



FOAM STABILITY EXPERIMENTS ON SOLUTIONS CONTAINING SUPERPLASTICIZING- AND AIR-ENTRAINING AGENTS FOR CONCRETE

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SYNOPSIS

Combined use of superplasticizers and air-entraining agents - each with a well documented behaviour in concrete - has on some occasions resulted in bad air-void-systems in the hardened concrete and thereby reduced frost resistance. A simple method to predict the nature of the resulting air-void system has been developed through experiments including measurements of surface tensions and foam heights and foam stabilities of the agents in solution. Concrete mixes have been made on laboratory scale and the air-pore-systems have been measured.

Key-words: Superplasticizers, air-entraining agents, foam, flowing concrete, air-void-system, surface tension.



1. INTRODUCTION

To obtain an acceptable durability of a concrete in a freeze-thaw and/or aggressive environment, a low water-cement ratio and a well entrained air-void-system are required. Furthermore, a good workability of the fresh concrete is often wanted.

The requirement of a water-cement ratio, typically lower than 0.45, combined with the requirement of an easy-workable concrete, has implied the use of a superplasticizing admixture. In the past few years, the use of superplasticizing admixtures has increased continuously in the concrete industry.

A number of combinations of air-entraining and superplasticizing admixtures, each having a well documented behaviour, has behaved badly when the objective was to obtain a good air-void-system in the hardened concrete. On some occasions entrained air simply disappears and sometimes one gets an air-void-system consisting of large, unwanted air-voids, all of which reduces the frost resistance and the durability of the concrete. This experience was gained from a number of consulting cases from the concrete industry, mostly from ready-mix concrete suppliers.

In this investigation compatibility of different types of admixtures, altogether 39 admixtures, were evaluated. Experiments have included measurements of surface tensions and foaming abilities of solutions of the admixtures alone and in combinations. Also concrete mixes (flowing concrete) have been made for selected combinations of admixtures and the air-void-systems have been measured on the hardened concretes.

Due to lack of knowledge of the exact chemical compositions of the commercial admixtures, the project does not seek to explain the mechanisms of the performances of different combinations of admixtures. A number of observations has been made and it is the authors' hope that these observations will be of use in formulating deeper investigations concerning actual mechanisms.

2. MATERIALS AND METHODS

2.1 Admixtures

Commercial superplasticizers, plasticizers and air-entraining agents are represented in the project as follows:

- 8 air-entraining agents of Vinsol Resin type (marked VR)
- 8 air-entraining agents of synthetic type (marked S)
- 8 superplasticizers of naphthalene-based type (marked N)
- 4 superplasticizers of melamine-based type (marked M)
- 7 combined superplasticizers of naphthalene + lignosulphonate type (marked N+L)
- 4 plasticizers of lignosulphonate type (marked L).

In this article only admixtures from the four first mentioned groups (VR, S, N, and M) will be discussed as the results concerning lignosulphatecontaining admixtures only are few and concrete trials have not been performed with these admixtures.

2.2 Basic solutions of admixtures

Measurements of surface tension and foam stability have been made on basic solutions of the admixtures in water, where the dosage of admixture, respectively combinations of admixtures, is the same as recommended for use in concrete and related to the water phase in a concrete with a water-cement ratio of 0.5.

Example: Agent No. S19 has a recommended dosage of 0.05% (of weight of cement). Related to the water phase in a concrete with $w/c = 0.5$, the concentration of S19 will be 0.10%. The basic solution of S19 then contains 1.0 g of S19 and 1000 g of water.

2.3 Concrete mixes, materials

In the concrete trials a rapid hardening Portland-Cement, supplied by Aalborg Portland A/S, Denmark, was used (300 kg/m^3). Aggregates consisted of 40% (weight) quartz sand and 60% sea gravel (8/18 mm) of a rounded shape.

In mixes with only air-entraining agents added, the water-cement ratio was 0.57 to obtain a high slump ($200 \text{ mm} \pm 20 \text{ mm}$).

To obtain the same slump in mixes with superplasticizers + air-entraining agents, the water-cement ratio was 0.45.

2.4 Surface tensions

On selected basic solutions of single admixtures and combinations of admixtures the surface tensions in mN/m have been measured using a Krüss Tensiometer, plate method.

2.5 Foam stabilities

A 100 ml measuring cylinder with 1 ml graduation and a tight-fitting stopper was used in the foam test. 50 ml of the basic solution was placed in the cylinder which was closed and then shaken vigorously by hand in 30 sec. Then the cylinder was placed on a table and the foam height in mm was measured at 1, 10, 30, and 60 minutes respectively, after the shaking. Furthermore, the foam was described (small/big bubbles).

