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Scientific paper

Critical Size of Entrained Air to Stability of Air Volume in Mortar of Self-Compacting Concrete at Fresh Stage

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Abstract

The objective of this paper is to determine the critical size of air bubbles, which harmfully affects the stability of air in mortar of self-compacting concrete (SCC). Mortar samples produced by different type of mixing procedures and mixing time with various dosage of air-entraining agent (AE) were tested. Air diameter distribution of these mortar samples was measured at fresh stage with air-void analyzer (AVA). With AVA machine, size of air bubble measured is considered as the chord length, which is assumed to be 2/3 of the diameter of air bubble (according to ASTM C 457). It was assumed that air bubbles with over the critical size were easily to escape either by collapsing or floating upward. It was found that instability in volume of air in fresh mortar of SCC was caused mainly by the existence of air bubbles with chord length of over 1000 μm and partially by 500 to 1000 μm due to unification between air bubbles with chord length of less than 1000 μm.

1. Introduction

1.1 Background

Self-compacting concrete (SCC) is defined as concrete having self-compactability, a capacity of concrete to be uniformly filled and compacted into every corner of formwork by its own weight without vibration (JSCE 1999). SCC was developed to facilitate the handling of concrete with no requirement of any vibrator. With high content of cement in the mix-proportion, SCC was mainly welcome for high strength concrete building. According to Okamura (2003), to achieve self-compactability of self-compacting high performance concrete, aggregate content needed to be limited, water to powder ratio was lowered and superplasticizer was employed.

However, the high unit cost has limited the selection of SCC for other general construction, which requires the normal strength concrete. Air-enhanced self-compacting concrete (air-SCC), with lower unit cost than that of the conventional SCC, was developed to ease the handling of the normal strength concrete. To develop air-SCC, the mix-proportion of convention SCC was modified so that the cement content maybe reduced by increasing both the water to cement ratio (W/C) and the fine aggregate content in mortar (s/m). To enable increasing W/C and the fine aggregate content without lowering the level of self-compactability of fresh concrete, the intention to introduce higher volume of air entrainment was conducted. The air volume in air-SCC was initially targeted to be 10%, which corresponds to be around 15% in mortar since the coarse aggregate volume was limited to be 30% in concrete mix-proportion. It was expected that finer air bubbles working as ball bearing mitigating the friction between particles in fresh concrete enable producing air-SCC with lower unit cost and applicable to many targeted construction site. The example of mix-proportion for 1 m^3 of ordinary concrete, conventional SCC and air-SCC is shown in **Table 1**.

Entraining air to a concrete has become no exception for concrete serving in the cold-weather country, according to Lianxiang and Kevin (2005). According to ACI 318, for concrete produced with the nominal maximum size of coarse aggregate of 19 cm, the air content required to achieve the freeze-thaw resistance was 6%, 5% and 3.5% for severe exposure, moderate exposure and mild exposure respectively. However, in SCC, due to the self-compactability, obtaining an adequate air entrainment is a difficult task, according to Khayat (2000, 2002). The necessity for stabilizing the air entrainment was indispensable for air-SCC. The stable air volume ensures the reliability of concrete design as much as the self-compactability of fresh mortar with time passed.

1.2 Objective of research

The objective of this study is to determine the critical size of air bubbles, which harmfully affects the stability

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Table 1 Example of mix-proportion for 1m^3 of ordinary concrete, conventional SCC and air-SCC.

	Air volume (%)	W/C (% by weight)	s/m (% by volume)	(kg/m^3 of concrete volume)			
				Water	Cement	Fine aggregate	Coarse aggregate
Ordinary concrete	5	55	50	181	329	764	1018
Conventional SCC	5	30	40	194	646	713	764
air-SCC	10	45	55	166	369	929	724

of air. In this study, experiments were conducted with fresh mortar produced by different types of mixing procedures. Mortar was chosen instead of concrete since its property is more sensitive when various adjustments of mix-proportion and mixing procedure was attempted to be conducted. Critical size of air bubbles to the stability of air was then determined using air diameter distribution test results of mortar at fresh stage obtained from the air void analyzer (AVA-3000).

