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Effect of AEA-SP Dosage Sequence on Air Entrainment in FA Concrete

Andrei Shpak
Ph.D. candidate
Norwegian University of Science and Technology,
Richard Birkelands vei 1a, NO-7034 Trondheim
e-mail: andrei.shpak@ntnu.no

Stefan Jacobsen
M.Sc., Ph.D., professor
Norwegian University of Science and Technology,
Richard Birkelands vei 1a, NO-7034 Trondheim
e-mail: stefan.jacobsen@ntnu.no

ABSTRACT

Laboratory measurements show that varying the dosage sequence of air-entraining agent and co-polymer in the mix (SP added before, after or together with AEA) greatly affects air entrainment in fresh and hardened fly ash concrete. Image analysis shows a somewhat lower specific surface when SP is added together with AEA. Foam Index measurements on the same binder materials, admixtures, and dosage sequences were therefore found less useful for studying the effect of admixture combinations. Obtaining a certain air content using the experience with AEA-SP dosage was found to be an untrivial task if there is a lack of parameter control. Finally, examples of successful mixing procedure for air entrainment in a series of high-volume fly ash concrete are shown.

Keywords: Air entrainment, Dosage sequence, Fly ash, Admixtures.

1. INTRODUCTION

Most of the studies on air entrainment and air-void stability in fly ash (FA) concrete focus either on the effectiveness of certain admixtures or on the influence of different types of FA. And there is a general agreement on that properly air-entrained FA concrete simply requires a higher dosage of AEA to compensate for the loss of active ingredient to unburned carbon in FA. However, thus far, the production of frost durable FA concrete with a stable and protective air void system has still proven to be difficult. The problem has been ascribed to the variable carbon content in the fly ash causing variations in the required dosage of an air-entraining agent (AEA) [1]. Additionally to the carbon, other contributors to adsorption of AEA can be hollow FA spheres (cenospheres) and FA spheres filled with numerous small spheres (plerospheres) [2]. A common measure of the increase in dosage of AEA to compensate for the loss of the active ingredient to carbon cannot take variations in fly ash properties into account, and that entails unwanted variations in air entrainment [3]. Trial mixing to ensure quality output is therefore unavoidable even for batch-to-batch variations in fly ash.

The problem can hypothetically be resolved by reducing the number of sorption sites on carbon before the AEA encounters them. Justnes and Ng [4] stated that adsorption of the active ingredient of AEA by carbon in FA can be solved by increasing AEA dosage or add some "sacrificing admixtures that will preferentially adsorb to the carbon". They also assume that it is likely that carbon may preferentially adsorb other organic admixtures like (super-) plasticizers. Plasticizing admixtures will still be attracted the most by AFt phases and alite to disperse the cement, and also FA particles (the glass phase) have some interaction with the admixture, though weaker than with cement and weakest among all the SCMs [5].

Therefore, we think that superplasticizer (SP) could block access of AEA to some carbon in one of the AEA-SP combinations.

Previous measurements have shown large effects on foaming in OPC (ordinary Portland cement) -fly ash water slurries of various SP/AEA combinations and dosage sequences [6]. The foam study indicated that a combination with SP drastically affects the adsorption kinetics. The same materials from that study [6] were also used to investigate the effect of the addition of the admixtures on air entrainment [7].

The sequence of the addition of SP and AEA in concrete has been debated among practitioners for a long time, but the authors do not know any experimental studies of SP-AEA dosage sequence in FA concretes in the literature. For OPC concrete, some authors [8, 9], suggest adding AEA after blending SP in the mix to give a stable air-void spacing factor; others [10–13] say that SP should be added after AEA, providing time for AEA to precipitate.

No standards, committees, or guidelines specify the AEA-SP interaction [14]. Moreover, there is no documentation provided by concrete admixture producers about the compatibility of AEAs and SPs. According to specialists from a Norwegian admixture producer [15], all admixtures get pre-qualified separately from other admixtures. In the company standard, for example, AEA is tested in OPC concrete, targeting slump at 50mm and 4-6% total air content. It is not understood how the admixture producers announce the compatibility of admixtures without providing the meaning of it. In addition, we know of only two studies [16, 17] which revealed the composition of air-entraining agents, and it makes it impossible to assess the performance of AEA without trial mixing.

In the industry, SP-AEA dosage sequence practice varies due to the limitations of the concrete plant, economic reasons, or the producer's or client's established practice. The concrete producers reviewed in this study recommend that AEA is added either before or simultaneously with SP in the concrete mixes containing either pre-blended or separately added FA. Also, the mixing time varies from one to two minutes depending on strength and durability class, and in case of sampling – it increases to 3 minutes. Variability of the production parameters, the inexistence of the regulations and maybe some inaccuracy of the concrete producers reduce chances to control air entrainment in FA concrete.

With an increased need for high volume fly ash concrete the need for real knowledge about AEA and SP interaction in FA concrete is growing. Yet worth mentioning that in the near future the availability of "pure" FA may be reduced due to combusting coal together with waste products (rubber etc.), which might complicate the task of making concrete which fulfills the demands for XF4.

The scope of this work was to investigate air void content and structure from laboratory Fly Ash concrete mixes where both the type of AEA and the dosage sequence of AEA- and a co-polymer SP were varied. If effective, it would be a practical and simple way of remedying the problem.

2. MATERIALS AND MIXES

Two main series of concrete mixes (d_{max}>6mm) were made to investigate the effect of admixture combinations and dosage sequence on air-void parameters:

- ***M-series*** [7] where compositions were constant while comparing the effect of dosage sequence
- ***O-series*** [18] where two different binder types were investigated and where much more emphasis was put on controlling workability and total air content by varying AEA dosage, which is more related to practice.

2.1 Constituent materials

Table 1 – Material parameters

Material	Density [kg/m^3]	Carbon [%]	Loss on ignition [%]	Blaine [m^2/kg]
M-series				
Norcem Standard cement (CEM I 42,5R)	3 150		2,35	396
Norcem Fly Ash	2 300	1,74	2,27	334
Limestone filler	2 730		37,66	362
O-series				
Norcem Anlegg cement (CEM I 52,5N)	3 140	0,42	2,33	360
Norcem Anlegg FA cement[1] (CEM II/A-V – 42,5N)	3 020	0,79	2,74	384
Norcem Fly Ash, LN3-17	2 310	3,01	3,16	334
Silica Fume 940D	2 200			

[1] Norcem Anlegg FA cement contains 14,1% fly ash as a replacement by mass

[2] Carbon content in fly ash was measured by ELTRA (combustion and infrared detection)

Table 2 – Aggregate grain-size distribution

Aggregate	Cumulative [%] passing for sieve opening [mm]															
	11,2	8	5,6	4	2	1	0,5	0,25	0,125	0,063	0,032	0,016	0,008	0,004	0,002	0,001
Sand 0-8	100	98,9	89,7	79,8	62,1	44,6	28,8	16,1	7,2	2,7						
Limestone filler									100	87,2	67,1	46,7	30,1	18,7	9,9	3,3

Table 2 shows the aggregate size distribution. It was the standard Norwegian gneiss-granitic 0-8 mm sand supplied by NorStone Årdal.

Admixtures

Anionic air-entraining agents:

- AEA4 (***M-series***) – ready to use olefin sulfonate, synthetic tenside,
- AEA5 – a concentrate based on synthetic tensides and tall oil derivatives (natural):
  - AEA5 fresh (***M-*** and ***O-series***) – AEA5 blended 1:9 with water shortly before mixing;
  - AEA5 pre-blended (***O-series***) – aged AEA5 up to 2 months after blending the concentrate of it with water 1:9.

Note: AEA4 and AEA5 of the same batch were used in the Foam Index study [6], and, therefore, the coding for the admixtures was kept unchanged for traceability.