In the same way foam heights were measured where 1.00 g of cement was added to the 50 ml of basic solution before shaking.

2.6 Concrete trials

Concrete mixes were made in the laboratory in a 50 l Eirich mixer with a rotating container. Each mix was 35 litres. The mixing procedure was as follows:

- Cement is added to aggregates + $3/4$ of the water and mixing starts
- In mixes with both superplasticizer and air-entraining agents the superplasticizer is added immediately together with the rest of water. Mixing time is 120 sec. and thereafter slump and air content (pressure-meter) are measured

- The mixing continues and air-entraining agent is added immediately. Mixing time is 150 sec. Then again slump and air content are measured. Cylinders are cast (stamping) for measurement of the air-void-system in the hardened concrete.
- Mixing continues again, now for 300 sec., there- after slump and air content are measured. Total time elapsed since start of mixing is about 15 minutes.

Measurement of the air-void-system on the hardened concrete took place on an automatic image analyser according to ASTM C 457-71.

3. RESULTS AND DISCUSSION

Only part of the results will be presented in the following text as the space does not permit presentation of i.a. the several hundred foam tests. The chosen results are typical for the different groups of admixtures and combinations of admixtures.

The results are presented in three series:

- Series 1: Measurements of surface tension of basic solutions
- Series 2: Foam heights versus time are presented graphically for air-entraining agents, respectively air-entraining agents in combination with superplasticizers (all in basic solutions)
- Series 3: Concrete mixes including measurement of air content in the fresh state and measurement of the air-void-system on hardened concrete.

3.1 Series 1, surface tension

In Table 1 results of surface tension measurements are shown for basic solutions of synthetic and vinsol resin air-entraining agents, respectively superplasticizers of naphthalene and melamine type. Also the dosage of admixtures is shown (weight % of water). The dosages are related to the dry matter content of the admixtures and the recommended dosages in concrete.

It is seen that combinations of synthetic air-entraining agents and superplasticizers of the melamine type give results of surface tensions which are very low, even in some cases lower than the pure synthetic agents. This could be explained by some kind of interaction between agents. Other combinations give higher results.

Table 1. Surface tension measurements, mN/m.

Air- entrain- ing agents	Superpla- stici- zers	÷	N3 2.0%	N8 2.0%	N12 2.2%	M4 2.2%	M9 2.0%	M10 4.0%
÷			73.8	66.4	68.0	73.5	71.8	64.0
S19	0.1%	45.6	56.8	57.0	51.0			32.5
S25	0.12%	32.2	68.5	67.7	57.8	38.5	38.4	37.5
S28	0.08%	50.0	72.4	68.8	67.6	46.6	51.3	47.0
VR26	0.12%	56.3	65.4	67.9	63.5	50.3	52.8	58.4
VR27	0.12%	57.9	60.0	63.5				61.3
VR39	0.2%	48.8	55.1					

3.2 Series 2, foam stability

In Figures 1-6 foam heights in mm measured at 1, 10, 30, and 60 minutes after shaking of the basic solutions are shown. Only results of basic solutions in water are presented as these give a clear distinction between agents. In the foam tests where 1 g of cement was added the picture is not so clear /1/.

Comments on Figures 1 and 2:

There is a marked difference in the foam heights and foam stabilities between the synthetic and vinsol resin agents. The synthetic agents give a higher and more stable foam, while the vinsol resin agents lose much of their original foam heights in 10-30 minutes.

Comments on Figures 3 and 4:

Combinations of synthetic air-entraining agents and naphthalene-based superplasticizers in most cases give a quick drop in foam height, approaching zero after 10-30 minutes.

Combinations of vinsol resins with naphthalene-based superplasticizers give a foam stability which is, in general, better than for the vinsol resins alone.

Comments on Figures 5 and 6:

Combinations of synthetic air-entraining agents and melamine-based superplasticizers give foam heights and stabilities which are better or similar to the pure synthetic agents.

Combinations of vinsol resins and melamine-based superplasticizers show a picture which resembles the results from Fig. 4 (vinsol resins + naphthalene-based superplasticizers).

General comments:

Double determination of foam heights were carried out in a number of cases and the maximum deviation has been found to ± 10 mm for the largest foam heights.

Where the foam drops quickly (synthetic agents + naphthalene-based agents) the bubbles are registered as large. In the other cases the foam consists of small, dense bubbles.

