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Air Entrainment in Fresh Concrete with PFA

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Abstract

The results of a study into the influence of PFA on air entrainment in fresh concrete are discussed It is shown that the required dosage of AEA to produce an air content of $5{\cdot}5 \pm 0{\cdot}5\%$ in a PFA modified concrete mix is two–six times that required in the corresponding neat OPC concrete mix. The dosage of a vinsol based air entraining agent (AEA) required appears to be directly related to the PFA content of the mix. Similar direct relationships were obtained with a range of different PFAs. The dosage of an AEA based on the salt of a fatty acid appears to be sensitive to both PFA and OPC contents. For the type of PFA used, the variability of measured air content or the amount of air retained after continued agitation both indicated that vinsol based AEAs show the highest variability whilst fatty acid based AEAs show low variability. The between batch variability of air content was significantly improved by the addition of PFA regardless of the AEA used. © 1997 Elsevier Science Limited

Keywords: Air entrainment, fresh concrete, PFA, air entraining agents, dosage, variability.

INTRODUCTION

Air entrainment is the process whereby many small air bubbles are incorporated into fresh concrete to become part of the matrix that binds the aggregate together in the hardened concrete. This technique is primarily used to improve the freeze–thaw resistance of a concrete. With the increasing use of PFA as a cement replacement material it is important to understand how the air entrainment process is affected by such additions.

Air entrainment is usually accomplished by the use of an air entraining agent (AEA).[1,2] These are generally referred to as admixtures, as they are added to the other ingredients at the time of mixing. There is a range of materials which can be used as AEAs, which vary in their effectiveness in concrete.[3,4]

Current knowledge concerning the specific interaction of AEAs with various materials is insufficient to predict their behaviour in advance of preparation of a concrete batch.[5] While manufacturers do supply information on recommended dosage rates for their products, these should be viewed as no more than starting estimates.

PFA may make the problem of air entrainment much more complicated, as it may affect both the air content and the stability of the entrained air voids.[6,7] Although the importance of air entrainment and the properties of PFA concrete are well known,[8,9] very little research appears to have been devoted to the study of air entrained PFA concrete. This paper presents the results of work undertaken to investigate the influence of PFA on air entrainment in fresh concrete.

EXPERIMENTAL DETAILS

Programme of investigation

The work undertaken in this study was divided into three areas:

Firstly, the effect of PFA on the required dosage of AEA for a given air content was investigated.

Secondly, the effect of PFA on air content stability was examined. The air content stability includes statistical variability of the same mix with different types of PFA and the repeatability of the same concrete in repeat castings.

Thirdly, and finally, the air loss in air-entrained PFA concrete was explored.

Materials

A single ordinary Portland cement (complying with BS 12: 1991) was used in this investigation (Table 1). PFA (complying with BS 3892: Part 1: 1982) was obtained from nine different sources, representing the range of ashes produced in the UK. The physical properties and model analyses of the PFAs are given in Table 2. The coarse aggregate used was a natural gravel of 20 mm maximum size, prepared from two single sized fractions, 20–10 mm and 10–5 mm. The fine aggregate was a natural sand of zone M grading (complying with BS 882: 1983). Eight air entraining agents (complying with BS 5075: Part 2: 1982) were used in this investigation. The generic types of the AEAs are summarised in Table 3.

Table 1. Properties of the cement used

Property	*% by weight*
Oxide composition	
CaO	62.4
SiO_2	21.9
Al_2O_3	4.9
Fe_2O_3	3.5
SO_3	2.6
MgO	3.4
Na_2O	0.8
LOI	0.8
Bogue compound composition	
C_3S	42.3
C_2S	31.1
C_3A	7.0
C_4AF	10.5
Physical properties	
Fineness (m^2/kg)	306
Specific gravity	3.1
Setting times (min)	
Initial	200
Final	265
Compressive strength (N/mm^2)	
3 day	17.5
7 day	28.0
28 day	46.5

Mix proportion and preparation

In Part 1 of this study, initially the effect of PFA on AEA dosage requirement was investigated for a fixed PFA content. The required dosage of each AEA (A1–8) to achieve an air content of $5{\cdot}5 \pm 0{\cdot}5\%$ was determined for Mixes OPC and PFA (Table 4) using PFA P1. A 28-day strength of 35 N/mm^2 was achieved for these two mixes. PFA P1 was chosen to establish the base dosage requirement as it had a fineness and LOI which was nearest to the mean of the available PFA samples.