2. Mixing procedures and materials

2.1 Mixing procedure for mortar

A total of 24 mortar samples were produced with two different types of mixing procedures as shown in **Fig. 1**. One of the two types of mixing procedures is named here as $A_{(Y)}$ in which Y is the mixing time and was varied by 2 minutes, 4 minutes or 6 minutes. In this mixing procedure, after the fine aggregate and cement were mixed for 30 seconds, water, SP and AE agent were all put together and mixed for Y min(s). The other mixing procedure named here as $B_{(1,Y)}$ in which Y is the mixing time with AE and was varied by 2 minutes, 4 minutes, 6 minutes, 8 minutes or 10 minutes. In this mixing procedure, after the fine aggregate and cement were mixed for 30 seconds, first portion of water and SP was added and mixed for 1 minute. The first portion of water was chosen so that the water to cement ratio of the mixture at this step was 30% by weight. Finally, the other portion of water and AE agent were added and mixed for Y minutes. According to Rath (2015), modifying mixing procedure by introducing superplasticizer (SP) and mixing before adding AE agent was effectively improved the stability of air in fresh mortar of air-SCC. The effect of different mixing procedure on distribution air diameter in mortar tested at hardened stage by linear traverse method can be found in the study of Rath (2016).

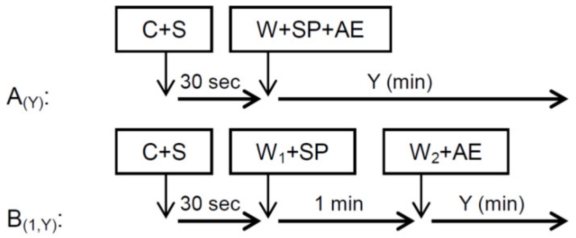

Fig. 1 Mixing procedure for mortar for air diameter distribution tests.

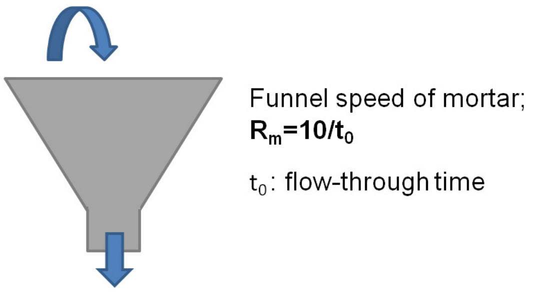

Fig. 2 Test of funnel speed of mortar.

Table 2 Material used for mortar samples in this study.

Cement (C)	Ordinary Portland cement (3.15 g/cm^3)
Fine aggregate (S)	Crushed limestone sand (2.68 g/cm^3, F. M. 2.96)
Superplasticizer (SP)	Polycarboxylic based with viscosity agent
Air-entraining agent (AE)	Alkyl ether-based anionic surfactants *(AE: water concentration is 1:99)*

2.2 Materials and mix-proportion

Cementitious material used in this study was ordinary portland cement only. Fine aggregate was crushed limestone sand. Superplasticizer (SP) was a high-range water-reducing agent working as cement dispersants through elector-steric repulsive force. Electro-steric dispersion is a combination of electrostatic dispersion and steric dispersion. In this study the SP of polycarboxylic based with viscosity agent was employed. Polycarboxylate based type SP are composed of a main carbon chain with carboxylate groups working as electrostatic repulsion and polyethylene oxide side chains working as steric hindrance (Steven *et al.* 2011). Alkyl ether-based AE was employed. All the materials and its properties used for producing mortar of air-SCC are shown in **Table 2**.

After finishing mixing, fresh mortar was tested with flow, funnel speed and air volume with measurement of weight. The funnel speed of mortar can be calculated from the flow-through time of mortar passing the funnel test as shown in **Fig. 2**. The mix-proportion and the properties at initial stage of all mortar samples are shown in **Table 3**.

There are 10 mortar samples produced with mixing procedure A with different mixing time and with variation of dosage AE. There are another 14 mortar samples produced with mixing procedure B with different mixing time and with variation of dosage of AE. It can be observed from the **Table 3** that the dosage of AE of only 0.020% of cement weight can entrain air volume up to 24.0% with the mixing procedure A whereas the dosage of AE up to 0.150% of cement weight can entrain air volume of only 16.2% with the mixing procedure B.

Table 3 Mix-proportion and properties of mortar samples tested with AVA-3000.

