Effect of AEA-SP dosage sequence on air content and air void structure in fresh and hardened fly ash mortar

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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 mortar. Image analysis shows a good correlation between total air content and the air void spacing factor, though with a somewhat lower specific surface when SP is added together with AEA. Foaming measurements on the same materials and dosage sequences were therefore found less useful for studying the effect of admixture combinations. More work, including AVA measurements, is in progress to learn how to combine AEA, SP and fly ash in concrete.

Key words: Admixtures, Fly ash, Air entrainment, Testing.

1. INTRODUCTION

1.1 General

Production of frost durable fly ash (FA) concrete with a stable and protective air void system has often 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 air entraining agent (AEA). 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. We think 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 cement-fly ash water slurries of various SP/AEA combinations and dosage sequences [1]. The foam study indicated that a combination with SP drastically affects the adsorption kinetics. The same materials from the foam study [1] were also used to study the effect of addition of the admixtures on air entrainment [2].

The sequence of the addition of SP and AEA in concrete has been debated among practitioners for a long time, but the authors know of no experimental studies of SP-AEA dosage sequence in fly ash concretes in the literature. For regular OPC concrete, some authors [4], with reference to [7], suggest adding AEA after blending SP in the mix to give a stable air-void spacing factor; others [3] say that SP should be added after AEA, providing time for AEA to precipitate. No *standards*, *committees,* or *guidelines* specify the AEA-SP interaction. *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 recommend that AEA is added either before or simultaneously with SP in the concrete mixes containing either preblended or separately added FA. The need for real knowledge about AEA and SP interaction in FA concrete is growing with the increased need for high volume fly ash concrete. *The scope of this work* was to investigate air void content and structure from laboratory mortar mixes where both the type of AEA and the dosage sequence of AEA- and a co-polymer SP were varied.

2 EXPERIMENTS

2.1 Mixing sequences and materials

Table 1 shows the 5 (five) admixture combinations and mixing sequences chosen based on experience with Foam Index testing:

Table 1 – Admixture combinations and mixing sequences.

Series ID	Admixture	Mixing sequence
	No admixture	1 min dry materials, 3 min water
1	Only AEA	1 min dry materials, 3 min water+AEA
2	AEA then SP	1 min dry materials, 2 min water+AEA, 1 min SP
3	SP then AEA	1 min dry materials, 1 min ½ water+SP, 2 min ½ water+AEA
4	SP with AEA	1 min dry materials, 3 min water+AEA+SP

2.2 litre mortar mixes were prepared in a Hobart table mixer. The volume fractions of filler-modified paste (all liquid, admixture, binder and mineral filler with particle size < 125 microns) were 330 and 400 litres/m^3 mortar, corresponding to normal and rich concrete mixes. The w/(c+FA) ratio = 0.46 for the 400 series and 0.57-0.63 for the 330 series [2]. FA/(c+FA) = 0.35 for all mixes. Two different anionic AEAs were used: AEA5 was based on synthetic tensides and tall oil derivatives, whereas AEA4 was an olefin sulfonate. The SP was an acrylic polymer. The cement used was a CEM I with a Blaine of 396 m^2/kg, additional low lime fly ash with 1.74 % carbon and a Blaine of 334 m^2/kg, and a limestone filler with a Blaine of 362 m^2/kg was used. All powders were supplied by Norcem-Heidelberg in Brevik, Norway. For details about admixtures and powders, see [1]. The aggregate was NS3099 standard Norwegian granitic 0-8 mm sand supplied by Norsk Stein Årdal.

2.2 Fresh and hardened mortar measurements

In the fresh mortar, the following measurements were made for each series: slump (40/80/120 slump cone on plexiglass plate) after 5 minutes, density after 8 minutes, pressure meter (1 L for mortars) after 9 minutes, and casting of 3 specimens 40 x 40 x 160 mm^3. All the mixes for AEA5 were reproduced at least twice, whilst mixes with AEA4 were made once.

In the hardened mortar, we measured the total air content from the demoulding density and using the PF-method, and air void parameters using image analysis (IMA) [6]. The samples were polished, inked, air voids filled with 2-4 μm $BaSO_4$ powder, scanned, and analysed after 90-120 days of storing in water.

3 RESULTS AND DISCUSSION

3.1 Fresh mortar measurements

Table 2 shows the 5 (five) different mortar mixes and their workability values.