When we compare the foam test results with the measurements of surface tension, it is seen that there is no clear relation between foam stabilities and surface tension except for the combination of synthetic + melamine agents.

Foam height

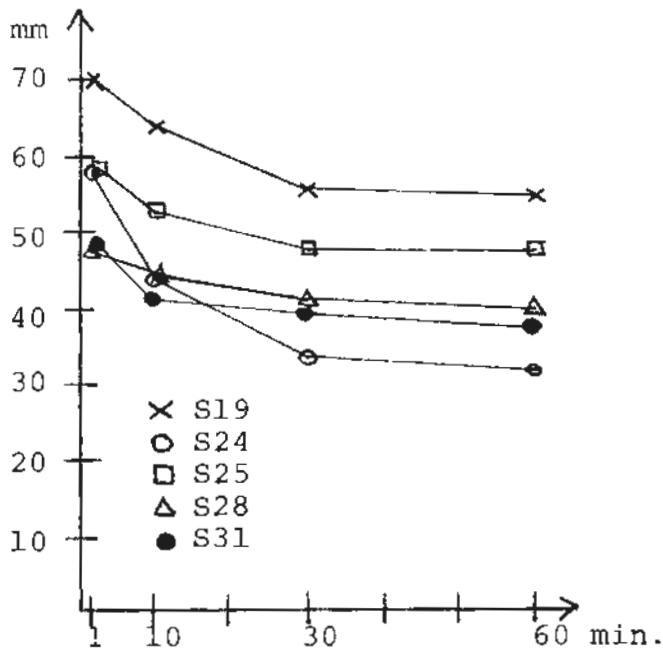


Fig. 1. Synthetic air-entraining agents

Foam height

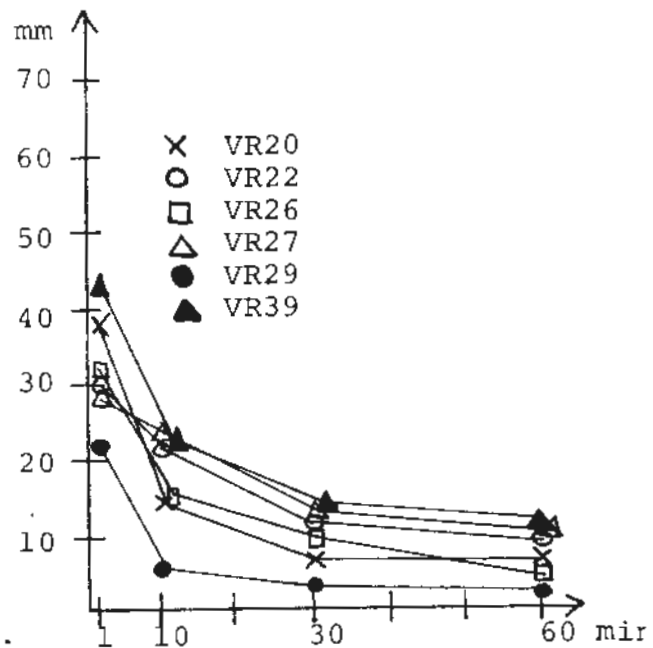


Fig. 2. Vinsol resins

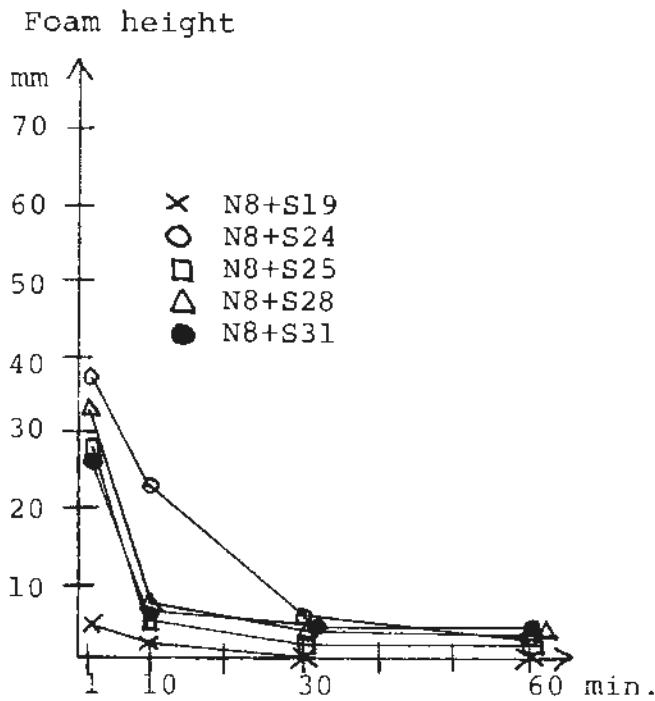


Fig. 3. Naphthalene-based agents combined with synthetic agents