The effect of PFA quantity on the required dosage of AEA was then investigated. It was studied using the combination of a single AEA (A1) with three PFAs (P1–3) and also a single PFA (P1) with three AEAs (A1, A5 and A8). Control mixes (M1–1, M2–1, M3–1, M4–1 in Table 4) were designed with cement contents of 250, 300, 350 and 400 kg/m^3 to give a slump of 50 mm. For each cement content, mixes were designed using PFA–OPC replacement levels of 10, 20, 30, 40, and 50% by volume. (Percentage volume was used since there is a big difference in specific gravity between OPC and PFA.) In

Table 2. Physical properties and model analysis of the PFAs

PFA Code	*P1*	*P2*	*P3*	*P4*	*P5*	*P6*	*P7*	*P8*	*P9*
Physical properties									
LOI	3.6	1.3	2.8	4.4	2.4	3.3	3.5	3.0	2.3
Fineness*	7	5.4	3.3	11.8	7.5	6.7	5.3	4.0	5.7
WR	90	90	89	90	90	89	90	88	91
PAI	101	101	108	92	93	107	103	112	101
OM	0.42	0.02	0.37	0.75	0.12	0.24	0.22	0.08	0.22
Model Analysis									
Quartz	1.81	0.86	1.47	3.92	4.37	3.10	3.26	2.12	5.66
Mullite	8.01	8.61	10.01	10.21	5.98	9.86	7.94	7.93	29.29
Magnetite	4.74	5.22	4.1	4.10	8.12	2.72	4.65	1.73	2.50
Haematite	1.65	1.88	1.45	2.20	2.50	1.73	2.30	0.93	2.27
Amorphous	80.19	82.13	81.17	75.17	76.63	79.29	78.35	84.29	57.98

*Mass (%) retained on 45 μm sieve.
LOI, loss on ignition; WR, water reduction; PAI, pozzolanic activity index; OM, readily oxidizable organic matter.

Table 3. Code name and generic type of the AEAs

Code name	*Generic type*	*Specific gravity (g/ml)*
A1	Blend of vinsol resin and synthetic surfactants	1·020
A2	Protenation surfactant	1·020
A3	Vinsol resin	1·020
A4	Sulphonated hydrocarbon	1·020
A5	Vinsol resin	1·062
A6	Epoxy sulphate	1·010
A7	Modified fatty acid	1·014
A8	Salt of fatty acid	1·020

this way the water/cementitious ratio and the aggregate contents within each of the six batches can be held constant, so that any influence of the aggregate might have on air entrainment can be discounted.

For all mixes in Part 1, the dosage of AEA required to obtain $5{\cdot}5 \pm 0{\cdot}5\%$ air content was determined for each mix using trial batches.

For Part 2 of this study, to assess the effect of PFA on the variability of air content, nine PFAs were used with eight AEAs. The AEA dosage used throughout was that found in Part 1 to be required for a $5{\cdot}5 \pm 0{\cdot}5\%$ air content in concrete containing PFA P1.