Figure 1 shows the effect of AEA-SP dosage sequence on the total air content in the fresh mortar, when the dosage of AEA [% of binder] is held constant, and the corresponding Foam Index (FI) values from [1] with the same binders and admixtures. For the mixing sequences 2-4, the workability was kept constant at 60±10mm for the 330 series and 100±10mm for the 400 series respectively. FI measurements in squares above the bars show either the number of drops of SP to stop foaming or the time in seconds of stable foam (with a requirement at 45 sec).

Table 2 – Properties of the fresh mortar.

Mix	Paste volume, L [2]	w/b	Type of AEA*	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**–370	0.46	AEA4	0.7	0-0.45	25-105

* AEA4 – Olefin sulfonate, AEA 5 – based on synthetic tensides and tall oil derivatives.

** 325L for series 4 (see Table 1), while series 1–3 range from 353L to 370L.

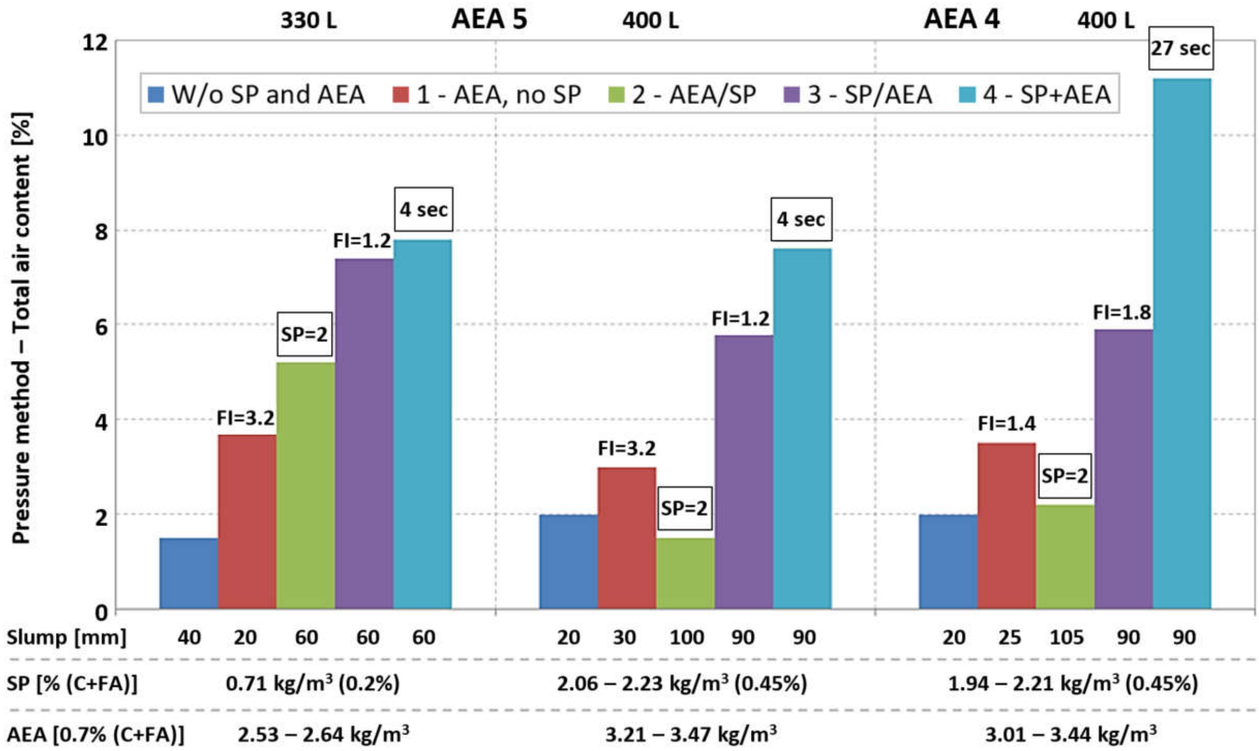

Figure 1 – Comparison of pressure meter results with FI measurements, Jacobsen et al. [1] for different matrix volumes and AEAs.

The results in Table 2 and Figure 1 show that both AEAs give big differences in air entrainment depending on the AEA-SP dosage sequence. The workability also affected air entrainment with some sort of reciprocal effect between air content and slump; most pronounced for AEA 5 when added first, see Figure1. The other series show negligible effect of workability. The highest amount of air voids is guaranteed by adding AEA and SP simultaneously (series 4), Eickschen E. and Müller C. [3] also mention this effect. When added together with SP, the pure synthetic surfactant AEA 4 shows a great contrast to the mixture of natural and synthetic components in AEA 5, but the difference is insignificant for other dosage sequences.

