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Yang Quanbing

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Preface

The author, Yang Quanbing, is researcher at the Materials Engineering Research Laboratory at Tongji University in Shanghai. Mr. Yang spent three months as a guest researcher at our division during the period June-August 1997. This report is the result of his work with us. During his time at our department he also made work on the self-desiccation of high performance concrete[1)].

The problem of salt-frost scaling treated here is of big practical importance. In countries like Sweden, and many other countries where frost occurs and where deicing salts are used, or where the concrete is exposed to sea water, scaling of the concrete surface is a severe problem that is difficult to cope with. The only practical solution is to use air-entraining admixtures added to the fresh concrete mix which must be of high quality. By such admixtures a protective air-pore system is created. It is difficult, however, to produce air-pore systems that have a quality sufficiently high, and that are stable during transport and placing of the concrete.

There is a great need of admixtures that are able to fulfil these requirements. For many decades, an admixture sold under the tradename Vinsol Resin has been used with good results in many countries. Professor Huang Shiyuan who led the research indstitute where Mr. Yang is employed has developed a new admixture, SJ-2, based on the surface active substance saponin. In the work presented here, this new admixture has been compared with the traditionally used admixture Vinsol Resin. The results show that both admixtures are able to produce concrete with a high degree of salt-frost resistance.

I sincerely thank Mr. Yang Quanbing for this excellent work which was performed during his short stay at our department.

Lund, 4 September 1998

Göran Fagerlund
Head of Department

1) Yang Quanbing: Inner RH and degree of saturation in high performance concrete cured in water or salt solution for 2 years. Division of Building Materials, Lund Institute of Technology, Report TVBM-7115, 1997.

FREEZE-THAW RESISTANCE OF CONCRETE WITH A NEW TYPE OF AIR-ENTRAINING AGENT (SJ-2)

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Abstract

In this report a comparison is made between a new type of air-entraining agent (SJ-2) based on saponin and the traditional air-entraining admixture Vinsol Resin. It is shown that both admixtures significantly improve the salt-frost resistance of concrete made with the water-cement ratio 0.50. Good salt-frost resistance, as judged by Swedish rules, is obtained when the air content of the fresh concrete exceeds about 6%. There is no significant difference between the two admixtures.

Both admixtures create air-pore systems that are very stable. The loss in air content between the fresh and hardened concrete is on an average only 0.3%.

The air-pore structure was analysed by automatic image analysis. The specific area of the pore system is very high with both admixtures, showing that the air-pores formed are small in both cases. There is no important difference between the two admixtures.

The strength loss for one percent increase in the air content is about 4-5%. This is a normal value found for almost all air-entraining admixtures. There is a slight indication of a smaller strength loss when the admixture SJ-2 is used. This difference, however, can not be regarded significant due to the limited amount of data.

All results indicate that the new admixture SJ-2 is well suited for the production of concrete with high salt-frost resistance.

1 Introduction

A large number of surface active chemical substances can be used as air-entraining agents in concrete. Many of these are refined by-products from various industrial processes, e.g. pulp and paper production, or petroleum production. The most commonly used chemicals are, [1].

- sodium salts of wood resin (e.g. sodium-abietate which is similar to Neutralized Vinsol Resin, or just Vinsol Resin, which has been widely used for a very long time)
- salts of fatty acids
- salts of sulphonated hydrocarbon
- alkyl-benzyl sulphonates

A new type of air-entraining agent (trademark SJ-2) has been invented by professor Huang Shiyuan at the Materials Engineering Research Laboratory at Tongji University, Shanghai. The admixture is refined from the fruits of some natural plants. Its main component is the surface actice substance saponin.

Research on SJ-2 was initiated by prof. Huang in 1984. This research was finished and evaluated by China Administration of Building Materials in May 1988, [2]. Production of SJ-2 started in 1990. Until now it has been used in many infrastructure projects. In total about 1 million cubic meter of air-entrained concrete made with SJ-2 has been produced.

One of the most commonly used air-entraining agent in the world is the so-called Vinsol Resin, see above. In order to evaluate the new admixture SJ-2, comparisons were made with Vinsol Resin marketed by the Swedish Cement company Cementa AB and sold under the trademark Cementa 88L. The comparisons were made at the Division of Building Materials, Lund Institute of Technology, Sweden. The two types of air-entraining admixture were investigated in a number of concretes that were freeze-thaw tested. The test used was a so-called salt-frost scaling test. The test procedure is similar to the internationally standardized so-called cube-test, CEN/TC51, [3]. This type of test method is also recommended by RILEM Technical Committee TC-117, [4].