N°	Mixing procedure	SP/C (% by weight)	AE/C (% by weight)	Flow (mm)	Funnel speed $=10/t_0$	Air content (%) _Gravitic		Air content (%) _AVA		
								before adjustment		with adjustment
						$A_{initial}$	A_{2h}	$A_{initial}$	$A_{>1\,000\,\mu m}$	$A_{>1\,000\,\mu m}$
1	A_2	1.4	0.005	247	2.48	11.1	7.5	7.6	2.7	6.6
2	A_6	1.2	0.005	260	3.73	13.5	6.0	5.9	2.1	10.2
3	A_2	1.4	0.010	252	2.65	13.0	10.5	12.8	1.9	2.3
4	A_4	1.2	0.010	256	3.24	18.0	13.4	18.1	5.3	5.6
5	A_6	1.2	0.010	251	3.55	21.6	14.5	19.4	6.6	10.3
6	A_2	1.4	0.020	245	2.46	18.3	17.5	19.7	1.5	0.0
7	A_4	1.2	0.020	254	2.89	22.8	22.2	27.4	0.8	0.0
8	A_6	1.2	0.020	258	3.09	24.0	22.9	28.8	0.3	0.0
9	A_2	0.7	0.005	196	2.10	11.6	7.6	9.6	4.8	7.8
10	A_2	0.7	0.010	193	2.19	15.8	10.3	10.5	3.3	10.4
11	$B_{1,2}$	1.1	0.010	236	3.32	6.4	6.9	5.6	0.8	1.7
12	$B_{1,4}$	0.9	0.010	227	3.73	6.8	7.2	6.9	0.7	0.9
13	$B_{1,6}$	0.9	0.010	237	3.88	7.1	7.0	7.1	0.2	0.5
14	$B_{1,2}$	1.1	0.020	262	4.07	6.2	6.7	6.7	0.7	0.3
15	$B_{1,4}$	0.9	0.020	240	3.82	8.4	8.5	7.8	0.0	0.7
16	$B_{1,6}$	0.9	0.020	242	3.83	9.2	9.6	9.4	0.4	0.3
17	$B_{1,10}$	0.9	0.020	234	3.83	10.5	11.0	10.8	1.0	0.6
18	$B_{1,4}$	0.9	0.040	240	3.98	9.4	10.7	11.1	0.5	0.0
19	$B_{1,6}$	0.9	0.040	241	4.20	11.2	11.3	11.7	0.8	0.3
20	$B_{1,8}$	0.9	0.040	238	3.95	12.2	12.6	13.3	0.6	0.0
21	$B_{1,6}$	0.9	0.080	230	4.00	13.4	13.8	13.1	0.7	1.1
22	$B_{1,8}$	0.9	0.080	242	4.20	14.2	14.7	14.1	0.0	0.7
23	$B_{1,10}$	0.9	0.080	234	4.10	15.6	15.8	17.1	0.7	0.0
24	$B_{1,8}$	0.9	0.150	233	4.20	16.2	16.3	18.0	0.8	0.0

The difference in capacity of entraining air volume between mixing procedures A and B was due to the difference in the viscosity. Higher viscosity of mixing procedure A which can be observed from the lower value of funnel speed produced higher volume of air than the mixing procedure of lower viscosity. For example, at the dosage of AE of 0.010% of cement weight and with the mixing time of 6 minutes, mixing procedure A produced air volume of 21.6% whereas the mixing procedure B produced air volume of only 7.1%.

Even though the capacity for entraining air volume with mixing procedure A was higher than B, the stability of air volume in 2 hours was poor in case of mixing procedure A. As in case of dosage of AE of 0.010% of cement weight with the mixing time of 6 minutes, the air loss in case of the mixing procedure A was 7.1% whereas there was only 0.1% of air loss in case of the mixing procedure B. It was obvious that the distribution of air diameter in mortar produced with these mixing procedures was different. Air diameter distribution of mortar at fresh stage was then measured by air-void analyzer (AVA-3000) and the target of this study was to determine the critical size of air bubbles which harmfully cause the dramatic loss in air volume.

