

Use of Defoaming Agent for Elimination in Large Entrained Air Bubbles to Improve Air Stability in Mortar of Self-compacting Concrete with High Volume Fly Ash

Nipat Puthipad^{1*}, Anuwat Attachaiyawuth¹ and Masahiro Ouchi¹

¹ School of Systems Engineering, Kochi University of Technology
185 Tosayamadacho-Miyanokuchi, Kami, Kochi, 782-8502, JAPAN

*E-mail: 196013j@gs.kochi-tech.ac.jp

Abstract: This paper presents an experimental study on the stability of entrained air bubbles in mortar of self-compacting concrete with high volume fly ash. The use of defoaming agent (DA) for promoting the entrainment of fine air bubbles in mortar with fly ash is also investigated. The stability of entrained air bubbles is quantified in term of change in volume of air within 2 hours. The size distributions of the air bubbles in the fresh mortar are measured by the air void analyser (AVA) to analyse the influence of bubble size on the air stability. The change in the air size distribution during the period of 2 hours is also used to analyse the unification of entrained air bubbles. The results suggest that the increase in fly ash content (fa/m) tended to reduce the volume of entrained air bubbles in mortar of self-compacting concrete. This is found be caused by higher amount of large and unstable entrained air bubbles produced when fly ash is employed, despite different types of air-entrained agent (AEA) used. Besides, the spherical shape of fly ash tended to ease the movement of entrained air bubbles. This can cause easier unification and escape of the air bubbles in the mortar of self-compacting concrete. However, in general, the use of DA can eliminate these large and unstable entrained air bubbles and improve the air stability in mortar of self-compacting concrete with high volume fly ash. Apparently, DA can enhance the air stability in mortar with fly ash to be the same level as that in mortar without fly ash and DA.

Keywords: self-compacting concrete, defoaming agent, air void analyser, fly ash, air-entraining agent, entrained air bubbles, air stability

1. Introduction

As a means to produce durable concrete without the necessity of skilled labour, self-compacting concrete (SCC) was developed (Ozawa et al, 1992) Mortar and paste phases in SCC are required to have suitable deformability and viscosity in order for the

fresh concrete to attain adequate self-compactability. In order to achieve the suitable deformability and viscosity of the mortar and paste phases, SCC typically composed of lower water to cement ratio, as well as higher cement content, when comparing with conventional concrete (Fig. 1). This suggests

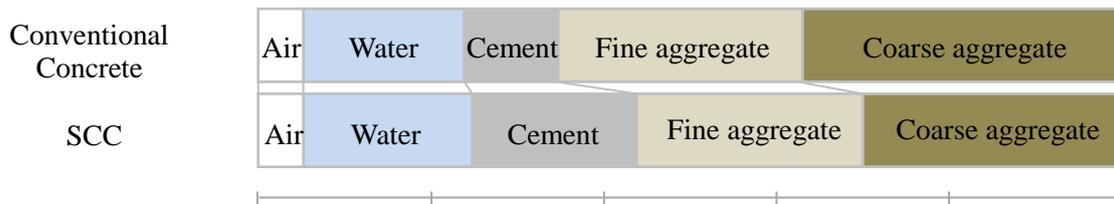


Fig.1 Volumetric mixture proportion of self-compacting concrete (SCC) and conventional concrete

higher costs and poorer sustainability, in terms of the environment, of SCC.

The employment of fly ash has been one of the most widely used approaches in reducing the cement content in SCC (Güneyisi et al, 2013, Khaleel and Razak, 2012, Liu, 2010, Naik et al, 2012, Puthipad et al, 2016 and Siddique et al, 2012). Since fly ash can undergo pozzolanic and hydration reactions, with suitable replacement ratio, partial replacement of cement with fly ash has been found to be beneficial in sustainability of SCC. Beside this, Puthipad et al (2016a) has investigated that, with spherical shape, the ball-bearing effect of fly ash can enhance the self-compactability of fresh concrete and allow the employment of higher fine aggregate content (s/m) in SCC. Therefore, the cement content in SCC can be further reduced.