Superplasticizer (SP) for both series – ether-based polycarboxylate from the same batch, solid content – 30±1.5%.

SP and AEA5 are from the same producer, and accordingly "compatible".

2.2 Mixes

Table 3 shows the ***M-series – "constant AEA dosage-variable workability"***, where three different mix compositions were dependent on the volume fraction of filler-modified paste (= matrix = all liquid, admixture, binder and mineral filler with particle size < 125 microns) and used air-entraining agent: 330 and 400 liters of matrix with AEA5 and 400 liters of matrix with AEA4.

Key requirements to ***M-series*** mixes:

- w/b – 0,46 (400L matrix), 0,57-0,63 (330L matrix)
- FA/(FA+C) – 0,30
- Limetone – appr. 24kg/m^3
- Slump cone (Mortar cone) - 100±10mm (only for 400L matrix mixes)
- Constant dosage of AEA.

Table 4 shows the ***O-series – "constant workability – variable AEA dosage",*** where two different binder types (see Table 4) were investigated and emphasis was put on controlling workability (100 +/-10mm) and total air content, which is more related to practice.

In both series, the idea was to find the most reliable dosage sequence of AEA and SP in terms of air void system and reproducibility.

Key requirements to ***O-series*** mixes:

- w/b – 0,40 (400L matrix)
- FA/(FA+C) – 0,35
- Slump cone (Mortar cone) - 100±10mm

- Air content – 6-8% (for d_{max} 8mm), corresponding to about 4-6% for concrete with d_{max} >16mm.

Table 3 – Mix design (corrected for measured density and fresh air) for ***M-series***

Mix	AEA	ID	Mass of constituent materials [kg/m^3]						
			Cement	Fly ash	0-8mm	Filler	Water	AEA	SP
330	–	0	289,3	124,0	1575,8	20,7	213,6		
	AEA 5	AEA	262,3	112,4	1647,6	18,7	194,2	2,6	
		AEA-SP	253,8	108,8	1617,7	18,1	193,3	2,6	0,7
		SP-AEA	251,7	107,9	1604,4	18,0	191,7	2,5	0,7
		AEA+SP	251,8	107,9	1605,2	18,0	191,8	2,5	0,7
400	–	0	341,1	146,2	1585,1	25,6	199,4		
	AEA 5	AEA	339,6	145,6	1564,6	25,5	195,3	3,4	
		AEA-SP	347,4	148,9	1594,1	26,1	197,8	3,5	2,2
		SP-AEA	335,1	143,6	1537,7	25,2	190,8	3,3	2,1
		AEA+SP	321,6	137,8	1476,0	24,2	183,1	3,2	2,1
	AEA 4	AEA	337,6	144,7	1555,3	25,4	194,1	3,4	0,0
		AEA-SP	344,4	147,6	1580,7	25,9	196,1	3,4	2,2
		SP-AEA	328,4	140,8	1507,2	24,7	187,0	3,3	2,1
		AEA+SP	302,1	129,5	1386,6	22,7	172,0	3,0	1,9

*Table 4 – Mix design (corrected for measured density and fresh air) for **O-series***

ID	Mass of constituent materials [kg/m^3]									SP/b,%	AEA/b,%
	CEM I[1]	CEM II[2]	SF	FA	Sand 0-8	Free water	Abs. water	SP	AEA		
AEA-SP		363,9	14,6	108,6	1560,2	194,9	4,6	2,9	3,7	0,60	0,76
SP-AEA		363,7	14,6	108,6	1560,2	194,8	4,6	3,8	3,9	0,77	0,79
AEA-SP		364,0	14,6	108,6	1560,2	194,9	4,6	2,4	9,7	0,50	2,00
SP-AEA		363,7	14,6	108,6	1560,2	194,8	4,6	3,8	3,4	0,77	0,70
SP-AEA		363,7	14,6	108,6	1560,2	194,8	4,6	3,8	3,7	0,77	0,76
SP-AEA		363,8	14,6	108,6	1560,2	194,8	4,6	3,4	3,9	0,69	0,80
SP-AEA	485,5		15,0	0	1560,2	200,2	4,6	3,5	4,1	0,71	0,82
SP-AEA	485,6		15,0	0	1572,4	200,2	4,6	3,5	4,2	0,71	0,84
SP-AEA	485,6		15,0	0	1572,4	200,2	4,6	3,5	4,2	0,71	0,84
SP-AEA	485,5		15,0	0	1560,2	200,2	4,6	3,5	3,9	0,70	0,78
SP-AEA	485,5		15,0	0	1560,2	200,2	4,6	3,5	3,5	0,70	0,70
SP-AEA	485,5		15,0	0	1560,2	200,2	4,6	3,5	4,5	0,70	0,91
SP-AEA	485,2		15,0	0	1560,2	200,1	4,6	4,5	3,7	0,91	0,73
SP-AEA	485,4		15,0	0	1560,2	200,1	4,6	3,9	4,9	0,78	0,99
SP-AEA		364,2	14,6	108,7	1545,5	195,0	4,6	3,4	4,8	0,70	0,99
SP-AEA		363,7	14,6	108,5	1560,2	194,8	4,6	3,9	5,4	0,80	1,11
SP+AEA		375,2	15,0	112,0	1545,5	201,0	4,6	2,5	8,0	0,49	1,60
SP+AEA		375,2	15,0	112,0	1545,5	201,0	4,6	2,5	8,0	0,49	1,60
SP+AEA		375,1	15,0	112,0	1545,5	200,9	4,6	3,1	3,5	0,62	0,70
SP-AEA		375,2	15,0	112,0	1545,5	201,0	4,6	2,5	3,9	0,49	0,78
AEA-SP		375,2	15,0	112,0	1545,5	201,0	4,6	2,5	10,1	0,49	2,01
AEA-SP		375,2	15,0	112,0	1545,5	201,0	4,6	2,5	10,1	0,49	2,01
AEA-SP		375,1	15,0	112,0	1545,5	200,9	4,6	3,1	7,6	0,62	1,52
AEA-SP		375,0	15,0	111,9	1545,5	200,5	4,6	3,5	4,5	0,70	0,90
AEA-SP		375,1	15,0	112,0	1545,5	200,9	4,6	3,1	6,0	0,62	1,19
SP-AEA		376,0	15,0	110,3	1560,2	200,5	4,6	3,9	3,5	0,78	0,70
SP+AEA	495,2		15,4	0	1560,2	204,1	4,6	3,6	3,8	0,70	0,74
AEA-SP	494,8		15,4	0	1560,2	204,1	4,6	3,8	3,9	0,74	0,76
AEA-SP	495,2		15,4	0	1560,2	204,1	4,6	3,6	3,9	0,70	0,76
AEA-SP		363,8	14,6	108,6	1560,2	194,8	4,6	2,4	9,7	0,50	2,00
SP+AEA		364,0	14,6	108,6	1560,2	194,8	4,6	2,4	7,8	0,50	1,60
SP		363,7	14,6	108,6	1560,2	194,8	4,6	3,9	0	0,80	0
SP		363,7	14,6	108,6	1560,2	194,8	4,6	3,9	0	0,80	0

[1] Norcem Anlegg cement; [2] Norcem Anlegg FA cement

Note: the shaded cells highlight mixes with "AEA5 fresh", while for unshaded cells "AEA5 pre-blended" according to the notation for admixtures given in 2.1.

3. METHODS

Table 5 presents admixture combinations and mixing sequences chosen based on the experience with Foam Index (FI) testing [6]. The FI testing is done in the following order: (1) add AEA with precision pipettes into a container with pre-shaken (10 Hz, 60 seconds) mix of binder and water (w/b 2,5), (2) close the lid and shake the container for 15 seconds (10 Hz), (3) remove the lid and observe the foam for 45 seconds, recording the time of stable foam. The procedure is described in detail in [6].