The repeatability of air entrainment was determined using Mixes OPC and PFA

Table 4. Mix proportions

Mix	*Constituent materials (kg/m³)*						
	Aggregates			*OPC*	$V_f/V_{(c+f)}$*	*PFA*	*Water*†
	20 mm	*10 mm*	*Sand*				
OPC	780	390	625	306	—	—	155
PFA	800	400	555	258	35	100	151
M1–1	795	400	660	250	0	—	152
M1–2	795	400	660	225	10	17.5	147
M1–3	795	400	660	200	20	35.0	143
M1–4	795	400	660	175	30	52.0	138
M1–5	795	400	660	150	40	70.0	134
M1–6	795	400	660	125	50	87.5	130
M2–1	790	395	620	300	0	—	152
M2–2	790	395	620	270	10	21.0	147
M2–3	790	395	620	240	20	42.0	143
M2–4	790	395	620	210	30	63.0	138
M2–5	790	395	620	180	40	84.0	134
M2–6	790	395	620	150	50	105.0	130
M3–1	775	390	590	350	0	—	152
M3–2	775	390	590	315	10	24.4	147
M3–3	775	390	590	280	20	48.9	143
M3–4	775	390	590	245	30	73.3	138
M3–5	775	390	590	210	40	97.8	134
M3–6	775	390	590	175	50	122.2	130
M4–1	770	385	550	400	0	—	155
M4–2	770	385	550	360	10	27.9	150
M4–3	770	385	550	320	20	55.9	146
M4–4	770	385	550	280	30	83.8	141
M4–5	770	385	550	240	40	111.8	136
M4–6	770	385	550	200	50	139.7	132

*Volume ratio of PFA to cement plus PFA.
†Water content adjusted to give constant workability with different AEAs.

(Table 4) with AEAs A1 and A8, i.e. four separate mixes, two OPC concretes and two PFA concretes. PFA P1 was used and each mix was cast 10 times using the required AEA dosage determined earlier.

For Part 3, the extent of air loss between mixing and finishing was studied using the concrete mixes with different AEA–PFA combinations made from Parts 1 and 2 in this study.

All concretes were mixed in a $0{\cdot}04\ m^3$ horizontal pan mixer using a mixing procedure adopted from BS 5075: Part 2: 1982. The weighed aggregates were placed in the mixer and mixed for 1 min, approximately half the gauged water was added and mixing continued for a further 1 min. After standing for 8 min with the pan covered, the cement was added and the mixer restarted. After 30 s, the remaining gauged water was added over the next 30 s and the concrete mixed for a further 3 min.

Using vibration compaction, 100 mm cubes were cast and stored under wet hessian for the first 24 h prior to demoulding. All the specimens were then cured in standard conditions, i.e. water at 20 ± 1°C.

Test methods

The slump of all the mixes used in the investigation was measured immediately after mixing, using the procedure given in BS 1881: Part 102: 1983. For some PFA concrete mixes the slump was measured again at 60 min after the initial mixing procedure had been completed.

The air contents of the fresh concretes were measured using a CAPCO pressure-type air meter, according to Method B given in BS 1881: Part 106: 1983. The calibration of the air meter was carried out regularly to ensure accuracy of the measured air contents. For OPC and OPC/PFA concretes, the air content was measured at both 15 and 60 min after mixing.

RESULTS AND DISCUSSION

Effect of PFA on AEA dosage requirement

The results of the dosage requirements of the eight different AEAs for an air content of $5{\cdot}5 \pm 0{\cdot}5\%$ with both the control OPC and OPC/PFA concrete mixes are given in Table 5. As can be seen, the required dosages for the neat OPC mix is different for different AEAs.

Also the required dosage for the corresponding OPC/PFA mixes is higher, but because of the varying base (OPC) levels it is difficult to make direct comparisons.

In order to overcome this we can define a dosage index (DI), which is the ratio of the amount of an AEA required when PFA is used to the amount of AEA required when neat OPC is used, i.e.

$$\text{Dosage index (DI)} = \frac{\text{AEA required with PFA}}{\text{AEA required with OPC}}$$

The DI can be used at any specified air content, or level of PFA. In the present study the air content was held constant at $5{\cdot}5 \pm 0{\cdot}5\%$, and all quoted DI values refer to this air content only. Therefore, the DI value gives us a method of directly comparing the relative efficiency of the various AEAs in concretes containing differing amounts, and types of PFAs.