Recently, air-entrainment has also been found to be effective in reducing the cement content in SCC (Attachaiyawuth and Ouchi, 2014, Attachaiyawuth et al, 2015 and 2016). Through the increase in total volume of SCC, the air-entrainment can reduce the cement content in SCC significantly. Moreover, with suitable characteristics, the entrained air bubbles can allow higher s/m and further reduce the usage of cement in SCC (Attachaiyawuth et al, 2015 and Attachaiyawuth et al, 2016). Although air-entrainment is known to considerably reduce the compressive strength of concrete, with appropriate air content, adequate compressive strength can be attained.

Up to now, Puthipad et al 2016b has suggested the potential of the combined effect of air-entrainment

and the employment of fly ash to potentially further enhance the sustainability, in terms of the environment, of SCC. However, the entrained air bubbles has been suggested to be less stable, due to the employment of fly ash. This attributes to higher amount of large and unstable air bubbles being entrained, when fly ash has been employed (Puthipad et al, 2016b). Also, the spherical shape of fly ash has suggested the easier movement, unification and escape of the entrained air bubbles.

Seemingly, Ouchi et al (2017) has reported that defoaming agent (DA) was able to eliminate large and unstable air bubbles in SCC. This suggests the potential of DA in enhancing the volumetric stability of entrained air bubbles in SCC with fly ash.

Therefore, in this research, the reduction in volumetric stability of entrained air bubbles, due to the employment of fly ash, is clarified. The employment of defoaming agent for the improvement in air stability of SCC with fly ash is also investigated.

2. Indices and Test Method Used for Evaluating the Volumetric Stability of Entrained Air Bubbles in Fresh Mortar of Self-compacting Concrete with Fly Ash

2.1 Indices for controlling flowability of fresh mortar

The flowability of fresh mortar is known to be influenced by fly ash and entrained air bubbles through the ball-bearing effect and reduction in water demand (Attachaiyawuth and Ouchi, 2014,

Attachaiyawuth et al, 2015, 2016 and Puthipad et al, 2016a and 2016b). The employment of fly ash and air-entrainment are known to change the deformability and viscosity of the fresh mortar, and hence, the self-compactability of fresh concrete. In this work, the deformability of fresh mortar was determined, in terms of relative flow area (Γ_m), by the mortar flow test (Fig. 2). The viscosity of the fresh mortar was quantified, in terms of relative funnel speed (R_m), by the mortar funnel testing. These tests and indices were used to control the flowability of the fresh mortar.

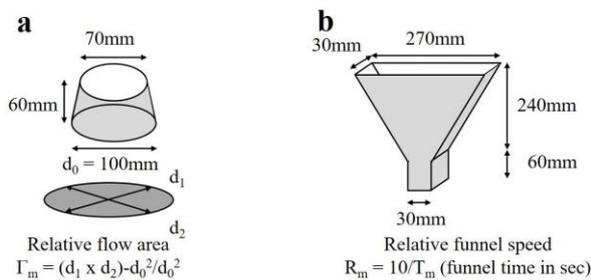


Fig.2 Mortar flow cone test (a) and mortar funnel test (b)

2.2 Test method for Evaluating the Volumetric Stability of Entrained Air Bubbles in Fresh Mortar of Self-compacting Concrete with Fly Ash

2.2.1 Materials

The properties of the materials used, in the experiments conducted, are presented in Table 1. Ordinary Portland cement (OPC) were used, as well as class 1 fly ash as specified by Japanese Industrial Standards (Japanese Industrial Standards, 2009 and 2015a). The spherical shape of class 1 fly ash used are suggested in Fig. 3. The admixtures used in the experiment included superplasticiser (SP), air-entraining agent (AEA) and defoaming agent (DA). The SP used was polycarboxylate base blended with a viscosity modifying agent (VMA) to enhance the stability of SCC. Two types of AEA were used. These include AEA1, which is modified rosin based, and AEA2, which is a long-chain

alkylcarboxylate based. AEA2 is also specified to prevent the carbon on fly ash surface from affecting air-entrainment. DA used, for eliminating large and unstable entrained air bubbles, was polyalkyleneglycol derivative.