Table 6 gives an overview of used methods and equipment during the testing. The prolonged mixing time of at least 2 minutes after the addition of AEA was chosen to assure full activation of surfactant [19] and reduced variability caused by fly ash [20]. We changed the mixing equipment from Hobart to Sandby mixer because of the unavailability of the first equipment and a need to increase the batch size for additional tests. It should be mentioned that despite similar mixture proportions for ***M-*** and ***O-series***, changing the mixer type could affect the performance of the admixtures. This could largely affect the size of the air bubbles [2], hence the stability of air content and the air-void structure.

Tables 7 and 8 give a summary of fresh concrete properties for ***M-*** and ***O-series***, respectively. Complete tables for fresh concrete properties for each mix in ***M-*** and ***O-series*** are in Tables A and B respectively, see Attachment.

Table 5 – Admixture combinations and mixing sequences

Series	Admixture	Mixing sequence
M-series	0	1 min dry materials, 3 min water
	AEA	1 min dry materials, 3 min water+AEA
	AEA - SP	1 min dry materials, 2 min water+AEA, 1 min SP
	SP - AEA	1 min dry materials, 1 min ½ water+SP, 2 min ½ water+AEA
	SP + AEA	1 min dry materials, 3 min water+AEA+SP
O-series	SP	1 min dry materials, 1 min water, 5 min SP, 2 min rest, 1 min mixing
	AEA - SP	1 min dry materials, 1 min water, 3 min AEA, 2 min SP, 2 min rest, 1 min mixing
	SP - AEA	1 min dry materials, 1 min water, 2 min SP, 3 min AEA, 2 min rest, 1 min mixing
	SP + AEA	1 min dry materials, 1 min water, 5 min SP and AEA, 2 min rest, 1 min mixing

Table 6 – Equipment and test methods

	Mixing		Properties of concrete		
Series	Batch size [l]	Equipment	Air content, air-void system, fresh state	Workability	Porosity, air-void system, hardened state
M-series	4	5L Hobart mortar mixer	Density method[1], Pressure method[2]	Mortar slump cone[3] 120x80x40mm^3	Image analysis, PF-method
O-series	5 or 6	10L Sandby SU10 Paddle mixer	Density method, Pressure method		

[1] According to ASTM C138/C138M - 17a by comparing unit weight with theoretical density

[2] Pressure device for mortars (1L) was used

[3] Same procedure as for the standard slump test EN 12350-2, but the mini-cone is filled in 2 layers, each is tamped with 25 strokes [7,18].

The Image analysis on hardened specimens 160 x 40 x 40 mm^3 was performed in accordance with [21] and ASTM C457 on two well-hardened specimens for each series. The specimens were cut normal to a casting surface, ground using SiC grinding papers of 320, 500, 1200 grit to a light-reflective surface and the air-voids with sharp edges. Then the ground surface was painted black with a marker Edding 850 3 times, and the air voids were filled with the BaSO4 powder (particles 1-4µm) by finger-tapping and pressing. The excessive powder was firstly dragged off by a straightedged plastic ruler, and secondly by a slightly moist finger. Further, cracks and blemishes on the aggregates that got filled by the barium sulfate powder and, therefore, could cause erroneous air void characteristics, were painted black under the microscope. Prepared samples were placed on transparent foil, scanned by Epson Perfection V600 Photo at 2400ppi and analyzed using the Matlab script, developed by Fonseca [21].

The consistency, air content and density measurements for both series were performed between 10 and 15 minutes after water was added to the mix.

'Table 7 – Properties of fresh concrete. ***M-series***

Matrix volume [l]	Paste volume [l]	w/b	Type of AEA[1]	AEA, [% (c+FA)]	SP, [% (c+FA)]	Slump [mm]
330	359	0.57	-	0	0	30
330	319 - 326	0,60 – 0,63	AEA5	0,7	0 – 0,20	20 - 60
400	371	0,46	-	0	0	20
400	346 - 373	0,46	AEA5	0,7	0 – 0,45	30 - 100
400	325[2] - 370	0,46	AEA4	0,7	0 – 0,45	25 - 105

[1] See 2.1.

[2] 325L for SP+AEA, while other admixture combinations ranged from 353L to 370L.

Table 8 – Properties of fresh concrete. ***O-series***

Matrix volume [l]	Paste volume [l]	w/b (w/c)	Mixing volume, L	Type of AEA	AEA, [% b]	SP, [% b]	Slump [mm]
CEM I							
400	363	0.40	5,0 – 6,1	AEA5 fresh	0,70 – 0,91	0,70 - 0,71	82 - 109
400	363-370	(0.41)		AEA 5 pre-blended	0,73 – 0,99	0,70 – 0,91	85 - 110
CEM II							
400	370	0.40	5,0 – 6,1	AEA5 fresh	0,50 - 0,70	0,70 – 2,0	82 - 105
400	370-381	(0.62)		AEA 5 pre-blended	0,49 – 0,80	0,70 – 2,0	85 - 107

4. RESULTS & DISCUSSION

4.1 Fresh air void content

Due to many factors affecting air entrainment, we have in the following made an effort to look at air entrainment effect of (1) workability, (2) AEA dosage and (3) AEA-SP dosage sequence. The latter is the main point of this study and we have therefore paid special attention to this in terms of analyzing air-void parameters of hardened concrete as the effect of AEA-SP dosage.

Figure 1 shows similar relationships between density-based and pressure-meter-based air-void content for the ***O***- and ***M-series***, though better correlated for the ***M-series***. This is presumably due to that in the ***O-series*** both dosage and sequence varied (in the ***M-series*** dosage was constant while the sequence of addition varied), accompanied by variations in batch size and use of less efficient mixing equipment for ***O-series***.

Figure 1 does not show a 1:1 relationship and some apparent negative values are displayed for the density method, presumably due to several factors. One could be that the constituent materials do not exhibit the same particle densities in the fresh mix as in the particle density measurements. Another reason is undoubtedly the very different principles with Boyle-Mariotte's law behind the pressure meter and different effect on air voids of different sizes due to their different compressibility. The smaller the void the larger the pressure needed to compress, but the more likely to dissolve the air void into the water. The two measurements were made on the same fresh concrete sample but the pressure meter measurement could, of course, have a systematic error for various reasons (equipment, calibration, operator dependent, etc). Still, from the ***M-series,*** it appears that the density method for a given set of part-material data and rather a simple lab equipment (container, balance) is capable of giving a very good correlation to the standard pressure meter.