The results from foam index measurements do not fully reflect the properties of the mortar mixes, because this indicative test does not predict the development of the air-void system from the fresh to the hardened state. Furthermore, the very high air content when AEA and SP are added together does not correspond to the "foam killing" observed in [1]. Figure 2 shows that air content increased for series 2 (AEA before SP) meeting the requirements for the air void system, but in the fresh state (Figure 1) this combination was less promising.

3.2 Hardened mortar measurements

Mortar samples 160 x 40 x 40 mm were cast and split in two for hardened air-void analysis (IMA).

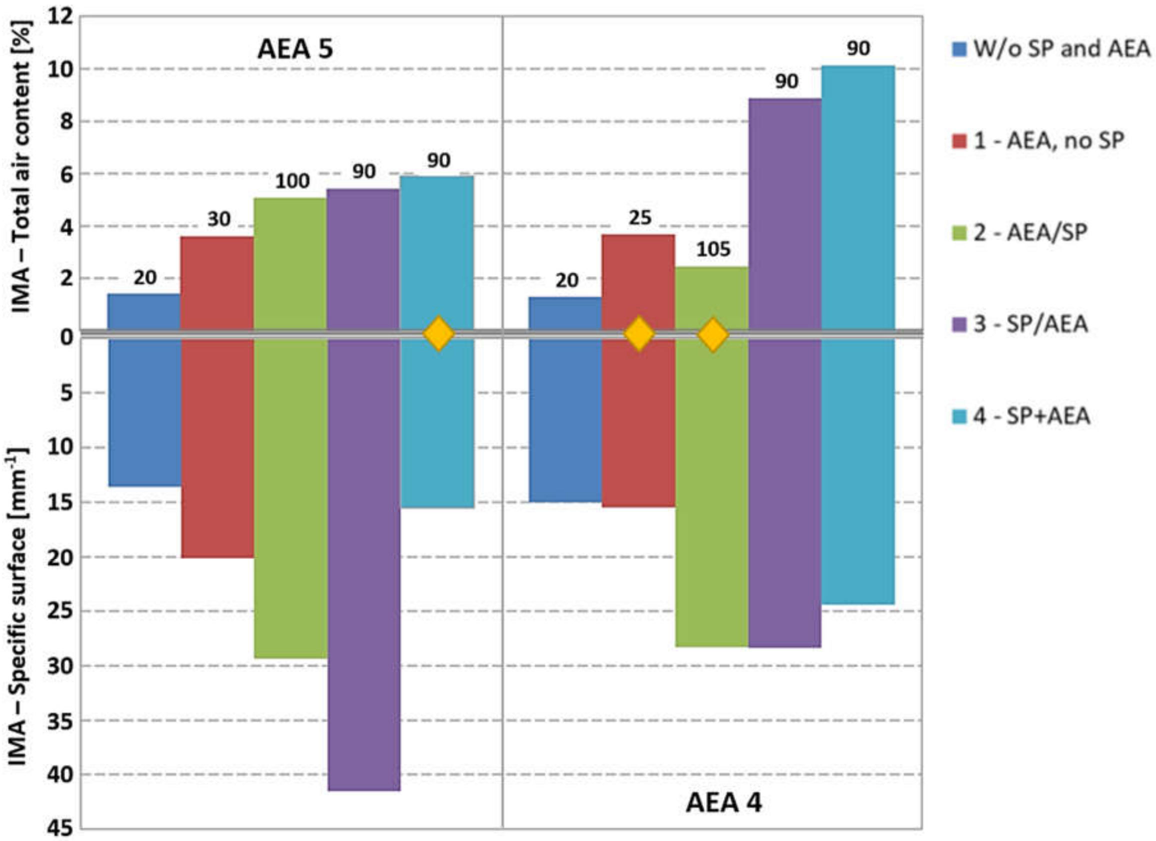

Figure 2 – IMA. Total air content and specific surface for mixes in the 400 L series with the two AEAs (the numbers over the bars show values for workability on 40/80/120 slump cone. Yellow rhombuses – done by a different operator)

Air void formation depends on several factors. Those that could possibly vary for mixtures with the two different AEAs are: the ratio of mixing time to activation time, and the susceptibility to coalescence and dissolution.

Further studies of fresh mortar with AVA are being carried out to study the impact of dosage sequence on air-void characteristics, including the ageing effect and the dosage for an acceptable air content and the resulting air void system.

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