2 Physical properties and and chemical composition of SJ-2

SJ-2 can be obtained in two different forms, as dry powder, or as liquid. In the present tests the powder was used. The properties of this are as follows:

- color.... brown
- density... $1.3 g/cm^3$
- water content... <5%
- water insoluble content... negligible
- volatile components and sugar content... negligible

SJ-2 is a non-ion type of surface active agent. The admixture is easily soluble in water and it is stable in acid solutions or alkali solutions. It is also stable in hard water.

The main component in SJ-2 is saponin. According to an IR-analysis the saponin is made up of numerous monosaccharide molecules of type fructose ($C_6H_{12}O_6$).

The generated air-bubble volume, the foam capacity, and the foam (bubble) stability of a water solution of SJ-2 were measured by the standardized method JGJ 56-84. The test results are shown in Table 1. The stability is defined by the foam volume after 5 minutes divided by the initial volume. No comparison of the foam capacity and stability with Vinsol Resin was made.

Table 1: The generated air-bubble volume (the foam capacity) and the foam (bubble) stability of a water solution of SJ-2. Test method JGJ 56-84.

Concentration of SJ-2 (%)	Air-bubble volume initially (ml)	Air-bubble volume after 5 min. (ml)	Stability (%)	pH
0.40	52	47	90	6.89
0.65	61	55	90	6.37
0.80	67	61	91	6.02

The admixture SJ-2 can conveniently be used in combination with superplasiticizers of type sulphonated naphthalene condensate or sulphonated melamine-formaldehyde condensate. No precipitation occurs.

3 Experimental

3.1 Materials

The following materials were used in all mixes:

- Ordinary portland cement with the specific area, 360 m^2/kg.
- Coarse aggregate (8-16 mm): crushed quartzite.
- Fine aggregate (0-8 mm): natural gravel of high quality, mainly granite.
- Air-entraining agents:
  (1) SJ-2 as described above
  (2) for comparison, Vinsol Resin with the tradename Cementa 88L. A water solution of Vinsol Resin with the concentration 20.8% of dry AEA was used.

3.2 Concrete mixes and specimens

In total 7 mixes were produced:

- one non-air-entrained mix
- three mixes with Vinsol Resin and three different air contents
- three mixes with SJ-2 and three different air contents.

All mixes had the W/C-ratio 0.50. No superplasticizer was used in any mix. All mixes had plastic consistency. The fresh air content was determined by the pressure method.

The detailed mix proportions, the fresh air content and the slump are shown in Table 2 together with the density and the 28-days compressive strength of the hardened concrete.

Table 2: Mix proportions and properties of the fresh and hardened concrete

Mix (1)	W/C	Cement kg/m^3	Sand kg/m^3 (2)	8-12mm kg/m^3	12-16mm kg/m^3	AEA kg/m^3 (3)	Slump mm	Air %	Density kg/m^3	Strength MPa
0	0.50	380	920	449	449	0	45	1.6	2400	61.2
V1	0.50	364	881	430	430	0.862	60	4.4	2323	49.1
V2	0.50	360	870	424	424	1.418	71	5.8	2285	45.1
V3	0.50	352	853	416	416	3.086	78	7.9	2219	34.8
C1	0.50	362	876	428	428	0.0362	50	5.0	2309	50.5
C2	0.50	354	856	418	418	0.0707	62	7.5	2231	40.1
C3	0.50	347	840	410	410	0.1096	74	9.5	2167	29.8

Notes:
(1) Mix V1-V3 based on Vinsol Resin
Mix C1-C3 based on SJ-2
(2) The moisture content of the sand was 2.4 weight-%
(3) For Vinsol Resin, the weight given includes water. For JS-2, the weight given is the dry weight

A number of 10 cm cubes were cast from each mix. The specimens were demoulded the day after casting and were then stored in water until they were 28 days old when the freeze-thaw test started and the compressive strength was determined.