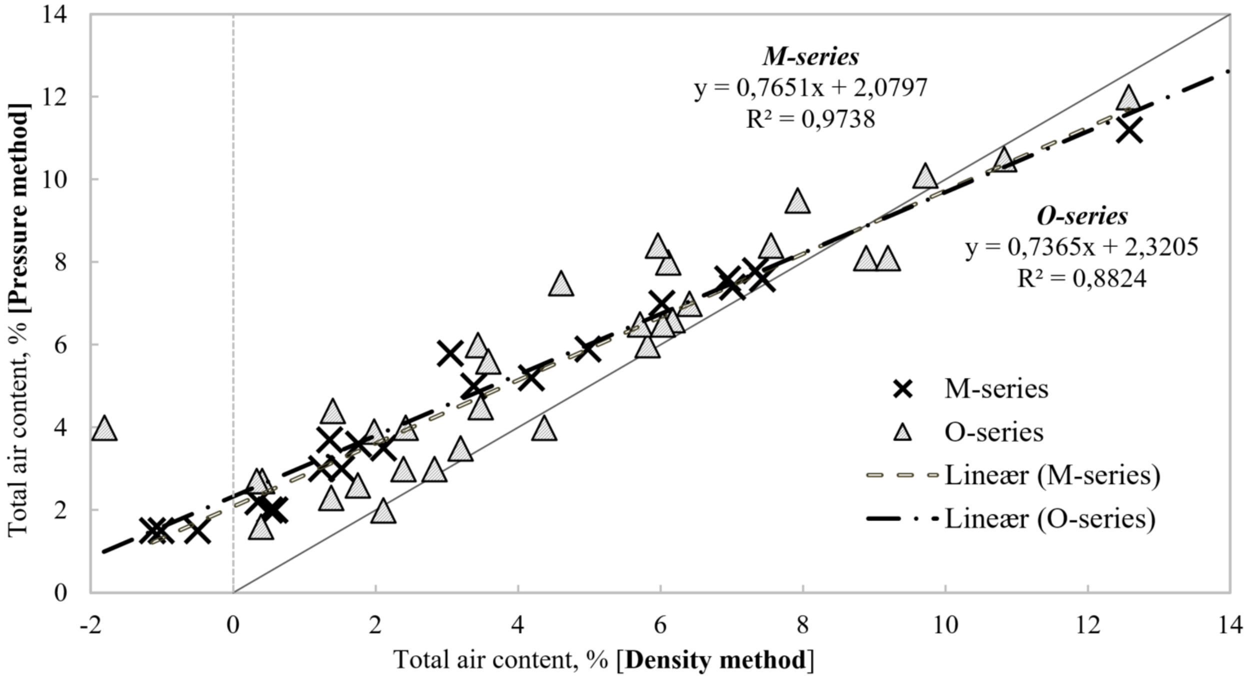

*Figure 1 – Correlation between density and pressure method for obtaining a value of the total air content in fresh concrete for **M-series** and **O-series.***

4.2 Effect of AEA-SP sequence on fresh air void content

Figure 2 shows the effect of AEA-SP dosage sequence on air entrainment in the ***M-series*** compared to reference mixes without any admixture and with only AEA. From the bar graph, it is clearly seen that the same dosage of AEA results in widely different air entrainment in fresh concrete depending on the sequence of dosage. Of the 3 sequences with both AEA and SP we see that 4-AEA+SP simultaneously always gives the highest air void content, 2- AEA before SP always give lowest, and 3-SP before AEA gives an intermediate fresh air void content. The references without admixture and the references with only AEA give low air content within each group of matrix volumes. Also, note that within each group of matrix volume the slump was almost constant: 60, 90-100 and 90-105 mm for 330L AEA5, 400L AEA5 and 400L AEA4, respectively.

The workability also affected air entrainment, as seen by comparing with the reference mixes without AEA: #0 without any admixture (20 – 40 mm slump) and #1 with only AEA (20-30 mm slump). Possibly, there is some sort of reciprocal effect between air content and slump. For all the mixes, comparing a sequence #1 without SP to other with SP, we could observe a general increment in values of the total air content with increased workability, except for matrix 400L and sequence #2 – AEA-SP (SP added after AEA).

<u>*Note*</u>: In Figure 2, FI (Foam Index) means a dosage of AEA in ml per gram of binder needed to obtain a stable foam for 45 seconds after shaking a water/binder suspension with AEA in a closed container. The framed text above the columns means that a stable foam was not obtained for the mixes. SP=2 means that after adding AEA and obtaining a foam it took 2 droplets of 20 µl SP each to kill the foam. Time in seconds is a lifetime of the foam on the surface.

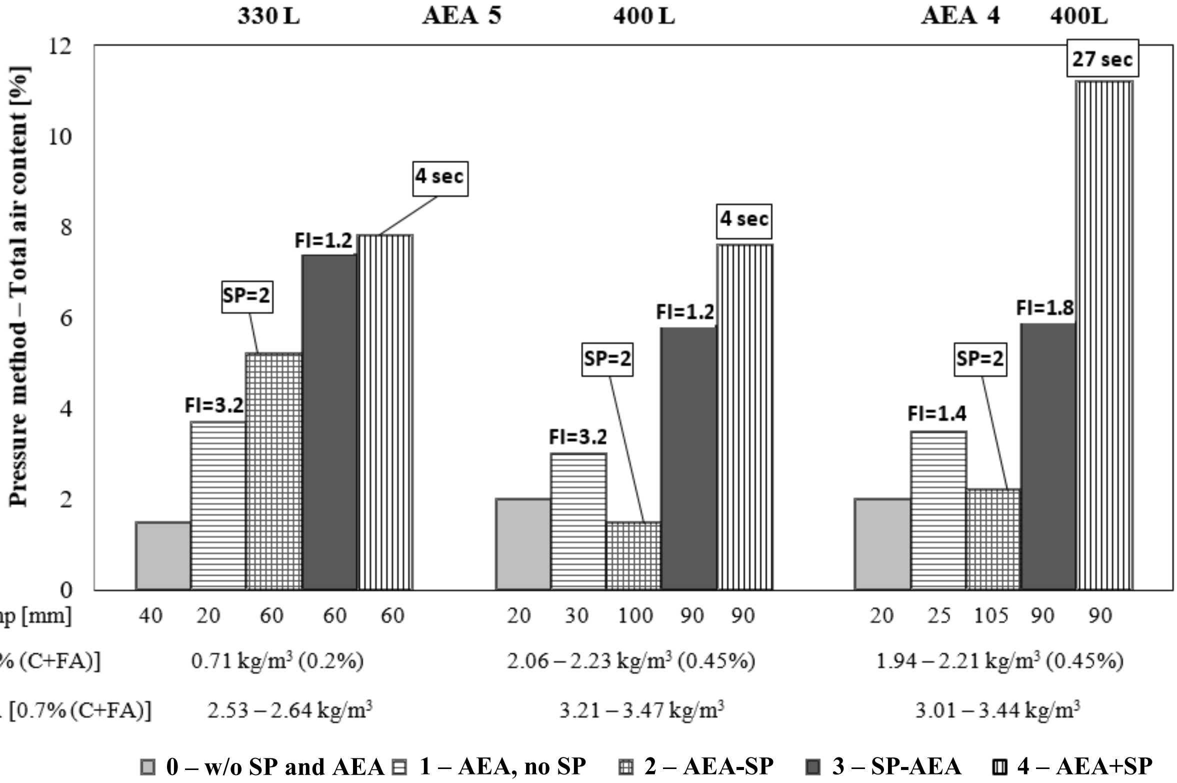

Figure 2 – Effect of AEA-SP dosage sequence on air entrainment. M-series: Comparison of pressure meter results with FI measurements, Jacobsen et al. [6] for different matrix volumes and AEAs.

Increase in matrix volume (or content of fines) for AEA5 generally led to a reduction of the total volume of air bubbles, and the drop is almost threefold for series #2 – AEA - SP. It means that the addition of SP after AEA to obtain flowable consistency in a rather refined system (400L of matrix contra 330L) causes coalescence and loss of stability for air bubbles (see discussion for Figure 3), and, hence, unwanted air detrainment [2, 9]. It is worth mentioning that for 330L matrix, the addition of SP (leading to 60mm in slump) led to an increase in air content for all the series with SP, meaning that it may be either an increase in workability to 100mm or refinement of the system that caused air detrainment for the sequence #2 for 400L matrix.

When other parameters are kept constant, the highest amount of air voids is guaranteed by adding AEA and SP simultaneously (series #4 – AEA+SP), Eickschen [10] and Puthipad [9] also mention this effect. When added together with SP, the pure synthetic surfactant AEA4 shows much higher air entrainment compared to the mixture of natural and synthetic tensides of AEA5, while the difference is small for other dosage sequences.

The results of the foam index measurements on slurries do not fully reflect the properties of the mixes, because this indicative test does not predict the development of the air-void system from the fresh to the hardened state (see corresponding Figure 3 displaying results of air void analysis of hardened concrete). Furthermore, the very high air content for series 4 – AEA+SP does not correspond to the "foam-killing" effect (instability of air) observed in [6].