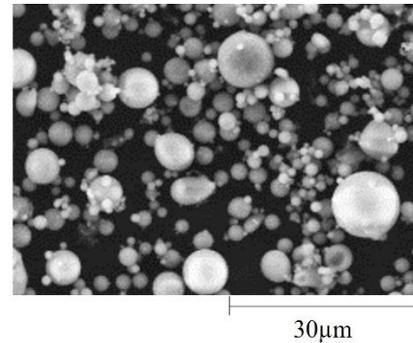


Fig.3 SEM image, with the magnification of 2500x, showing the spherical shape of the fly ash

2.2.2 Testing and mixing method

All batches of mortar were mixed using the mixing processes presented in Fig. 4. Since this method was found to be beneficial in entraining high volume of air and effective with defoaming agent, it was conducted in this experiment (Ouchi et al, 2017 and Rath et al, 2016). The batch size for mortar mixing in this experiment was 1.68 litres. The mixer used complied with the Japanese Industrial Standard (2015b).

All the tests, conducted in this experiment, for evaluating the volumetric stability of entrained air bubbles in SCC with fly ash, are shown in Table 2. The mortar tests in the initial state were repeated three times for each mortar mix proportion, and the averaged results were analysed. Additional batch of each mortar mix proportion was tested for investigating the volumetric stability of entrained air over time. The size distributions of entrained air bubbles were also measured, at both initial state and 2 hours, by air void analysis, for clarification of the research (Fig. 4). After tests at 5 and 20 min, every batch of mortar was rested and covered with moist towels to prevent moisture loss.

Table1 Properties of materials used

Materials	Type	Specific gravity (g/cm ³)	Fineness (cm ² /g)	LOI (%)	F.M.
Powder	OPC [23]	3.15	3490	1.96	-
	Fly ash I [24]	2.40	5500	1.90	-
S	Limestone	2.68	-	-	2.90
MCA	Glass beads	2.54	Diameter: 10mm		
SP	Polycarboxylate-based blended with VMA				
AEA1	Modified rosin based anionic surface active agent				
AEA2	Long-chain alkylcarboxylate based anionic surface active and non-ionic surface active agent				
DA	Polyalkyleneglycol derivative				

Table2 Mortar testing procedures

Initial state		At 120 minutes*
5 minutes	20 minutes	
- Flow test	- 5 sec remix	- 5 sec remix
- V-funnel test	- Flow test	- Flow test
- Air measurement (Gravimetric method and AVA*)	- V-funnel - Air measurement (Gravimetric method)	- V-funnel - Air measurement (Gravimetric method and AVA)

*Conducted only once for each mortar mixture proportion

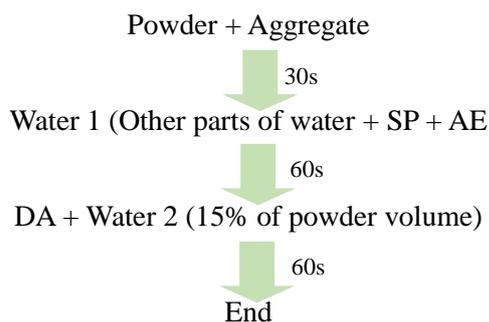


Fig.4 Mortar mixing procedure



Fig.5 Equipment for air void analysis (AVA)

2.2.3 Mortar mix proportions

Table3 presents all the mortar mix proportions tested in this experiment. Two ratios of fly ash to mortar, including air, (*fa/m*) were considered, including 0.04 and 0.10. The relative funnel speed (R_m) and relative flow area (Γ_m) were controlled by altering the water-to-powder ratio (*w/p*) and the dosage of superplasticiser (SP/P), respectively

(Table 4). The R_m of every batch of mortar was controlled to be in the range of 1.30-1.40, while Γ_m was in the range to 5.5-6.6 (Table 4). Besides the air content of every mortar mixture proportions was targeted to be in the range of 10-12%, by adjusting the dosage of AEA. Therefore, s/m, including air, in every batch of mortar was between 0.49 and 0.50. Moreover, the DA was employed into the mortar with the dosage of 0.05% and 0.10%, by total weight of powder to evaluate the enhancement in volumetric stability of the entrained air bubbles.