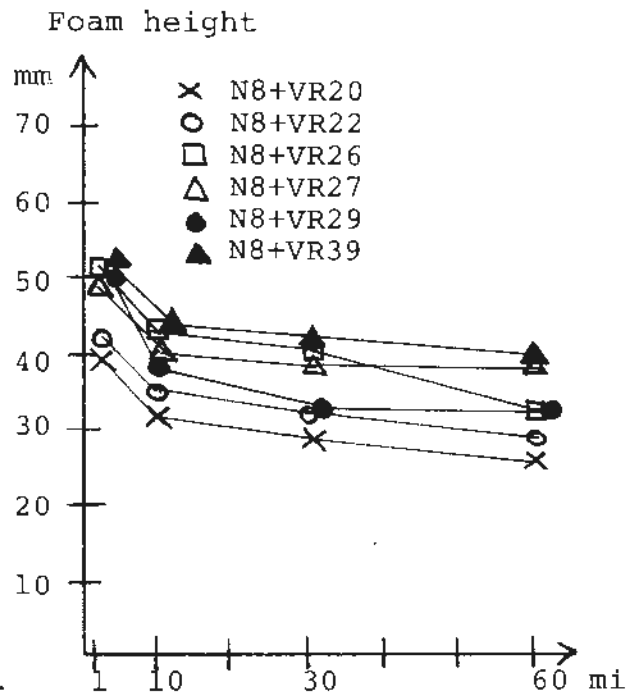


Fig. 4. Naphthalene-based agent combined with vinsol agents

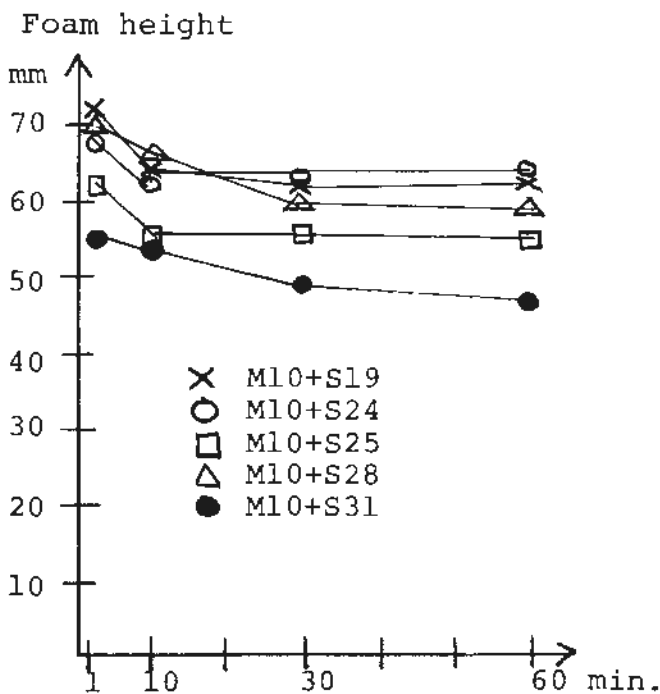


Fig. 5. Melamine-based agents combined with synthetic agents

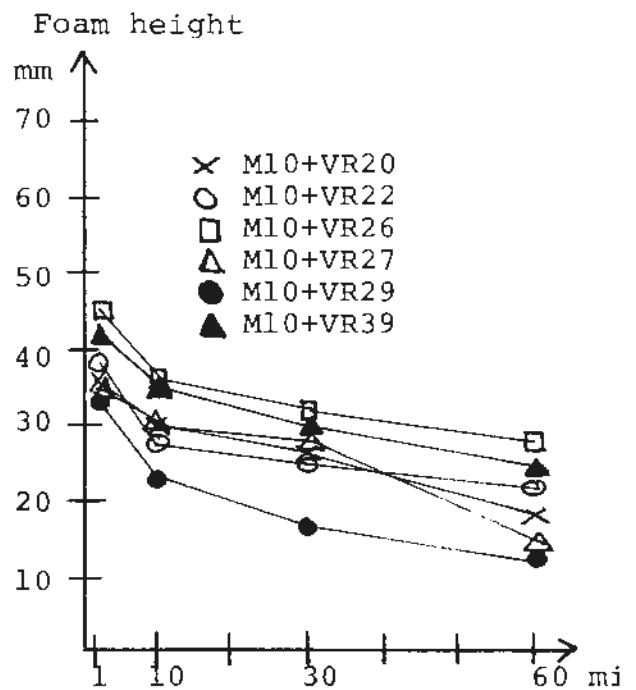


Fig. 6. Melamine-based agents combined with vinsol resin

3.3 Concrete mixes

In the concrete trials the aim was to make a flowing concrete where the problems in obtaining a well entrained air-void-system were highest according to our experience.