2.3 Air-void analyzer (AVA-3000) for measuring air distribution of mortar at fresh stage

(1) Significance of test method

The AVA test method was developed to measure air-void system in concrete or mortar at fresh stage. Before this was invented, it had been hard to fully understand the initial stage of air-void system which is necessary for adjusting target air entrainment. For this study, this test method was beneficial to fully understand the characteristics of the air-void system at fresh stage.

(2) Mechanism of measurement

The mechanism of AVA method is to expel all air bubbles present in a given mortar/concrete sample, collect the air bubbles, and record their quantities and size distribution. The sample is placed in a viscous release liquid and stirred by a magnet to release all air bubbles. With careful control on viscosity of the liquid, the air bubbles could retain their original size without neither coalesce nor disintegrate into smaller bubbles. According to Stokes law, the speed of the air voids rising through the liquids is dependent on their size. The viscosity of the release liquid slows down the initial rise of the bubbles providing a measurable separation in time to the arrival at the top of the column of bubbles of different sizes. The change in buoyancy is measured as a change in weight and is recorded as function of time.

Based on an established empirical calibration, the change in buoyancy can be related to the difference in sizes of air. From this data, air void parameters calculated includes the air content, specific surface and spacing factors. These parameters are calculated to correspond to those that would be obtained from linear traverse measurements on a planar surface of the hardened concrete using the assumptions outlined in ASTM C

457: (1) the average measured chord length is equal 2/3 of the true air void diameter and (2) for the calculation of specific surface and spacing factor, the voids are all of the same size and they are located in lattice points of a regular cubic array.

(3) Equipments and brief description of testing procedure

Equipments for air distribution test by AVA-3000 are shown in **Fig. 3**. Testing equipments include the water tank with its accessories (container with releasing liquid, funnel and brush) which is placed below the AVA base unit and riser column with its accessories (screws, stirrer, piston, buoyancy pan, and windshield).

First of all, the tap water is de-aerated and the releasing liquid is tempered in the water tank at least 24 hours prior to testing air distribution. After one day, the AVA base unit is placed on top of the water tank and connected by means of the connecting device to a computer having AVA application. Then the riser column connected with mortar sample and piston as seen in **Fig. 3** is placed on top of the AVA base unit and fixed with the screws. After that, the magnetic stirrer rod is put into the riser column's bottom. The water removed from the tank is poured to the riser column to a level of about 3 cm below its top edge and all air bubbles are carefully removed from the riser column by the brush. The funnel is filled with releasing liquid and then gently places the liquid into the riser column. Finally, the buoyancy pan is placed on the balance then the windshield is positioned on top of the riser column.

After finishing installation, the piston is gently removed and the mortar sample is inserted into the riser column. Then the AVA testing program is started. The magnetic stirrer will separate the fine aggregate from the cement paste and then the releasing liquid releases the air bubbles allowing bubbles to rise through the riser column and attach itself to the buoyancy pan. The rising of bubbles through riser column is shown in **Fig. 4**. Example of AVA-3000 printout result obtained from the measurement of the mortar sample N° 19 is shown in **Fig. 5**.

The horizontal axis shows the size (chord length) of air bubbles. The vertical axis shows the air volume in cement paste. The test results from AVA also include the air volume in mortar, air volume in paste, specific surface, and spacing factor at the chord length of over 2mm and that of over 1mm. The chord length of 2 mm and 1 mm corresponds to air void diameter 3 mm and 1.5 mm respectively, according to ASTM C 457.

3. Hypothesis

A definition of critical size of air bubbles was defined as shown in the **Fig. 6**. According to Fagerlund (1990), mechanisms of instability of air bubbles included large bubbles move upward the side of formwork by buoyancy force than are lost, during transporting and com-

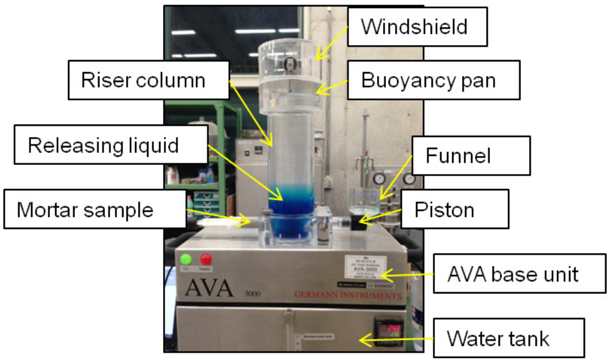

Fig. 3 Test equipment of AVA-3000.