The DI values calculated for the control mixes are given in Table 5, and plotted in bar graph form, in ascending order, in Fig. 1. As can be seen, there is a range of values from 1·9

Table 5. Dosage requirement of control mixes

AEA code	*OPC dosage (ml/50 kg cement)*	*OPC/PFA dosage (ml/50 kg (cement+PFA))*	*DI*
A1	40	144	3.60
A2	70	133	1.90
A3	75	245	3.27
A4	65	288	4.43
A5	40	101	2.53
A6	65	288	4.43
A7	50	288	5.76
A8	80	504	6.30

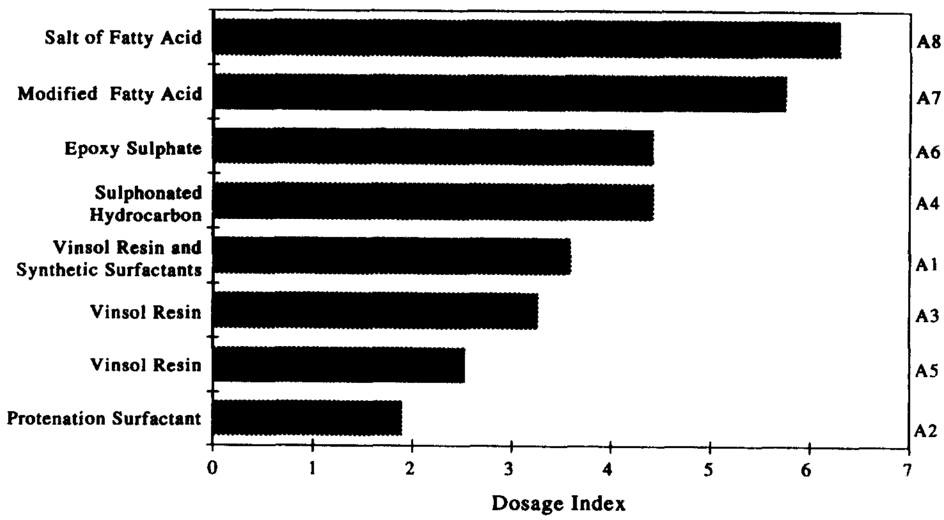

Fig. 1. Influence of AEA type on dosage index.

to 6·3, however, what is most important is the correlation between the dosage index and the generic type of the AEA. The data suggests that the protenation surfactant (A2) is more than three times as efficient in the presence of PFA than the salt of a fatty acid (A8). More correctly AEA A2 is less affected by the presence of PFA than AEA A8. In all cases, the presence of PFA in the concrete mix required a higher addition of AEA to achieve the desired air content. This is in general agreement with results of work by Gebler and Klieger.[7]

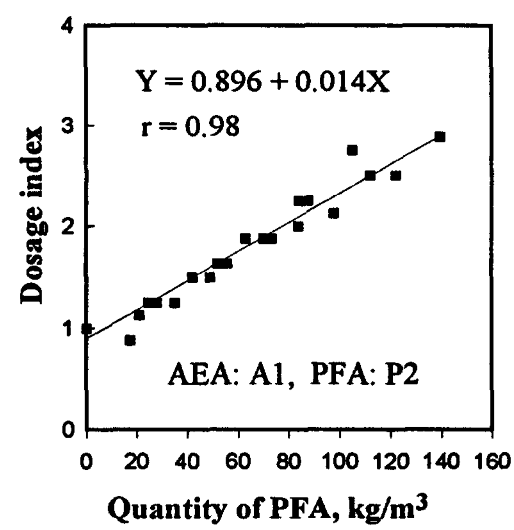

Fig. 3. Effect of PFA content on dosage index (A1–P2).

Effect of PFA type and content

The test results for the series of concretes using a single AEA (A1), with OPC and partial replacement with PFAs P1, P2 and P3 are given in Figs 2, 3 and 4.