3.2 Salt-frost scaling tests

In order to evaluate the effect of the two types of air-entraining agent on the salt-frost resistance of concrete a salt scaling test was performed. The test is similar to the international methods recommended by CEN/TC51/WG12, [3] and RILEM, [4].

After water curing for 28 days, the cubes for freeze-testing were completely submerged in 3% NaCl-solution in plastic containers which were covered by a tight lid during the test. One cube was placed in each container. Two cubes were used for each concrete mix. One of these specimens was placed in a "large" container and the other in a "small" container. By "large" is meant that a there was room for more solution. This caused a slower freeze-thaw cycle. The containers with their specimens are shown in Fig. 1, and the freeze-thaw cycle is shown in Fig. 2. The temperature was measured in the solution at the surface of the specimen. Each freeze-thaw cycle had a duration of 24 hours. The test was interrupted after 56 cycles (56 days).

Frost action caused a certain surface scaling. All surfaces of the cube were rinsed using the salt solution in the container and a soft brush. All particles that scaled from the specimen were collected in a paper filter (coffee filter). The filter with the scaled material was dried to constant weight at +106°C and weighed, whereafter the weight of the filter itself was subtracted. The measurement of the scaling was normally made each 7 days (each 7 cycles) up to 56 days when the test terminated.

3.3 Air-pore analysis

The geometrical characteristics of the air-pore system, such as the air content and the specific area was determined by the linear traverse technique using an automatic image analysis system. From these data the Powers spacing factor could be calculated. The calotte diameter distribution seen by the image analyser is used for calculating a pore diameter distribution using the theory in [5].

Before a measurement could be made the surface of the specimen had to be carefully polished and thereafter coloured so that each air-bubble could be clearly discerned as a small white circular spot against a dark background. The polished surface was stained by black or blue ink that was dried at +106°C for some minutes. After cooling, the surface was covered by a zinc white paste which was scraped off so that the white paste only filled the tiny cavities left by the air-voids.

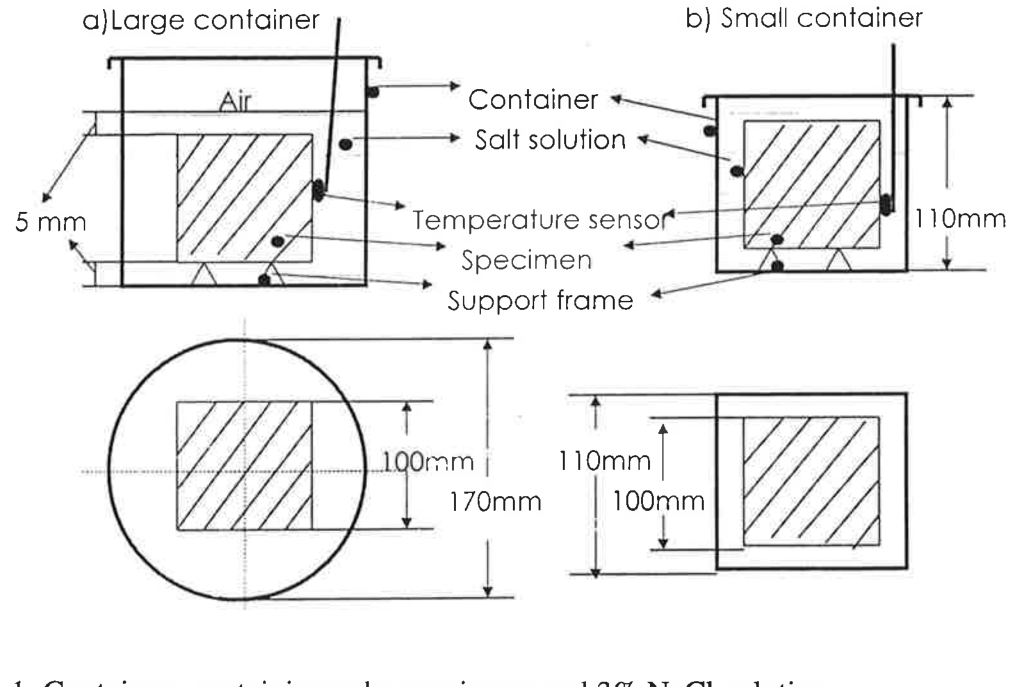

Fig. 1 Containers containing cube specimens and 3% NaCl-solution.