In concretes with only air-entraining agent, the water-cement ratio is 0.57 to obtain a high slump. The authors are well aware that the rheological conditions in this concrete are different from the conditions in a superplasticized concrete with a lower water-cement ratio (0.45), but of the same slump.

In Table 2 results are listed for different concrete mixes including measurement of the air-void system. Slump and air content (fresh state) are measured at the following steps (marked 1, 2, and 3 in Table 2):

1. After 120 sec. mixing time with or without superplasticizer added (without when only air-entraining agent is tested).
2. After 150 sec. additional mixing time with air-entraining agent added.
3. After 300 sec. further mixing time. Total time elapsed since start of mixing is about 15 minutes.

The air-void-system is measured on cylinders cast after step 2.

Concerning the mixes with only air-entraining agents, it is seen that there is nearly no slump loss and no reduction of air content after prolonged mixing time. The measured values of specific surface of the bubbles in the hardened concrete indicates that the air-pores are of satisfactory small sizes.

In mixes with superplasticizers a more or less pronounced slump loss is observed - as is also known from general experience in practice.

Concerning air content in the fresh state, it is seen that naphthalene-based superplasticizers in themselves entrain air, while the melamine-based superplasticizer has no influence.

The dosages of air-entraining agents in the mixes with superplasticizers are the same as in the mixes with only air-entraining agents.

For the synthetic agents combined with superplasticizers of the naphthalene type, the air-entraining effect is small, and even a reduction in the original air content is observed. The values of specific surface are low and the air-void-system is not acceptable.

Melamine-based superplasticizer combined with synthetic agent gives a result like the air-entraining agent itself (good).

Table 2. Concrete mixes.

Agents	Slump, mm			Air content, % fresh state			Air content, % hardened state	Specific Surface	Spacing factor
	1	2	3	1	2	3	2 (3)*	mm ⁻¹	mm
S19	215	235	215	1.0	8.0	8.5	7.7	39	0.09
S25	210	220	215	1.4	6.4	6.5	4.9	36	0.13
VR27	220	230	220	0.9	6.2	6.1	5.8	34	0.13
S19 + N8	190	175	110	3.6	5.3	4.4	5.8	18	0.25
S25 + N8	230	220	180	4.0	3.2	2.1	2.9	19	0.32
S19 + M10	205	190	110	1.2	9.0	8.5	9.1	34	0.09
VR27 + N8	235	220	180	4.3	8.0	4.7	8.6 (3.5)*	23 (32)*	0.14 (0.18)*
VR27 + N12	190	190	75	4.1	8.5	4.9	7.1	28	0.14
VR27 + M10	215	200	120	1.0	7.1	4.9	7.1	31	0.12

*) Values measured on cylinder cast after step 3.

With vinsol resin combined with both naphthalene and melamine-based superplasticizers an air-entraining effect is observed, but also a drop in air content after prolonged mixing time. The results concerning the air-void-system are acceptable.

4. CONCLUSIONS

Combinations of air-entraining and superplasticizing admixtures in concrete give different resulting air-void-systems in the hardened concrete according to the type of agents.

It has been found that a simple foam test indicates whether combinations of air-entraining agents and superplasticizers will succeed or not in the making of an air-entrained concrete with an acceptable air-void-system. When the foam test with combination of agents give a lower and more unstable foam than the foam test of the air-entraining agent alone, problems have been observed in the fresh concrete state as well as in the hardened concrete.

Research work concerning actual mechanisms of the action of the abovementioned admixtures in concrete is still in progress.

ACKNOWLEDGEMENT

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- /1/ Eriksen, K., Andersen, P.J. & Jensen, A.D.:
Projektrapport: 1. Kombineret anvendelse af luftindblandings- og superplastificeringsstoffer i beton. 2. Skumtest. 3. Resultater. (Project Report: 1. Combined use of air-entraining and superplasticizing admixtures in concrete. 2. Foam test. 3. Results.)
Teknologisk Institut, 1985.