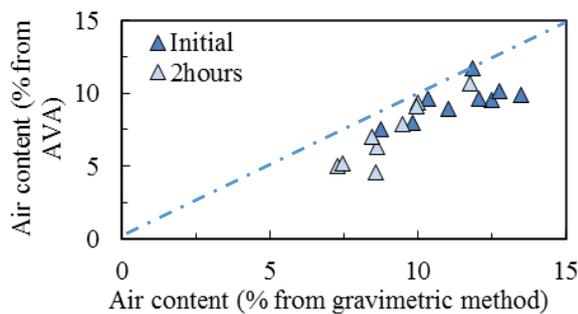


Fig.6 Lower air content measured by AVA, as compared to that measured by gravimetric method

2.3 Adjustment on the Size Distribution of Entrained Air Bubble

2.3.1 Adjustment on the size distribution of entrained air bubble by consideration on the lower air content obtained by AVA in comparison to gravimetric method

The results of air measurement have shown that the air content obtained by AVA tended to be lower than those obtained by the gravimetric method (Fig. 6). Magura (1996) has suggested that this can be attributed to the limitation of AVA, where the large air bubbles, with the size of over 3000 μm , are not measured. The preparation of the smaller samples for AVA, when comparing with that for gravimetric method, might also cause higher chance of the large entrapped air bubbles to be escaped. However, these large entrained air bubbles can influence the volumetric stability of the entrained air, as well as the self-compactability of fresh concrete. Thus, these

bubbles were also considered in the size distributions obtained. All the measured size distributions were adjusted by adding the difference in air content obtained by the two methods to the volume of air bubbles with diameter over 1500 μm , as illustrated in Fig. 7 (Puthipad et al, 2016b and Rath et al, 2017).

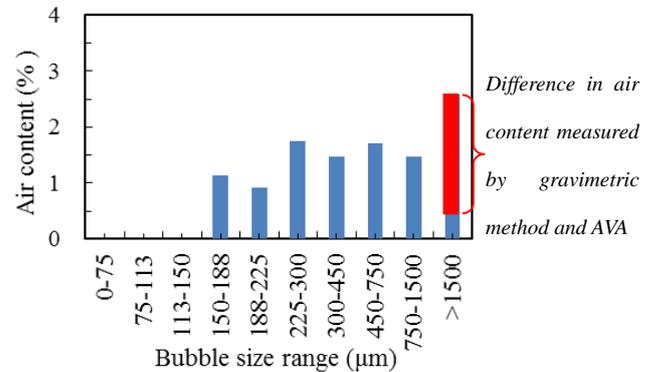


Fig.7 Adjustment of large air bubbles (size over 1500 μm) by considering the difference in air content measured by gravimetric method and AVA

2.3.1 Adjustment on the size distribution of entrained air bubble for analysing the unification of entrained air bubbles

The volumetric stability of entrained air bubbles has been suggested to be effected by the unification of fine entrained air bubbles, into large bubbles, and escaped (Puthipad et al, 2016b and Rath et al, 2017). Hence, in order to analyse the influence of the bubble size distribution on the volumetric stability of entrained air, the unification of the air bubbles was considered. This was conducted through the assumption that the change in fine entrained air bubbles, with the size smaller than 1500 μm , during 2hours, was caused by the unification. Thus, for evaluating the volumetric stability of entrained air bubbles, this change was added to the volume of large air bubbles to obtain the adjusted total amount of large air bubbles before they escaped (Fig. 8).

Table3 Mortar testing procedures

Mix	Mix proportion (kg/m ³)							SP/P (%)	fa/p*	fa/m* including air	s/m* including air
	Cement	Fly ash	Water	S*	AEA1	AEA2	DA				
M1	672	-	237	1474	0.047	-	-	1.65	0.00	0.00	0.50
M2	562	107	227	1474	0.074	-	-	1.55	0.20	0.04	0.50
M3	355	267	226	1474	0.124	-	-	0.84	0.50	0.10	0.50
M4	338	267	242	1474	0.423	-	0.302	0.82	0.51	0.10	0.50
M5	341	267	230	1474	1.095	-	0.608	0.99	0.51	0.10	0.50
M6	656	-	242	1474	-	0.525	-	1.20	0.00	0.00	0.50
M7	341	267	230	1474	-	3.162	-	0.65	0.51	0.10	0.50
M8	331	267	234	1474	-	5.742	0.299	0.56	0.51	0.10	0.50
M9	338	267	232	1474	-	7.257	0.604	0.58	0.51	0.10	0.50