Steinhoff [11] also confirmed difficulties in the application of FI test to verification of mutual performance of AEA and SP, and there was no more good correlation between the BET surface area of FA in concrete and FI, as other authors report [2, 16, 20, 22].

4.3 Effect of AEA-SP sequence on hardened air void content

Figure 3 shows the air entrainment in hardened concrete for two of the three matrix-volume series in Figure 2. Figure 3 confirms a clear effect of dosage sequence also in hardened concrete, especially for 6 mixes with 100 ±10 mm slump at constant SP and AEA dosages. The tendency from left to right for the total air content is the same as for the fresh concrete measurements (Figure 2).

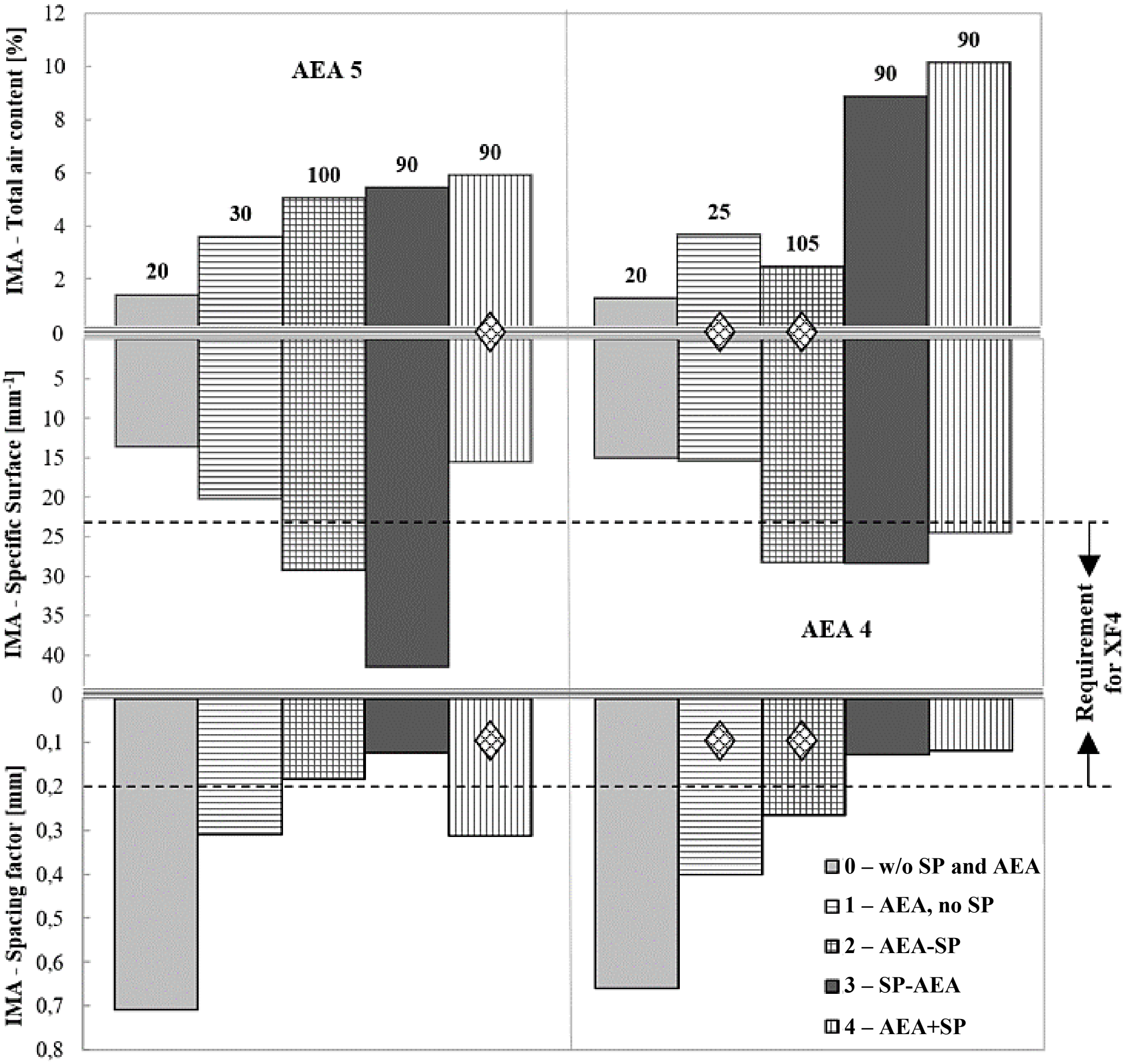

Figure 3 – M-series: Air-void analysis for mixes in the 400L series with the two AEAs (the numbers over the bars for total air content show values for workability on mortar slump cone. Shaded rhombuses – done by a different operator).

Note: Retention of air content in the fresh state over time (up to 60 min) was reported and summarized by Pedersen [3]. Only three long-chain hydrocarbon-based and epoxy sulfate AEAs showed an increase up to 40% after simulation of transportation for 45 min. In the case of the series #2 AEA – SP and AEA5, there is more than a double increase in total air content from fresh (Figure 2) to hardened state. This we could either assign to erroneous surface preparation for the air-void analysis or a time-dependent increase in the fresh state without re-agitation due to the coalescence of small voids with higher pressure inside into larger voids with lower pressure. This follows from Laplace giving the pressure difference over the air-liquid interface is $\Delta p = - 2\sigma/r$ with σ = surface tension water-air, r =void radius.

The effect of AEA in a stiff concrete is minor (sequence #1 – AEA, no SP) due to resistance against bubble growth from the stiff paste and a limited amount of sites for the AEA to adsorb on, because of the absence of a rather efficient dispersive agent. All mixes with SP give a better air void system than in stiff mixes.

We could assign the variation in air content for sequence #2 – AEA-SP to a difference in the influence of de-foaming agent within SP on the efficacy of AEA. From a limited number of mixes, the influence is stronger for synthetic AEA4, and it may be due to some compatibility of AEA5 and SP (the same producer).

Also, despite low air content (especially in a fresh state, Figure 2) for #2, the air-void specific surface remains high for both AE agents, hence, the reduction of air content for AEA4 can be associated with instability of the coarse air voids over 300 μm when SP is added after AEA. It also implies that having requirements for total air content solely is not the right approach, even though some country standards and organizations have it so [14].

The bars in Figure 3 for a sequence #3 – SP-AEA show that adding AEA in a flowable concrete led to an increase in coarse air voids for synthetic AEA and gave a drastic increase, compared with other sequences, in fine air bubbles (smaller than 300μm) for a semi-synthetic AEA5.

Eickschen [10] described the interaction between AEA and SP when SP comes first – the air void system is formed in a softer concrete, which results in a coarser system, explaining it by competitive adsorption of admixtures on cement particles, resulting in unstable air content. It does not seem to be valid for FA concretes. It was only Pathipad [8] who suggested adding SP before AEA to obtain the most refined air void system, even though he reported suitability of that mixing procedure only for OPC concretes.

The computed spacing factor values from the measured specific surface using Fonseca's method [21], see the solid dark-grey bars in Figure 3, stay well within the required limits (listed in Norwegian national Annex to EN 206 for the most severe frost exposure class XF4). This implies that adding SP first in FA concrete can likely improve the air-void system, comparing to adding SP after AEA (i.e. sequence #2).

As for sequence #4, with simultaneously added AEA and SP, the air void parameters for the concretes primarily depend on the efficacy of the AEA to compete for the sorption sites.
Reproducibility should be taken into consideration because according to the literature [12, 9] the combination #4 is least predictable. Speaking of all the series, mixes with AEA5 were reproduced at least two times, whereas with AEA4 – they were produced only once.