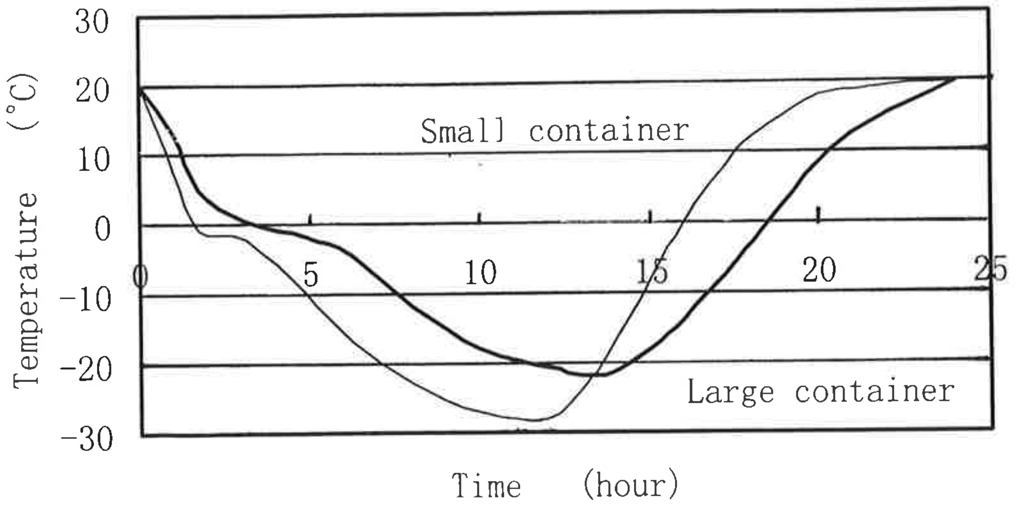

Fig. 2 The freeze-thaw cycles measured in the salt solution.

4 Results and discussion

4.1 The salt-frost test

The results ot the salt-frost test is shown in Appendix 1 and in Fig. 3 and 4. The following results were obtained:

1: The non-air-entrained concrete was severely scaled. The test had to be discontinued already after 28 cycles.

2: For air-entrained concrete the scaling increased with increased number of freeze-thaw cycles. For low scalings the relationship between scaling and number of cycles was more or less linear. For high scalings, this was accelerated with increasing number of freeze-thaw cycles.

3: The scaling decreased with increased air content. According to the Swedish Standard SS 13 44 27 a concrete is regarded as having *good* salt-frost resistance if the scaling after 56 cycles is below 0.5 kg/m^2, and *excellent* salt-frost resistance if it is below 0.2 kg/m^2. The limit for "good resistance" was obtained with about 6-7% of air. In order to reach the limit for "excellent resistance" about 9% of air was neeeded. Then it must be considered that the Swedish limits are based on a different test method, where the specimen is both exposed to salt and frozen from one side only, and where the specimen is pre-dried once before the test start. Possibly, this procedure leads to smaller scalings. The water-cement ratio used was fairly high, 0.50. This could contribute to the comparably high required air content. By lowering the w/c-ratio to 0.40-0.45 a lower air content might have been sufficient.

4: The larger container generally gave bigger scalings. One reason behind this behaviour could be the slower freezing-thawing rate.

5: There is no difference between the two types of air-entraining admixture. Both are able to produce frost resistant concrete at about the same air content.