*fa/p: fly ash to powder volumetric ratio; fa/m: volume of fly ash in mortar; s/m: fine aggregate to mortar volumetric ratio; S: fine aggregate

Table4 Mortar testing procedures

Mix	Air content by			
	gravimetric method (%)	Γ_m	R_m	R_{mb}
M1	10.70	5.7	1.36	0.80
M2	10.13	5.5	1.40	0.85
M3	10.94	5.7	1.36	0.90
M4	10.50	5.6	1.38	0.94
M5	10.67	5.5	1.36	0.97
M6	10.46	5.7	1.39	0.89
M7	10.86	5.8	1.39	0.97
M8	10.03	5.8	1.37	0.97
M9	10.73	6.1	1.35	1.02

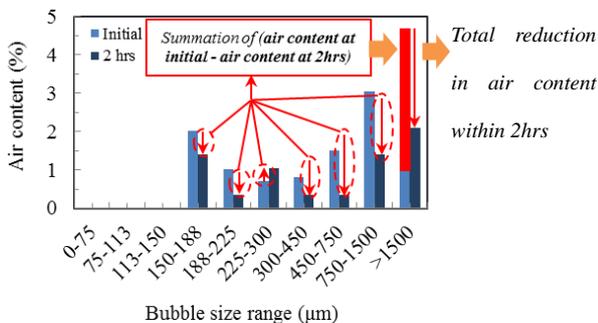


Fig.8 Adjustment of large air bubbles (size over 1500µm) by considering the unification of fine entrained air for analysing the volumetric stability of the air bubbles (Puthipad et al, 2016b)

3. Effect of Fly Ash on the Reduction in Volumetric Stability of Entrained Air Bubbles in Fresh Mortar of Self-compacting Concrete

The volumetric stability and the size distribution of entrained air bubbles was quantified in mortar mix proportions, M1, M2, M3, M6 and M7. The results have suggested that, due to the employment fly ash, the entrained air bubbles tended to be less stable, as illustrated in Fig. 9, with both AEA1 and AEA2. This can be caused by higher amount of large air bubbles, with the size over 1500µm, in mortar with fly ash, as compared to that in mortar without fly ash (Fig. 10). Besides, while fly ash content and types of AEA dependent relationship has been shown between the volumetric stability of entrained air bubbles and amount of large air bubbles, a unique relationship has been illustrated when using adjusted amount of large air bubbles as explained in section 2.3.1 (Fig. 11). This suggests that fly ash, due to its spherical shape, can lead to the easier movement, unification and escape of the fine entrained air bubbles.

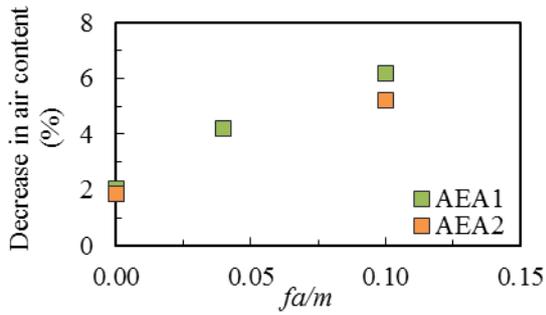


Fig.9 Reduction in volumetric stability of entrained air bubbles due to the employment of fly ash

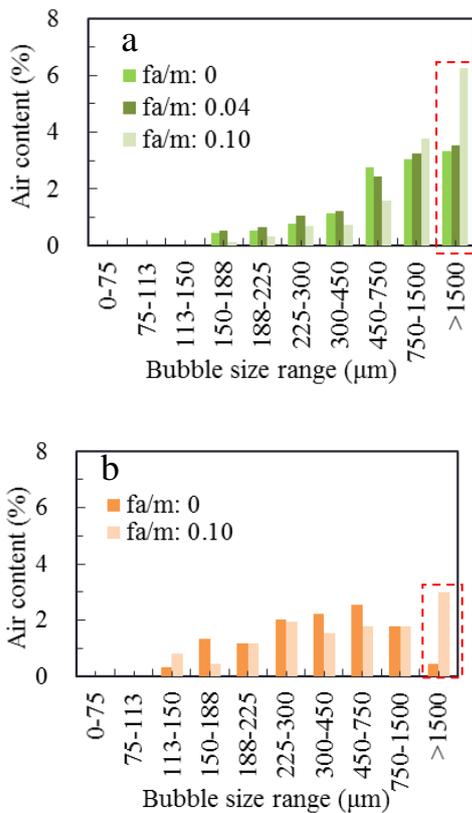


Fig.10 Higher amount of large air bubbles produced, due to the employment of fly ash in mortar with AEA 1(a) and AEA2 (b)