4.4 Effect of AEA-SP sequence vs effect of AEA-dosage

Figure 4 shows AEA dosage vs fresh air void content for all mixes of this study:
O-series: varying AEA-dosage with two different types of AEA: **1**: shaded legends – AEA5 pre-blended- natural (tall oil derivatives)-synthetic mix, diluted 1:9 and stored in lab up to 2 months before use. **2**: open legends – AEA5 fresh - natural-synthetic mix diluted 1:9 and used freshly blended, i.e within 1 hour.

M-series: the vertical bar at constant (0,7 %) AEA dosage (indicating the range for AEA4 Synthetic olefin sulfonate and AEA5 fresh natural-synthetic mix (same as ***O-series***), diluted 1:9 shortly prior to mixing.

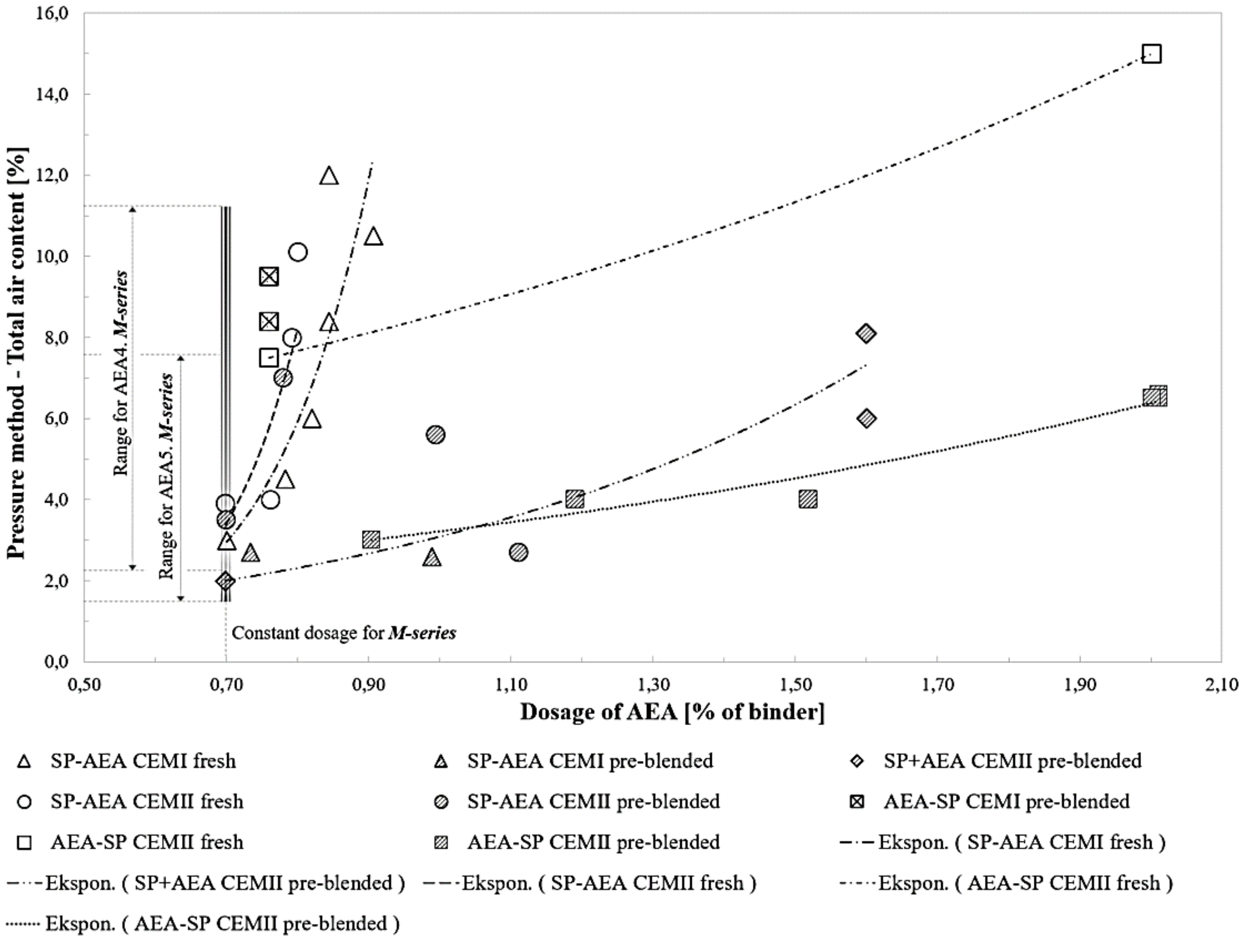

Figure 4 – Influence of AEA-SP dosage sequence on binder type, AEA type and dosage, and total air content.

The target of 6-8% air content in ***O-series*** at ≈ constant workability (see more on workability further below) caused the dosage of AEA for successful mixes to vary from 0,76 to 2,0 % of binder.

In Figure 4, we see that for the pre-blended AEA and the sequence AEA-SP (the hatched square legends), considering low variability of the curve for FA concrete, the dosage of AEA needed to reach circa 9% (the crossed square legend – OPC reference) of air should be quadrupled when FA is present. That agrees well with Zhang [23], who found that AEA dosage for obtaining air content as in OPC concrete should be 2-6 times higher for FA concrete. Nonetheless, for fresh AEA and the sequence SP-AEA (open triangle and open circle legends), we could read off the increased demand in AEA only up to 15% at most for the mix with 35% FA/b.

Zhang [23] also found that batch-to-batch variability of air content in fresh FA was lower than for OPC concrete. However, when comparing SP-AEA sequence for CEMI and CEMII for fresh AEA, variability is lower for CEMI.

There is, in fact, a large number of variables in addition to the three types of AEA: variable AEA dosage, variable dosage sequence, variable quality and volume fraction of filler-modified cement paste, variable binder type (CEMI – OPC and CEMII Fly Ash+OPC) and variable workability. However, compared to all these variables Figure 4 shows that the dosage sequence has a very large effect on air void content, particularly when considering that workability for most of these mixes was constant (100±10mm): By comparing AEA5 fresh for the ***M-*** and ***O-series*** we see that the magnitude of the dosage sequence effect (M-series, constant dosage, variable sequence) is approximately half (1.8 – 7.6 % air) of the magnitude of the variable dosage series (***O-series***, variable dosage, variable sequence) which varies 2 – 15 % air.

When looking at all three AEA types (AEA5 fresh, AEA5 pre-blend, AEA4) Figure 4 shows that the variation in air-void content is similar in the ***M-*** and ***O-series***. Hence, the dosage sequence has a very large effect on air entrainment. Within the ***O-series*** there is, however, a clear effect of the type of AEA since pre-blended (aged) AEA5 results in lower air entrainment compared to freshly blended AEA5. This is in accordance with Dodson [24] and Spörel [20], who noticed that the properties of AEAs change with age (especially synthetic) when conducting Foam index tests and who advised using solutions (AEAs) that are just a few days old. In connection to this, it is worth mentioning that admixture producers for economical reasons sell concentrated air-entraining agents to concrete producers to be diluted at the facility, but it has not been reported how the reduced performance of AEA with ageing is compensated and controlled.

Within the ***M-series,*** the type of AEA seems to have less effect than within the ***O-series*** except for the simultaneous addition AEA4-SP which gives much higher air void content than simultaneous AEA5-SP.

Looking at the hatched triangles (SP-AEA CEMI pre-blended) and circles (SP-AEA CEMII pre-blended) in Figure 4 one can see the series with the highest scatter. In fact, the hatched circles with dosage of AEA 0,7% (very first successful mix, i.e AEA was aged from a few hours to a few days) and 0,8% of binder represent concretes cast about 3 weeks apart, and the results fit well the exponential curve for the same order of addition of the admixtures with the fresh AEA (blank circles). Two other hatched circles and the hatched triangles were cast the same day, but 6 weeks later than the two abovementioned mixes. Here it is clear that the performance of the AEA had become unpredictable.