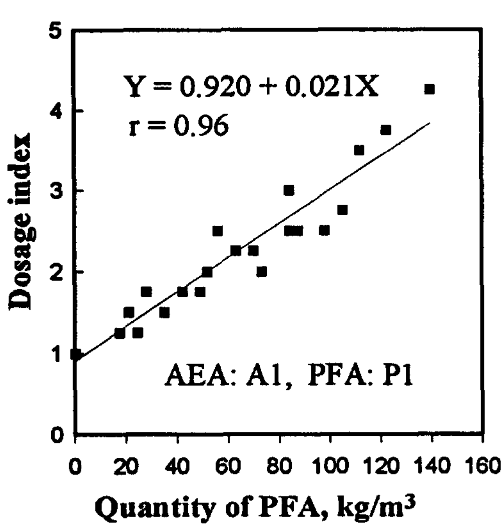

Fig. 2. Effect of PFA content on dosage index (A1–P1).

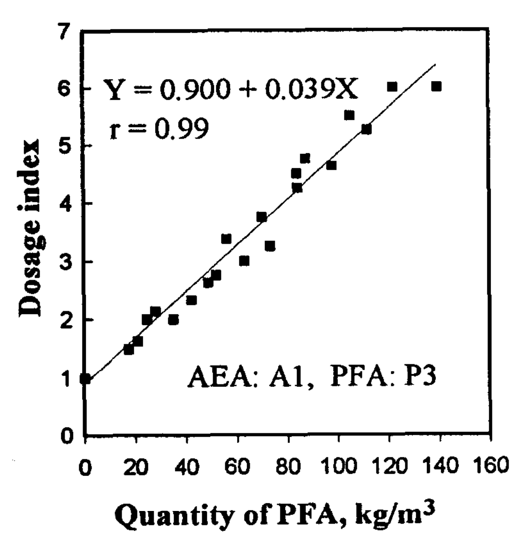

Fig. 4. Effect of PFA content on dosage index (A1–P3).

The calculated dosage indices are shown as a function of PFA addition in the figures. As can be seen from the figures, the data gives rise to a straight line relationship for each PFA used. Linear regression data are also given in the figures. The straight line relationships indicate that when AEA A1 is used, the dosage required to achieve the target air content is directly proportional to the amount of PFA in the concrete mix, and is independent of the total cementitious content. The slope of the line changes with the PFA used indicating that the source of the PFA influences the dosage requirement. However, from the available PFA physical and analytical data, this effect cannot be ascribed to a particular property of the PFA.

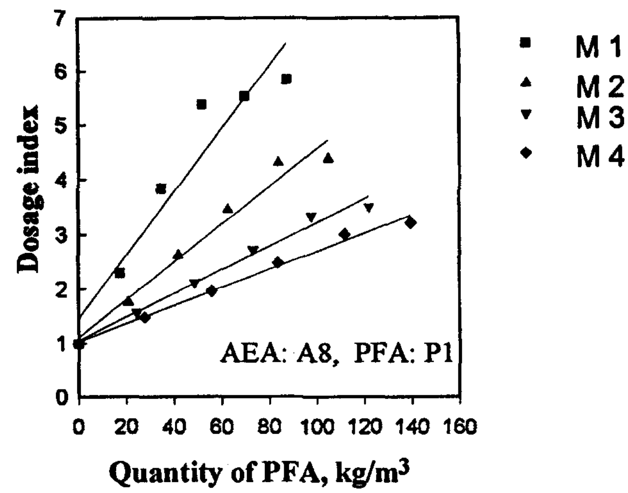

Fig. 6. Effect of PFA content on dosage index (A8–P1).

Effect of AEA type

In this study two additional AEAs, A5 and A8, were used in concretes modified with PFA P1. The P1–A1 combination from the previous study completed the data. As before the dosage indices were plotted as a function of the amount of PFA in the concrete mixes for each combination. The plots are shown in Figs 5 and 6.