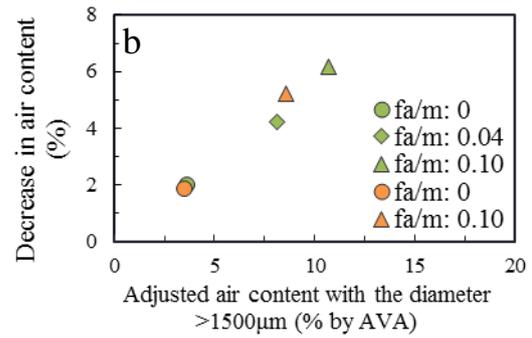
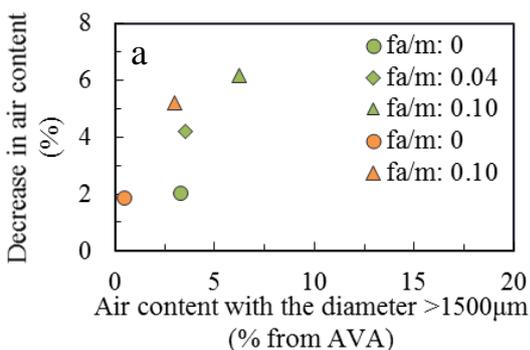


Fig.11 AEA types and fly ash content dependent (a) and unique relation (b) between volumetric stability and amount of large air bubbles before (a) and after (b) adjustment by considering unification of fine entrained air bubbles

4. Effect of Defoaming Agent on Elimination of Large Air Bubbles and Improvement in Volumetric Stability of Entrained Air in Fresh Mortar of Self-compacting Concrete with Fly Ash

The defoaming agent (DA) was employed in mortar mix proportions, M4, M5, M7 and M8. The volumetric stability and size distribution of entrained air bubbles were measured. The obtained results were analysed to evaluate the effect of DA on the volumetric stability of entrained air in SCC with fly ash.

Fig. 12 has illustrated that, generally, the employment of DA can improve the volumetric stability of entrained air bubbles in fresh mortar with fly ash, with both AEA1 and AEA2. Besides, the volumetric stability of entrained air in fresh mortar with fly ash has been improved, by employment of DA, to an equivalent level to that in fresh mortar without fly ash and DA (Fig. 11). This may be mainly attributed to the lower amount of large entrained air bubbles, with the size over 1500µm, in fresh mortar with fly ash, when DA has been employed (Fig. 13). Furthermore, similarly to the previous results, reported by Puthipad et al (2016b), the linear relationship between the adjusted amount of air bubbles with the size over 1500µm and the

decrease in air content of mortar over 2 hours have been shown (Fig. 14). In addition, the results have suggested an increased content of large entrained air bubbles, during the 2 hours, in mortar with fly ash, AEA1 and DA of 0.10% (Fig. 14). This might be caused by re-entrainment of air bubbles during testing and re-mixing.

Equivalent volumetric stability of entrained air

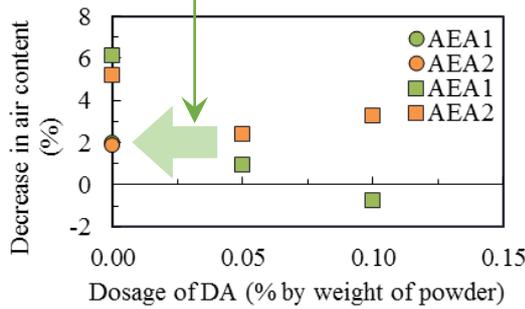


Fig.12 Enhancement in volumetric stability of entrained air in mortar with fly ash, due to employment of DA, to the similar level as that in mortar without fly ash

