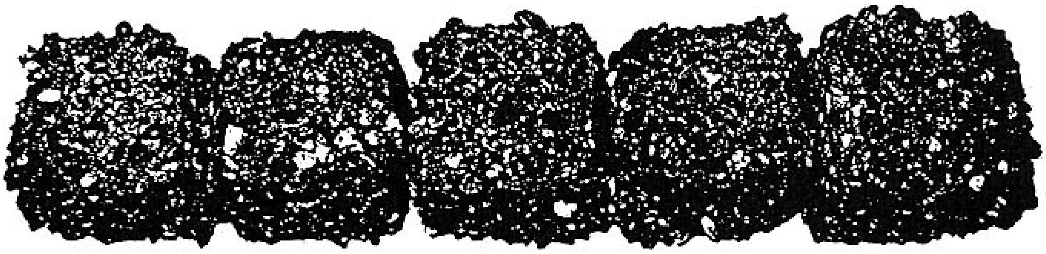


Fig. 16. Test specimens after 100 freeze-thaw cycles. Impregnating agents 12...15 and reference specimens

According to the agent of PCS the 14 days curing time used in the test is half of that recommended. This may have affected the best result.

4.6 Fros-salt test with aged test specimens

The average carbonation depth of the two specimens after 102 days carbon dioxide treatment was 4...5 mm.

Figure 17 illustrates the average volume change of the test specimens after 10, 25, 50 and 100 freeze-thaw cycles.

Photograph of the test specimens after 100 freeze-thaw cycles are shown in figures 18...20.

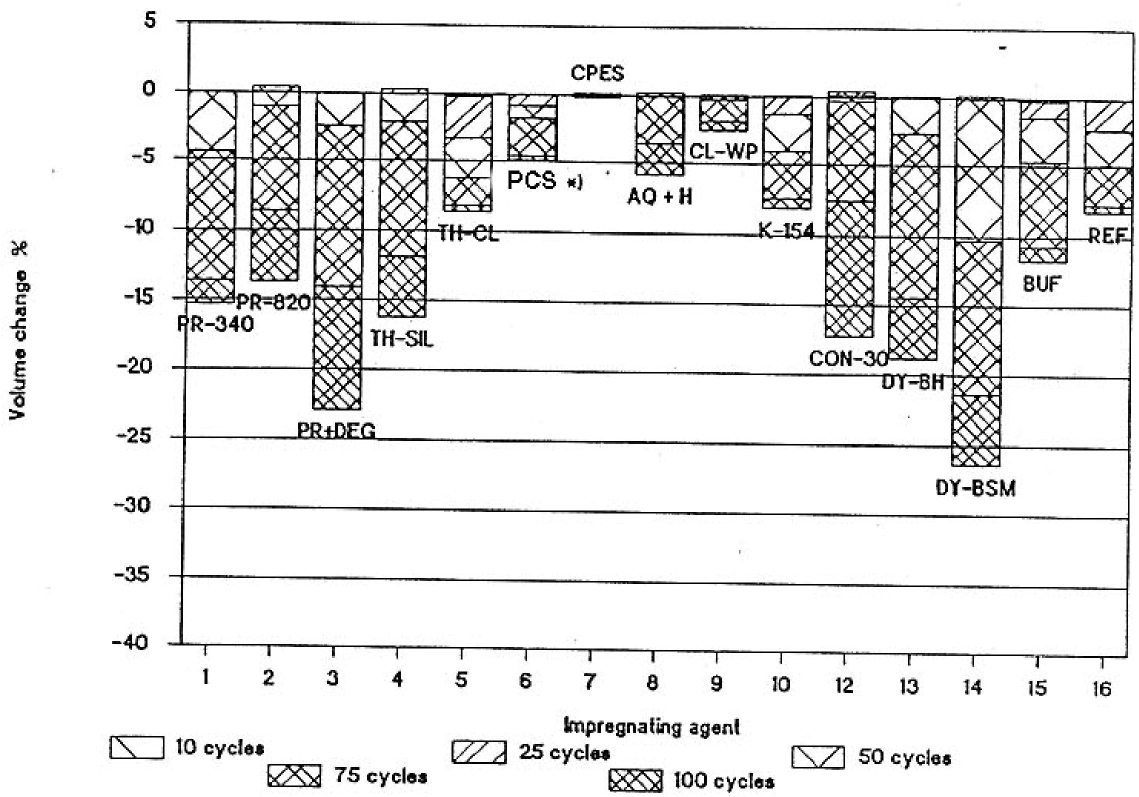


Fig. 17. The average volume change of the test specimens after 10, 25, 50 and 100 freeze-thaw cycles.
(* curing time was half of that recommended.

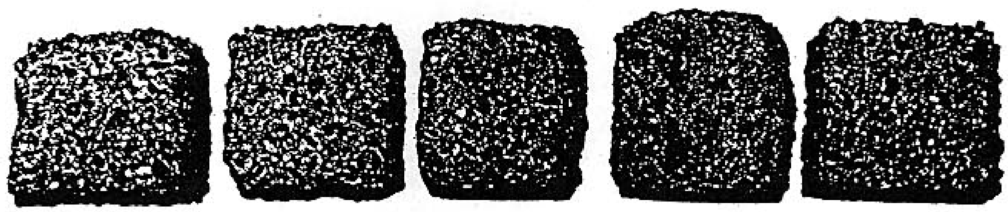


Fig. 18. Test specimens after 100 freeze-thaw cycles. Impregnating agents 1...5.