From the example above, it could be that within a certain period of time the pre-blended AEA performs as well as freshly blended. But if we look at the hatched and blank squares for AEA-SP sequence with pre-blended and fresh AEA5 in FA concrete respectively, there is a drop of the effectiveness of the AEA of about 2,5 times, and it is despite the fact that the AEA solution was also aged in about 3 weeks, as in the previous example.

We assume that the reason for the different behavior of the mixes is that adsorption susceptibility of the active ingredient in AEA to the carbon in FA is higher when the AEA is added in stiff concrete, i.e before SP. It may be that with ageing AEA loses the active ingredient easier to the carbon in FA, comparing to the freshly mixed AEA. This conclusion can presumably be valid for simultaneous addition of AEA and SP (the hatched rhombuses in Figure 4, age of AEA – ca 3 weeks).

4.5 Fresh air void content, AEA and workability

Figure 5 shows slump vs total air content with shaded legends for the ***M-series*** and open legends and "X" for the ***O-series***. Again, we see the somewhat higher variation in air content for the ***O-series*** (variable AEA dosage) than for the ***M-series*** (constant AEA dosage – variable sequence).

Exponential trendlines (added to facilitate readability of the figure) for the same dosage sequences are drawn in one style, and we can see that the effects of dosage sequences in the ***O-*** and ***M-series*** can be somewhat related despite different mixing equipment, quality of AEA (fresh or pre-blended), batch size used and, even, constituents (limestone filler in ***M-series*** was replaced with 4% Si/b in ***O-series***).

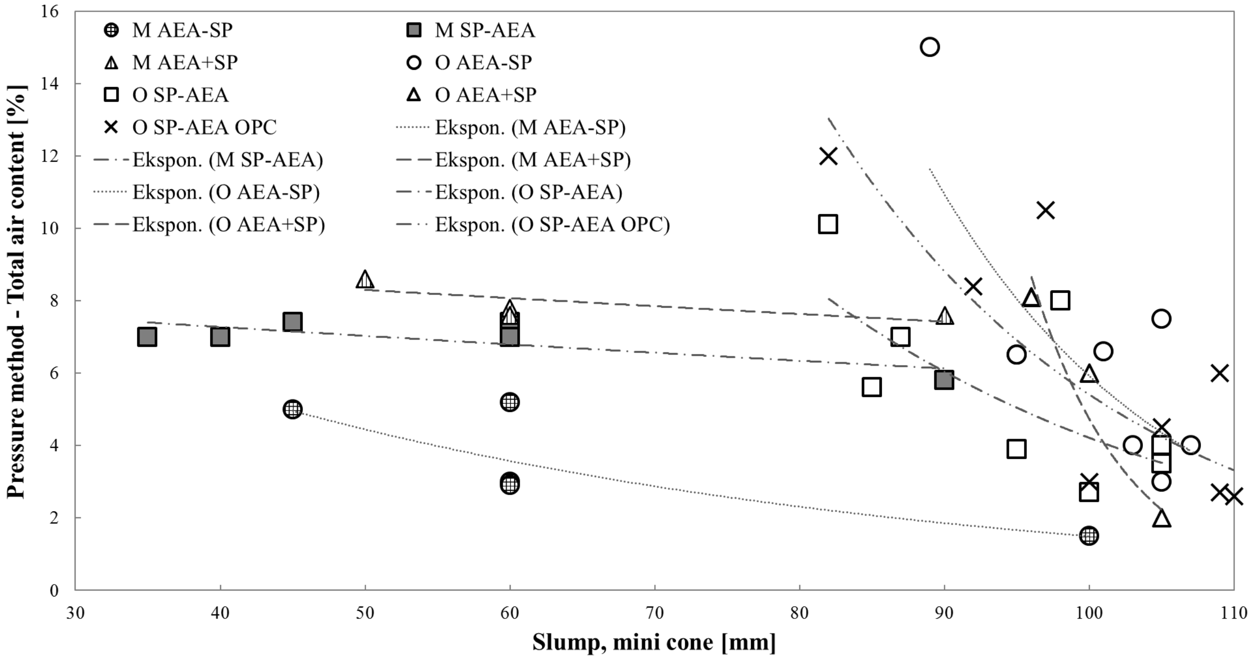

Figure 5 – Influence of AEA-SP dosage sequence on workability and total air content. AEA5

As mentioned, there could be a reciprocal effect between air voids and workability. The too low viscosity of the paste could allow air voids to rise and disappear whereas increased paste volume due to air voids increases slump.

Dodson [24] noted that air content increases by an increasing slump from 75 to 150 mm, but above 150 mm the air content drops because of the reduced viscosity in the paste insufficient to withstand buoyance forces by large air bubbles. This is in line with the "reciprocal effects" including the right-hand side of Figure 5 where there is a tendency of low air void content at high slump values, i.e. air voids escaping more easily in a fluid mix. Sequence AEA-SP for both ***M-*** and ***O-series*** gave the highest decrement in air content with an increased slump, which could indicate susceptibility to air detrainment when SP enters the system after AEA.

On top of this, there is the effect of surfactants on increasing the yield stress and hence the slump which counteracts the effect of the air voids [25]. The effect of air entrainment on workability, therefore, is hard to predict.

5. CONCLUSIONS

- Traditional thinking of admixture dosage sequence in OPC concretes cannot be applied for FA concretes. The most favorable admixture combination for air entrainment in FA concrete seems to be when SP is added before AEA.
- The results from Foam Index measurements [6] do not fully reflect the properties of fresh and, especially, hardened concrete. Hence the Foam Index test seems unsuitable for a combination of admixtures.
- To fulfill requirements to effective air void spacing while maintaining as low air void content as possible within the requirements for exposure class XF4 seems possible. However, it requires specific procedures based on trial mixing including requirements to the sequence of AEA-SP dosage.
- Performance of a selected AEA plays an important role, which could possibly allow ignoring differences in AEA-SP dosage sequence when the needed dosage is found and reproducibility achieved during a pre-qualification phase.
- Performance of selected SP was not affected by AEA-SP dosage sequence as the amount of SP was kept constant for ***M-series***. It means that the selected polycarboxylate may not lose its active ingredient to the carbon in FA. Yet the polycarboxylate SP, when added first, seems to shield AEA from being adsorbed and becoming less efficient.
- The workability affects air entrainment with some sort of reciprocal effect between air content and slump. However, we cannot confirm that the results described for OPC on Abrams slump cone [2, 24, 12] fit the obtained relationship for fly ash concrete on mortar-cone.

6. FUTURE WORKS

Comparison of air void structures in hardened concrete for varying only matrix volume and dosage sequence of the admixtures could be of use to pick out the most favorable combination for fly ash concrete produced in laboratory conditions.

Studies on air void stability for different dosage sequences with AVA (Air Void Analyser), using a similar approach as Pathipad [9] and Spörel [20], will be an important supplement to the present paper.

In addition, more systematic research on the variability of the fresh AEA demand and the total air content for concrete with and without fly ash and various AEA-SP orders of addition is required.

We cannot draw a conclusion about the effect of the AEA-SP order of addition on the demand for AEA (see Figure 4) because of a lack of data and unconfirmed effectiveness of pre-blended AEA for different mixes. Therefore, we think that more systematic research on the variability of the fresh AEA demand and the total air content for fly ash concrete (and without fly ash) and various AEA-SP orders of addition is required.

Further investigations with a rather systematic approach aiming at obtaining a reproducible air-entrained fly ash concrete with d_{max} increased to at least 16mm is required.

6. EXAMPLE OF APPLICATION

Based on the positive response (in terms of air entrainment) of FA-mix on the addition of SP first and subsequent addition of AEA, the main concrete mixes (d_{max} 16mm) for the Ph.D. project "Production and documentation for frost durable concrete" were successfully produced.