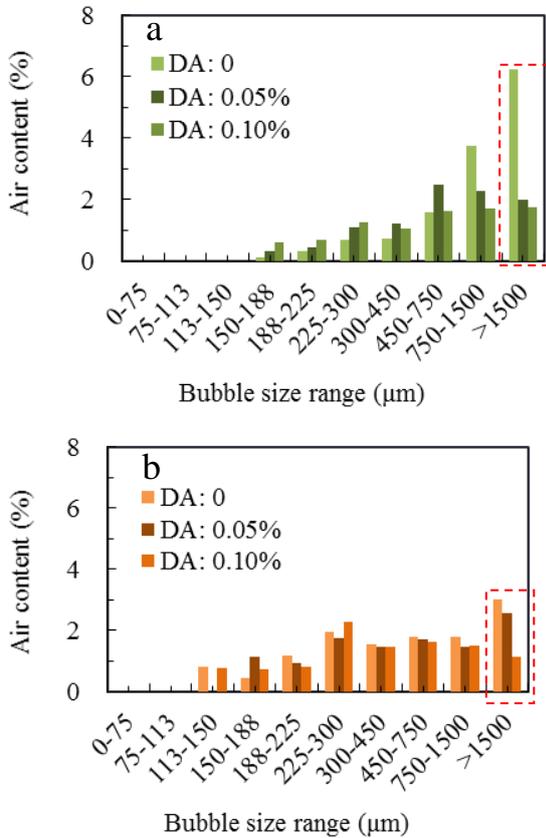


Fig.13 Reduction in amount of large air bubbles, owing to employment of DA, in mortar with AEA1 (a) and AEA2 (b)

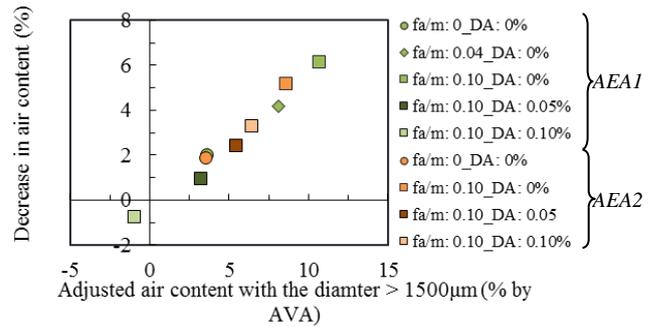


Fig.14 Unique relationship between volumetric stability of entrained air and adjusted amount of large air bubbles after considering unification of fine bubbles

5. Conclusion

In this paper, the volumetric stability of entrained air bubbles in various mortar mix proportions, with and without fly ash, were quantified in terms of the change in air content within 2 hours. The dosage of superplasticiser and water-to-powder volumetric ratio were adjusted to control the relative flow area and funnel speed of all mortar mixes. The dosage of air-entraining agent was also altered to attain the targeted air content. The size distributions of the entrained air bubble in all mortar mixes were measured by the air void analyser. The results were analysed to clarify the effect of fly ash on the volumetric stability of entrained air in fresh mortar of self-compacting concrete. Furthermore, the improvement in volumetric stability of entrained air in fresh mortar with fly ash, by the employment of defoaming agent, was investigated. The results can be concluded as follows:

1. The employment of fly ash leads to the reduction in volumetric stability of entrained air bubbles in fresh mortar of SCC. This attributes to the higher amount of large air bubbles being entrained in mortar with fly ash, as compared to that in mortar without fly ash.
2. The spherical shape of fly ash leads to easier movement, unification and escape of the entrained

air bubbles. This can be caused by lower surface area, to restrain the entrained air bubbles, of fly ash than cement.

3. Employment of defoaming agent eliminates the large entrained air bubbles and enhance the entrainment of fine air bubbles in fresh mortar of self-compacting concrete with fly ash.

4. The employment of defoaming agent improves the volumetric stability of entrained air in fresh mortar, with fly ash, to the same level as that in mortar without fly ash and defoaming agent. This mainly attributes to lower amount of large entrained air bubbles, when defoaming agent has been employed.

The spherical shape of fly ash was shown to reduce the volumetric stability of the entrained air. However, the employment of defoaming agent was found to enhance the entrainment of fine entrained air bubbles. This was found to enhance the volumetric stability of entrained air in mortar of self-compacting concrete with fly ash, to the similar level as that in self-compacting concrete without fly ash. This suggests the potential of defoaming agent for improving the air entrainment in self-compacting concrete with fly ash.