As can be seen for the A5–P1 combination a straight line relationship similar to those found previously is evident, whilst the A8–P1 combination shows a completely different relationship. The linear regression data for the A5–P1 combinations are included in Fig. 5, and the slope of the line is higher than that found for the A1–P1 combination, possibly indicating that AEA A5 is more sensitive to the presence of PFA than AEA A1.

Figure 6 shows that AEA A8 gives rise to a family of related curves apparently dependant upon the total cementitious content as well as the PFA content. AEA A8 is the salt of a fatty acid and it may be that the polarised molecules are being attracted to the surface of the cementitious particles, thereby reducing the amount available for air entrainment. However, the effect seems to be ameliorated at higher total cementitious contents, which may be due to the higher amount of soluble lime available (because of the higher OPC content), and the subsequent precipitation of the fatty acid, as the calcium salt, on the surface of the particles.

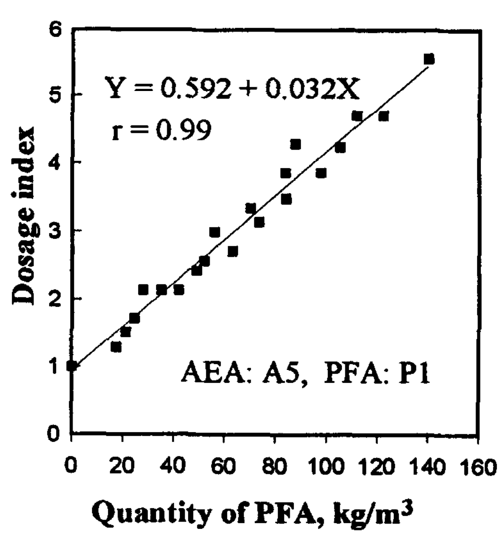

Fig. 5. Effect of PFA content on dosage index (A5–P1).

Effect of PFA on air content stability

Variability

The results of a statistical analysis of air content variability are given in Table 6. Significant differences in air content were found in concretes using different sources of PFA. The highest air content (11·5%) was obtained when PFA P2 was used with AEA A3, and the lowest air content was obtained (3·8%) when PFA P4 was used with AEA A5. Thus the observed range of air content was 7·7%. The highest coefficient of variability (33·5%) was seen in the nine PFA concretes which used AEA A1. AEAs A2, A3, A5, and A6 also gave high percentage CV values (18·8–29·3%), whilst AEAs A4, A7, and A5 gave much lower percentage CV values (9·9–12·9%).

Table 6. Statistical analysis of air content variability

Concrete type	*Air content*					
	No. of readings	*Mean (%)*	*Minimum (%)*	*Maximum (%)*	*SD (%)*	*CV (%)*
PFA	10	0.79	0.55	0.95	0.11	13.8
PFA–A1	10	6.8	4.5	11.4	2.26	33.5
PFA–A2	10	6.8	4.1	11.0	2.00	29.3
PFA–A3	10	8.2	5.2	11.5	2.01	24.7
PFA–A4	10	5.4	4.1	6.1	0.70	12.9
PFA–A5	10	7.2	3.8	11.0	2.07	28.7
PFA–A6	10	6.9	5.0	9.3	1.30	18.8
PFA–A7	10	5.4	4.4	6.2	0.55	10.2
PFA–A8	10	5.0	4.4	5.8	0.50	9.9

A high value of percentage CV indicates that the AEA in question is very sensitive to the source of PFA; conversely a low value of percentage CV indicates a low sensitivity to PFA source in terms of air entrainment. The AEAs with the highest percentage CV values are those based on vinsol resin, surfactants or epoxy sulphate. As we have seen previously, these types of AEAs tended to have the lowest dosage indices when used with the control mixes. Conversely, those AEAs based on sulphonated hydrocarbons or fatty acids which tended to have the highest dosage indices, yield the lowest percentage CV values and therefore appear to be less sensitive to the source of PFA.