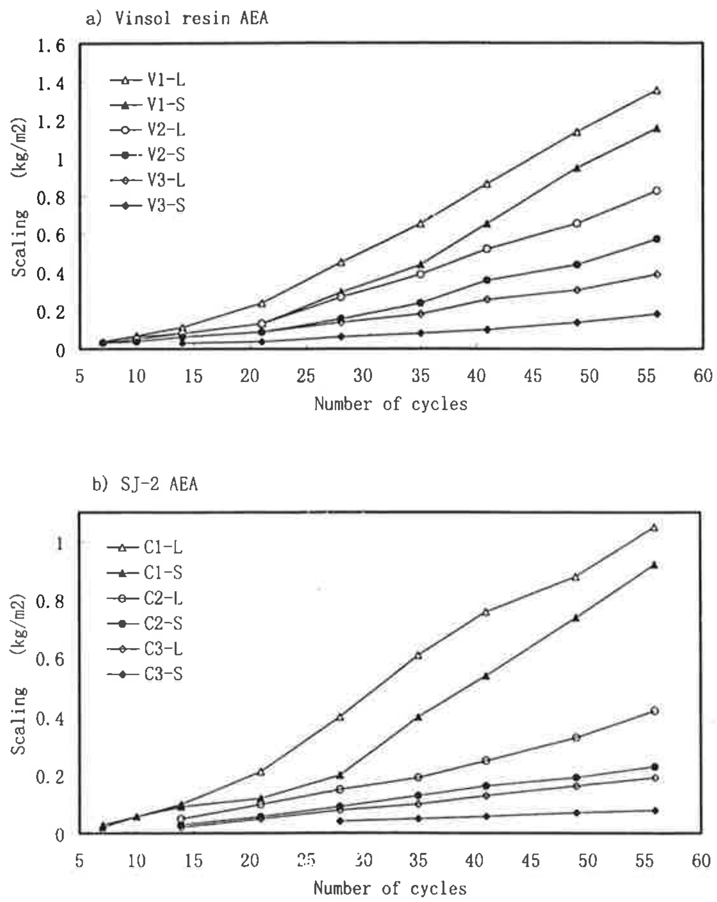

Fig. 3 The salt-frost scaling as function of the number of freeze-thaw cycles.

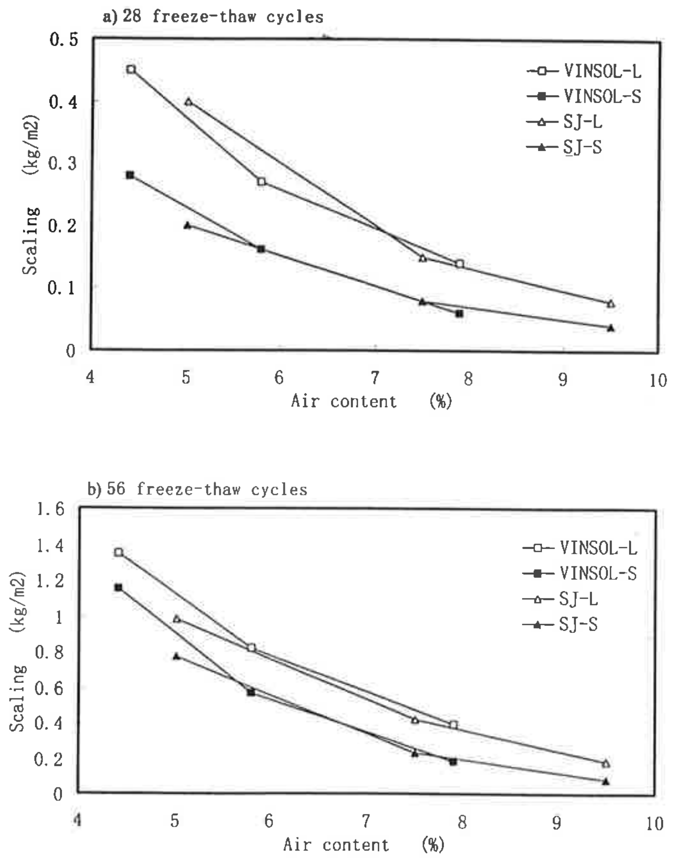

Fig. 4 Relationship between the air content of the fresh concrete and the salt-frost scaling.

4.2 The compressive strength

The strength results are shown in Table 2 and Fig 5. The following results were obtained:

1: The strength decreases when the air content increases. At the same air content the strength is about the same irrespectively of the type of air-entraining agent. See Fig. 5(a).

2: For one percent increase in the air content the strength reduction is about 5-6%. There is a slight tendency of a lower strength loss when the air-entraining agent SJ-2 is used, see Fig. 5(b). The experimental material is however too small to draw firm conclusions concerning eventual differences between the two admixtures.