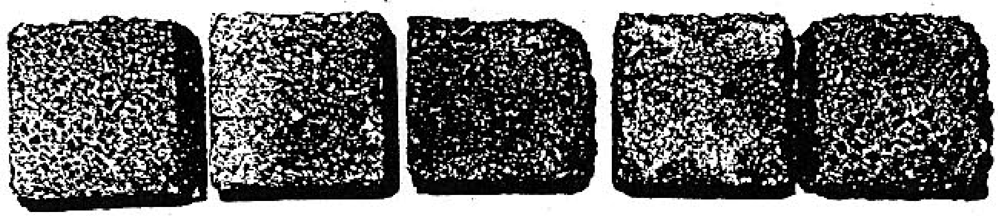
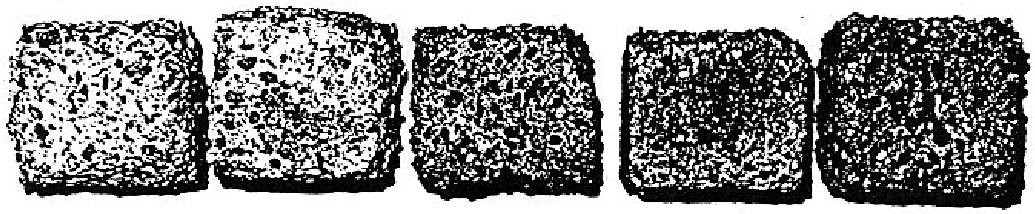


Fig. 19. Test specimens after 100 freeze-thaw cycles.
Impregnating agents 6...10.
(* curing time was half of that recommended.



Conservado-30 | Dynasylan BH N | Dynasylan BSM 40 N | Büfasilan | Ref. ~~Vertailu~~

Aged 100 cycles

Fig. 20. Test specimens after 100 freeze-thaw cycles. Impregnating agents 12...15 and reference specimens.

According to the agent of PCS the 14 days curing time used in the test is half of that recommended. This may have affected the test result.

4.7 Penetration depth of the impregnating agents

The mean penetration depths of the impregnating agents are shown in figure 21.

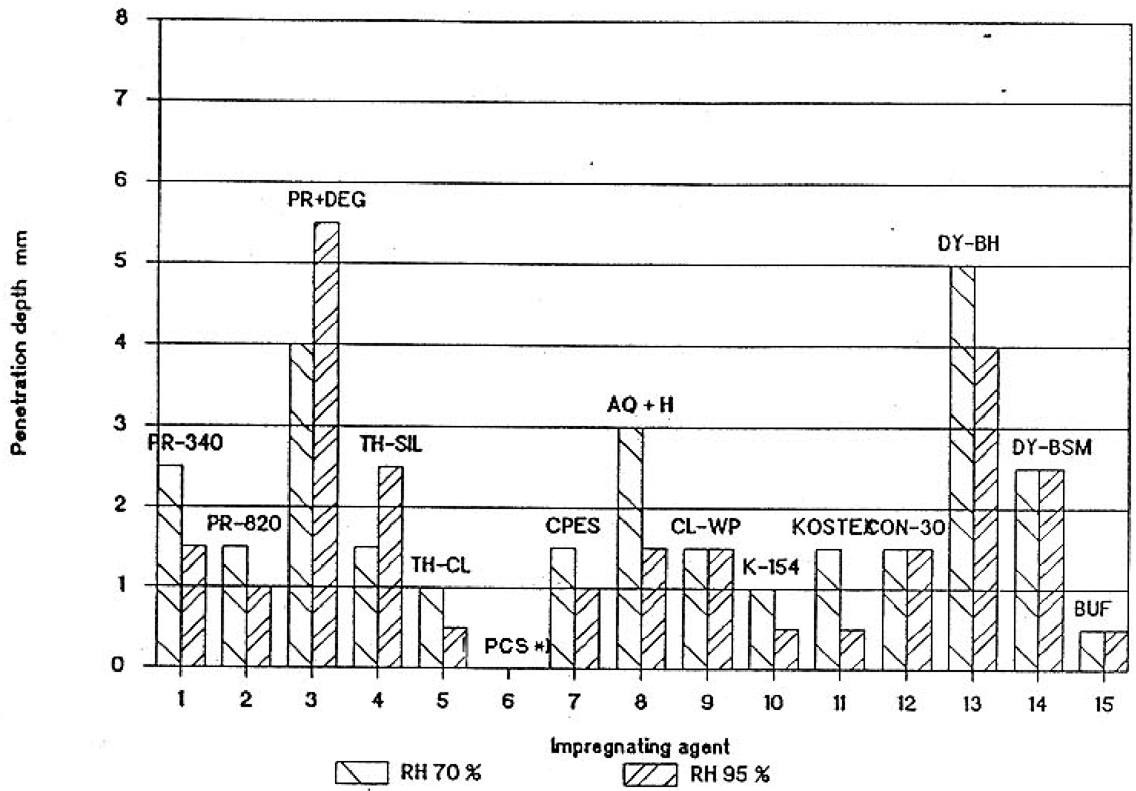


Fig. 21. The penetration depths.
(* can not be determined with the method used.

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