Repeatability

The test results of the air contents measured in the fresh concretes in the repeatability series clearly show that the fluctuation in air content of fresh PFA concretes between batches of the same mix is less than that for OPC concretes. The coefficient of variability for OPC mixes using AEAs A1 and A8 were 11·8 and 13·8%, respectively, whereas for the PFA modified mixes values of only 7·2 and 7·9%, respectively, were found. This indicates that the inclusion of PFA reduces the variability of air entrainment.

Air loss due to handling and agitation

The air contents retained after 60 min, expressed as a percentage of the 15 min level, were used to measure the air loss due to handling and agitation.

In order to simulate the handling and agitation of concrete *in situ*, the concretes were firstly mixed as before according to BS 5075, and the slump and wet density measured. At 15 min after mixing the air content was determined, and cubes were cast. The remaining concrete was then mixed for 5 min and allowed to stand for 5 min. This was repeated twice more. The mixer was covered to prevent evaporation of the mixing water during the three cycles. The air content was measured again 60 min after the completion of the initial mixing. The cycles of agitation were intended to simulate the effects of the certain types of delivery, placing and compaction.

Test results shown in Table 7 indicated that the type of AEA used has a significant effect on the amount of air that is retained in the PFA modified concrete, while the effect of PFA source is less. The lowest retained air content was found using AEA A2, a protenation surfactant. The vinsol resin based AEAs, A3 and A5, showed low retention of air: 70·7 and 53·2%, respectively. The vinsol–surfactant blend, AEA A1, showed reasonable air retention at 92·9%. However the long chain hydrocarbon based AEAs, A4, A7 and A8 showed good air retention, with a slight increase at 60 min. The epoxy sulphate AEA, A6, showed a large increase in air content with continued agitation.

It is noticeable that the lowest variability is again found for the long chain hydrocarbon based AEAs, with the two fatty acids based AEAs (A7 and A8) showing very low variability. This reinforces the earlier findings that these AEAs are the least sensitive to the source of the PFA.

CONCLUSIONS

For a given PFA level, the dosage required to produce an air content of $5{\cdot}5 \pm 0{\cdot}5\%$ in a PFA

Table 7. Percentage retained in air after 60 min

PFA code	*AEA used*							
	A1	*A2*	*A3*	*A4*	*A5*	*A6*	*A7*	*A8*
P1	73	51	78	94	47	126	138	127
P2	96	41	74	131	56	187	112	114
P3	96	31	70	108	55	117	125	135
P4	34	34	63	80	26	69	109	123
P5	108	36	102	118	68	162	120	104
P6	73	31	60	124	49	125	106	112
P7	92	51	51	127	48	159	117	119
P8	141	51	69	145	68	190	130	120
P9	123	44	69	144	62	161	103	106
Mean	92.9	41.1	70.7	119.0	53.2	144.0	118.0	117.8
SD	31.3	8.5	14.2	21.8	12.9	38.5	11.6	9.5
CV%	33.5	20.8	20.1	18.3	24.4	26.7	9.9	8.3

modified concrete mix is two–six times that required in the corresponding neat OPC concrete mix. AEAs based on long chain hydrocarbons (sulphonated hydrocarbons, fatty acids) are at the higher end of the range, while those based on surfactants and vinsol resin are at the lower end of the range.

The dosage of a vinsol based AEA required to produce a set air content appears to be directly related to the PFA content of the mix, and is independent of the OPC content. Similar direct relationships were obtained with a range of different PFAs. However, the dosage of an AEA based on the salt of a fatty acid appears to be sensitive to both PFA and OPC contents.

The variability in air content obtained when different PFAs are used with set replacement levels and AEA dosage is highest for the vinsol resin based AEAs. AEAs based on fatty acids show remarkably low variability with change of PFA.

The between batch variability of air content in air entrained concrete was significantly reduced by the addition of PFA regardless of the AEA used.

The amount of air retained in air entrained PFA concrete after continued agitation simulating handling, placing and compaction, was dependant on the AEA used and relatively independent of the PFA source. Vinsol resin based AEAs gave the lowest retention and highest variability with PFA source, whilst fatty acid based AEAs gave good retention and low variability with PFA source.

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