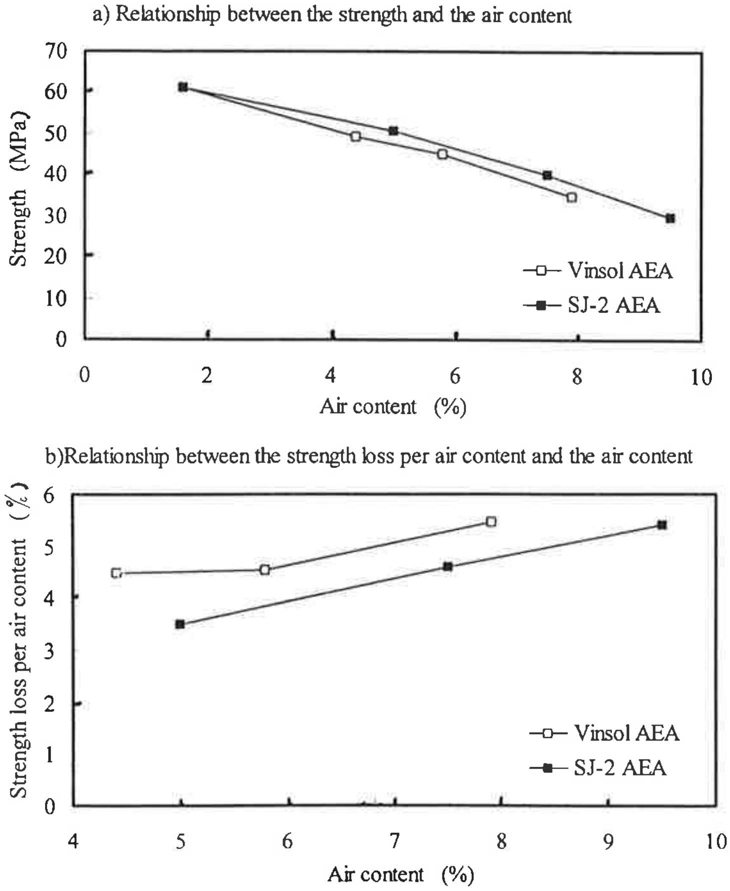

Fig. 5 Relationship between the air content of the fresh concrete and the cube strength.

4.3 The air-pore structure

The air-pore parameters are shown in Appendix 1 and in Fig. 6. Examples of the size distribution of the air-pore diameters is seen in Appendix 2. The following results were obtained:

1: The specific area is high and about the same for both types of air-entraining agents, see Fig. 6(a). There is one concrete with the air content 5% for which the specific area is bigger when SJ-2 is used. This is only one result of this type, however, and, therefore, it is hardly significant.

2: The fact that the specific area is about the same means that the spacing factor is also about the same for both admixtures when the air content is constant, see Fig. 6(b).

3: The correlation between the air content of the hardened concrete and the fresh concrete is excellent, cf. Table 2 and Appendix 1. This indicates that the stability of the entrained air-pore sysem is excellent for both types of admixture. The following regression line is obtained:

$$a_{hardened}=a_{fresh} - 0.31\ (\%) \qquad (1)$$

Thus, the air content loss is only 0.3% which is an exceptionally low value.

The similar air-pore structure obtained with the two types af air-entraining agents could explain why the salt-frost scaling of concrete made with the two admixtures is almost the same at equal air contents.