About 60%-80% of SP was added together with water to obtain consistency of about 170-180 mm for standard Abrams slump cone. AEA5 (ready to use diluted by the producer) was added 1 minute after SP (ether-based polycarboxylate, solid content – 23±1.5%) followed by 2 min rest, then dosing more SP to obtain slump of 200 ± 10mm and remixing for about 1 minute. Concrete volume – 57 liters. 35% FA/b, 4% SF/b.

Table 9 – The practical application of research results in the Ph.D. project

Concrete mix	Dosage of admixtures per mass of binder, [%]		Slump, [mm]	Density method		AVA measurements						Air content in hardened concrete, PF-test, [%]
						Sampling after 20-25 min			Sampling after 60-70 min			
	AEA	SP		Fresh density, $[kg/m^3]$	Air content, [%]	Micro air[1]	Spacing factor, [mm][2]	Specific surface, $[mm^{-1}]$[2]	Micro air [1]	Spacing factor, [mm]	Specific surface, $[mm^{-1}]$	
0,40w/b, 35%FA	0,43	0,85	200	2319	5,6	2,4	0,24	25,3	2,2	0,25	24,2	5,0
0,45w/b, 35%FA	0,49	0,72	200	2299	5,8	2,1	0,24	26,7	2,6	0,23	25,4	5,9
0,293w/b, 35%FA	0,60	1,46	220	2346	5,9	3,4	0,15	35,8	3,2	0,17	33,0	5,2
0,45w/b, 0%FA	0,20	0,80	190	2327	5,1	3,7	0,20	24,4	2,6	0,25	23,3	5,4

[1] Chord length <0,35mm

[2] Requirements for the air-void spacing factor – max 0,25mm, specific surface – min 24 mm^{-1}, micro air – 1,8%.

Table 9 shows that it is possible to produce a robust air-entrained concrete with a high volume of FA, workable and stable. It took just one 30L-trial mix for each FA-concrete to obtain a material of the required parameters. OPC concrete mix required three trial mixes and two additional 57l-mixes with the same mixing procedure, time-dependent AVA results show that the air-void system is not persistent. The last can only confirm Eickschen's theory [10] about the instability of air bubbles when AEA is added into soft (somewhat flowable) concrete.

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APPENDIX A. FRESH CONCRETE PROPERTIES

Table A – Properties of fresh concrete. Full range of the mixes. ***M-series***

Sequence	Paste volume [l]	w/b	Admixture dosage [% b]		Air content [%]		Slump cone [mm]	Fresh density, [kg/m3]
			AEA	SP	Pressure method	Density method		
AEA5. 330l matrix volume								
0 – No AEA, no SP	359	0,57	0	0	1,5	-1,1	30	2223
	359	0,57	0	0	1,5	-1,0	40	2221
1 – AEA	325	0,59	0,7	0	3,6	1,8	20	2227
	326	0,59	0,7	0	3,7	1,4	20	2238
2 – AEA-SP	321	0,62	0,7	0,2	5,2	4,2	60	2195
	324	0,60	0,7	0,2	5,0	3,4	45	2221
3 – SP-AEA	319	0,62	0,7	0,2	7,4	7,0	60	2177
	321	0,61	0,7	0,2	7,0	6,0	35	2199
4 – AEA+SP	319	0,62	0,7	0,2	7,8	7,3	60	2178
	318	0,61	0,7	0,2	7,6	7,4	60	2178
AEA5. 400l matrix volume								
0 – No AEA, no SP	371	0,46	0	0	2	0,5	20	2299
	371	0,46	0	0	2	0,6	20	2297
1 – AEA	367	0,46	0,7	0	3	1,2	25	2281
	366	0,46	0,7	0	3	1,5	30	2274
2 – AEA-SP	367	0,46	0,7	0,2	3	1,3	60	2279
	368	0,46	0,7	0,2	2,9	1,2	60	2281
	373	0,46	0,7	0,45	1,5	-0,5	100	2320
3 – SP-AEA	348	0,46	0,7	0,2	7	6,6	40	2157
	347	0,46	0,7	0,2	7,4	6,8	45	2152
	348	0,46	0,7	0,3	7	6,4	60	2161
	360	0,46	0,7	0,45	5,8	3,1	90	2238
4 – AEA+SP	341	0,46	0,7	0,2	8,6	8,4	50	2115
	340	0,46	0,7	0,2	8,6	8,5	50	2116
	346	0,46	0,7	0,45	7,6	6,9	90	2148
AEA4. 400l matrix volume								
1 – AEA	364	0,46	0,7	0	3,5	2,1	25	2261
2 – AEA-SP	370	0,46	0,7	0,45	2,2	0,3	105	2300
3 – SP-AEA	353	0,46	0,7	0,45	5,9	5,0	90	2193
4 – AEA+SP	325	0,46	0,7	0,45	11,2	12,6	90	2018

Table B – Properties of fresh concrete. Full range of the mixes. ***O-series***

Sequence	Mixing volume, [l]	Paste volume [l]	Admixture dosage [% b]		Air content [%]		Slump cone [mm]	Fresh density, [kg/m3]
			AEA	SP	Pressure method	Density method		
Cem I:								
SP-AEA	6,1	363	0,82	0,71	6,0	3,4	109	2302
SP-AEA	6,1	363	0,84	0,71	12,0	12,6	82	2086
SP-AEA	6,1	363	0,84	0,71	8,4	7,5	92	2205
SP-AEA	5	363	0,78	0,70	4,5	3,5	105	2301
SP-AEA	5	363	0,70	0,70	3,0	2,4	100	2327
SP-AEA	5	363	0,91	0,70	10,5	10,8	97	2126
SP-AEA	5	363	0,73	0,91	2,7	0,4	109	2374
SP-AEA	5	363	0,99	0,78	2,6	1,8	110	2343
SP+AEA	5	370	0,74	0,70		19,7	85	1913
AEA-SP	5	370	0,76	0,74	8,4	6,0	107	2239
AEA-SP	5	370	0,76	0,70	9,5	7,9	95	2192
Cem II:								
AEA-SP	6,1	370	0,76	0,60	7,5	4,6	105	2239
SP-AEA	5	370	0,79	0,77	8,0	6,1	98	2203
AEA-SP	6,1	370	2,00	0,50	15,0	17,8	89	1930
SP-AEA	5	370	0,70	0,77	3,9	2,0	95	2300
SP-AEA	5	370	0,76	0,77	4,0	2,4	105	2290
SP-AEA	5	370	0,80	0,69	10,1	9,7	82	2118
SP-AEA	6,1	370	0,99	0,70	5,6	3,6	85	2260
SP-AEA	6,1	370	1,11	0,80	2,7	0,3	100	2338
SP+AEA	5	381	1,60	0,49	8,1	8,9	96	2131
SP+AEA	5	381	1,60	0,49	6,0	5,8	100	2203
SP+AEA	5	381	0,70	0,62	2,0	2,1	105	2289
SP-AEA	5	381	0,78	0,49	7,0	6,4	87	2189
AEA-SP	5	381	2,01	0,49	6,6	6,2	101	2195
AEA-SP	5	381	2,01	0,49	6,5	5,7	95	2205
AEA-SP	5	381	1,52	0,62	4,0	4,4	107	2237
AEA-SP	5	381	0,90	0,70	3,0	2,8	105	2273
AEA-SP	5	381	1,19	0,62	4,0	-1,8	103	2381
SP-AEA	5	381	0,70	0,78	3,5	3,2	105	2266
AEA-SP	5	370	2,00	0,50	6,5	6,0	95	2205
SP+AEA	5	370	1,60	0,50	8,1	9,2	96	2131
Only SP	5	370	0	0,80	2,3	1,4	100	2314
Only SP	5	370	0	0,80	1,6	0,4	105	2337

Note: the shaded cells highlight mixes with "AEA5 fresh", while for unshaded cells "AEA5 pre-blended" according to the notation for admixtures given in 2.1.