References

- 1) Ozawa, K., Maekawa, K. and Okamura, H., 1992. Development of High Performance Concrete. *Journal of the Faculty of Engineering, the University of Tokyo*. 41(3): 381-438.
- 2) Naik, T.R., Kumar, R., Ramme, B.W. and Canpolat, F., 2012. Development of high-strength, economical self-consolidating concrete. *Journal of Construction and Building Materials*. 30: 463-469.
- 3) Güneyisi, E., Gesoğlu, M. and Algin, Z., 2013. 9 – Performance of self-compacting concrete (SCC) with high-volume supplementary cementitious materials (SCMs). *Journal of Eco-Efficient Concrete*. 198-217.
- 4) Puthipad, N., Attachaiyawuth, A. and Ouchi, M., Ball-bearing effect of fly ash for higher fine aggregate content in self-compacting concrete. *Proceedings of the Japan Concrete Institute*, 38, 1437-1442, 2016a.
- 5) Liu, M., 2010. Self-compacting concrete with different levels of pulverized fuel ash. *Journal of Construction and Building Materials*. 24 (7): 1245-1252.
- 6) Siddique, R., Aggarwal, P. and Aggarwal, Y., 2012. Influence of water/powder ratio on strength properties of self-compacting concrete containing coal fly ash and bottom ash. *Journal of Construction and Building Materials*. 29: 73-81.
- 7) Khaleel, O.R. and Razak, H.A., 2012. The effect of powder type on the setting time and self compactability of mortar. *Journal of Construction and Building Materials*. 36: 20-26.
- 8) Attachaiyawuth, A. and Ouchi, M., Effect of entrained air on mitigation of reduction in interaction between coarse aggregate and mortar during deformation of self-compacting concrete at fresh state. *Proceedings of the Japan Concrete Institute*. 36: 1444-1449, 2014.
- 9) Attachaiyawuth, A., Tanaka, K., Rath, S. and Ouchi, M., Air-enhanced self-compactability of fresh concrete with effective mixing method.

- Proceedings of the Japan Concrete Institute. 37:* 1069-1074, 2015.
- Proceedings of the Japan Concrete Institute. 38:* 1413-1418, 2016.
- 10) Attachaiyawuth, A., Rath, S., Tanaka, K. and Ouchi, M., 2016. Improvement of self-compactability of air-enhanced self-compacting concrete with fine entrained air. *Journal of Advanced Concrete Technology*, 14, 2016, 55-69.
 - 11) Puthipad, N., Ouchi, M., Rath, S. and Attachaiyawuth, A., 2016b. Enhancement in self-compactability and stability in volume of entrained air in self-compacting concrete with high volume of fly ash. *Construction and Building Materials*. 128: 349-360.
 - 12) Ouchi, M., Kameshima, K. and Attachaiyawuth, A., 2017. Improvement in self-compacting properties of fresh concrete eliminating large air bubbles using an antifoaming agent. *Journal of Advanced Concrete Technology*. 15: 29-37.
 - 13) Wattanalarmlerd, C. and Ouchi, M., Flowability of fresh mortar in self-compacting concrete using fly ash. *1st International Symposium on Design, Performance and Use of Self-compacting Concrete*. 261-270, 2005.
 - 14) Japanese Industrial Standards, 2009. *Portland cement*. JIS R5210.
 - 15) Japanese Industrial Standards, 2015a. *Fly ash for use in concrete*. JIS A6201.
 - 16) Rath, S., Attachaiyawuth, A. and Ouchi, M., Fineness of air bubbles affected by mixing procedure in mortar in self-compacting concrete.
 - 17) Japanese Industrial Standards, 2015b. Physical testing methods for cement. JIS R5201. –Equivalent to – International Organization for Standardization, 2009. *Cement-Test methods-Determination of strength*. ISO 697.
 - 18) Magura, D.D., 1996. Air void analyzer evaluation. *U.S. Department of Transportation*. Report no. FHWA-SA-96-062.
 - 19) Rath, S., Puthipad, N., Attachaiyawuth, A. and Ouchi, M., 2017. Critical size of entrained air to stability of air volume in mortar of self-compacting concrete at fresh state, *Journal of Advanced Concrete Technology*. 15: 29-37.