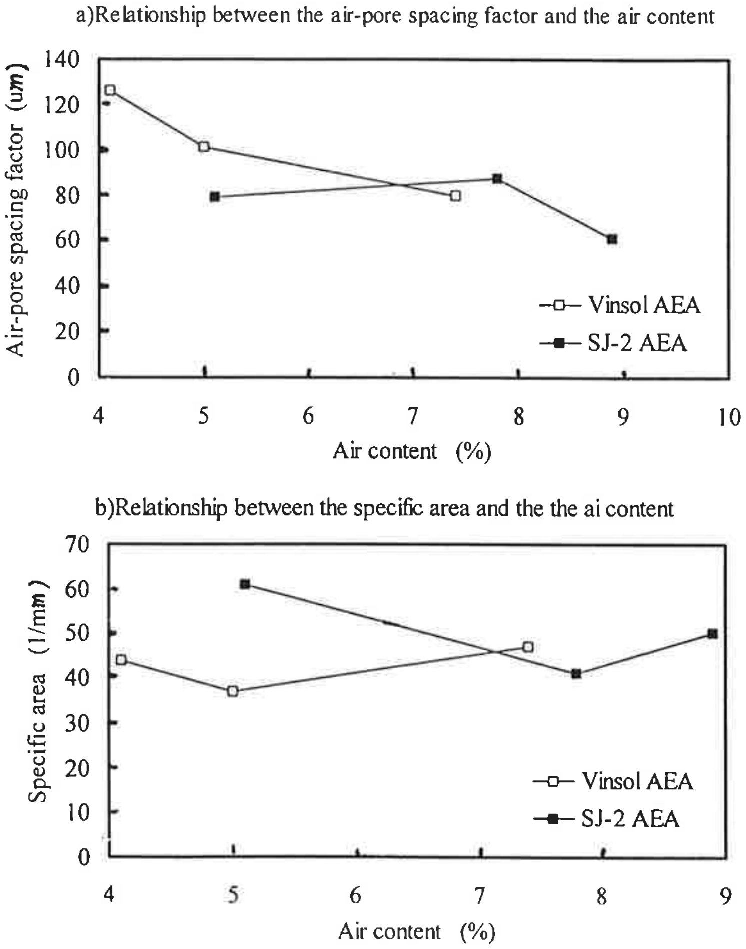

Fig. 6 Effect of the air content on the spacing factor and specific area of the air-pore system.

4.4 Relationship between the Powers spacing factor and the salt-frost resistance

There are many studies showing a fairly close relationship between the salt-frost scaling and the spacing factor, the larger the spacing factor the lower the frost resistance, [1], [6]. Also this study indicated a certain relationship betwen scaling and spacing factor, see Fig. 7. For Vinsol Resin the relationship is fairly linear. For SJ-2 the relationship is less well-defined.

The low spacing factors required for concrete with low scaling is unexpected. Normally, spacing factors of the order 0.18-0.20 mm are sufficient. There is no good explanation of this result. One reason might be that the air-pore system absorbs water before and during the test. This possibility has been studied in [7] where it is shown that a water absorption in air-pores implies that the theoretical background to the Powers spacing factor can be questioned. Theoretically there could not exist one single critical Powers spacing factor that is valid for all concretes. In [7] it is also shown that fine air-pores absorb water more readily than big pores. Therefore it is not necessarily an advantage to have a fine-porous air-pore system.This is further discussed in [8]. A more important observation made in the present study is that high salt scaling resistance can be obtained if the air content is high enough and not necessarily if the Powers spacing factor is low enough.

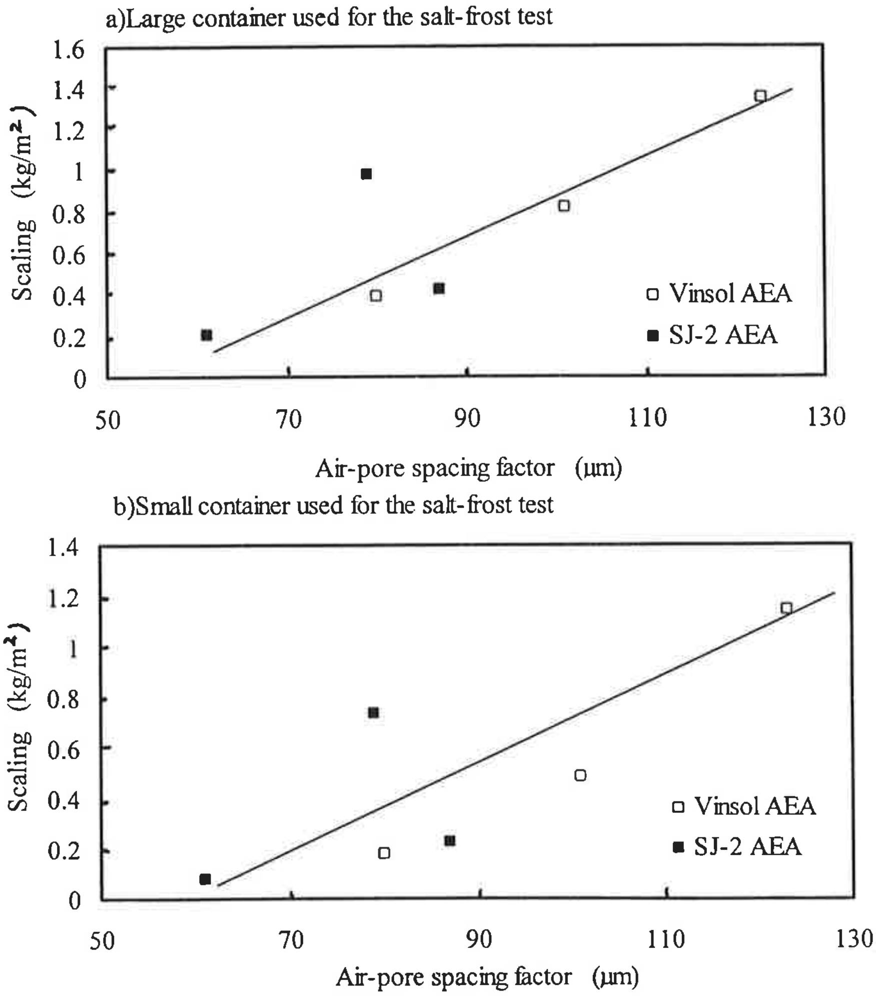

Fig. 7 Relationship between the salt-frost scaling and the spacing factor.

5 Summary

Both air-entraining admixtures investigated significantly improve the salt-frost resistance of concrete. There is no significant differences between the two air-entraining agents, SJ-2 and Vinsol Resin. Both admixtures give about the same air requirement for the same maximium allowable scaling. Both admixtures produce air-pore systems of about the same fineness. The investigation is too limited to state whether the small differences observed in the air-pore structure are significant or not or if they are reproducible.

The strength loss when the air content is increased is almost the same for both admixtures, as expected. There is a slight tendency of a smaller loss in strength with SJ-2. Due to the limited investigation performed it is not possible to say if this small change is significant or not.

Both admixtures give air-pore systems that ate remarkably stable during casting. The air content of the hardenbed concrete is almost the same as for the fresh concrete.

The investigation shows that the new air-entraining admixture **SJ-2** ought to be just as well suited for production of concrete with high degree of salt-frost resistance as the well-tried admixture Vinsol Resin.

Acknowledegements

This research work was carried out by the author as a guest researcher at the Division of Building Materials, Lund Institute of Technology. I deeply appreciate the support and advice given by professor Göran Fagerlund.

I would like to express my gratitude to all collegues who assisted me during this study. Special thanks are directed to Mr. Bo Johansson for his kind help during manufacturing concrete and measuring the strength, to Ms. Katja Nordstöm, Mr. Sture Lindmark and Mr. Ingemar Larsson for their friendly cooperation and help during the measurements of the salt scaling and in making the image analysis. Finally, I am grateful to Professor Huang Shiyuan for his correcting the final manuscript.

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APPENDIX 1: Results of the salt-frost test and the air-pore analysis

Type	Scaling after a given number of cycles, kg/m²									Air-pore parameters		
	7	10	14	21	28	35	41	49	56	air %	Specific area mm^{-1}	Spacing factor mm
O-L	0.50	1.21	2.70	5.47	8.19					1.5	16	0.53
O-S	0.33	0.88	1.92	3.97	6.03							
V1-L	0.04	0.07	0.11	0.24	0.45	0.65	0.86	1.13	1.35	4.1	41	0.13
V1-S	0.04	0.06	0.08	0.13	0.28	0.44	0.65	0.94	1.15			
V2-L	0.03	0.05	0.08	0.13	0.27	0.39	0.52	0.65	0.82	5.0	37	0.10
V2-S	0.03	0.04	0.06	0.09	0.16	0.24	0.36	0.44	0.57			
V3-L			0.06	0.09	0.14	0.18	0.26	0.31	0.39	7.1	49	0.08
V3-S			0.03	0.04	0.06	0.08	0.10	0.14	0.18			
C1-L	0.03	0.06	0.10	0.21	0.40	0.61	0.76	0.89	0.98	5.1	61	0.08
C1-S	0.02	0.06	0.09	0.12	0.20	0.40	0.54	0.65	0.77			
C2-L			0.05	0.10	0.15	0.19	0.25	0.33	0.42	7.8	41	0.09
C2-S			0.03	0.05	0.08	0.11	0.14	0.19	0.23			
C3-L			0.02	0.05	0.08	0.10	0.13	0.17	0.21	8.9	50	0.06
C3-S					0.04	0.05	0.06	0.07	0.08			

APPENDIX 2: Examples of air-pore diameter distributions