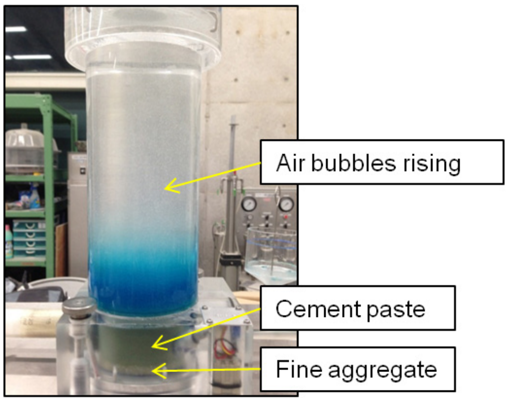

Fig. 4 Rising of air bubbles through rising column during testing with AVA-3000.

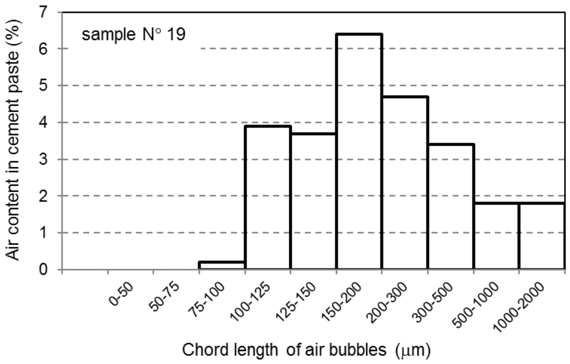

Fig. 5 An example of distribution of diameter of air measured with AVA of mortar sample N° 19.

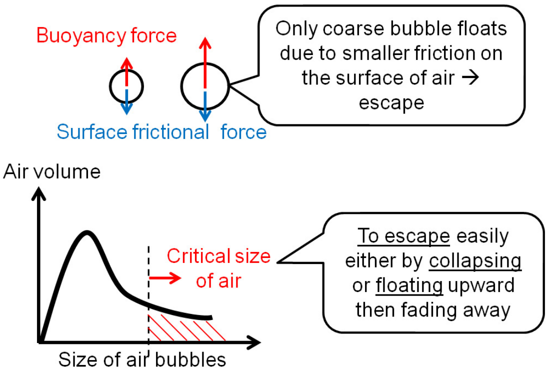

Fig. 6 Definition of the critical size of air bubbles.

pacting of concrete. Another mechanism is the collapse of bubbles by pressure arising from surface tension thus causing air bubbles dissolved in the water. It was assumed that air bubbles with over the critical size were easily to escape either by collapsing or floating upward. The coarse air bubbles having lower internal pressure were easily to collapse in the mixture than the finer air bubbles having higher internal pressure. Furthermore, the coarse air bubbles floated upward faster than the finer ones thus increasing the rate of escaping of air. To improve the stability of air, it is necessary to determine the critical size of air bubbles then minimize the air volume over that critical size.

4. Results and discussion

4.1 Determination of approximate critical size of air bubbles directly from AVA

A relationship between the stability of air and the air content at fresh stage of each chord length range (>100 μm, >300 μm, >500 μm, or >1000 μm) is shown respectively in **Fig. 7**. The increase in air content in 2 hours was observed for some points in this figure. All the cases that air content was slightly increased in 2 hours were the mortar samples produced with mixing procedure B. As mentioned earlier, in the mixing procedure B, SP was added with some portion of water and mixed prior to adding AE with the rest portion of water. The introduction of SP prior to adding AE allowed the particles of mixture to be more dispersed resulting in a lower viscosity mixture as observed by the funnel speed of mortar. When AE was added, the formation rate of large size of air bubbles was lower than the higher viscosity mixture. As the results, mixing procedure B, usually accompanied with higher dosage of AE, produced finer air entrainment system than the mixing procedure A. And the increase in air content in 2 hours for these mortar samples could be the result of coalescence between air bubbles forming the larger ones which have larger volume then the original ones. The unified air bubbles were not escaped or collapsed easily in these cases as the dosage of AE was high enough to stabilize this low air content comparing to the mortar samples produced with mixing procedure A. This means that the

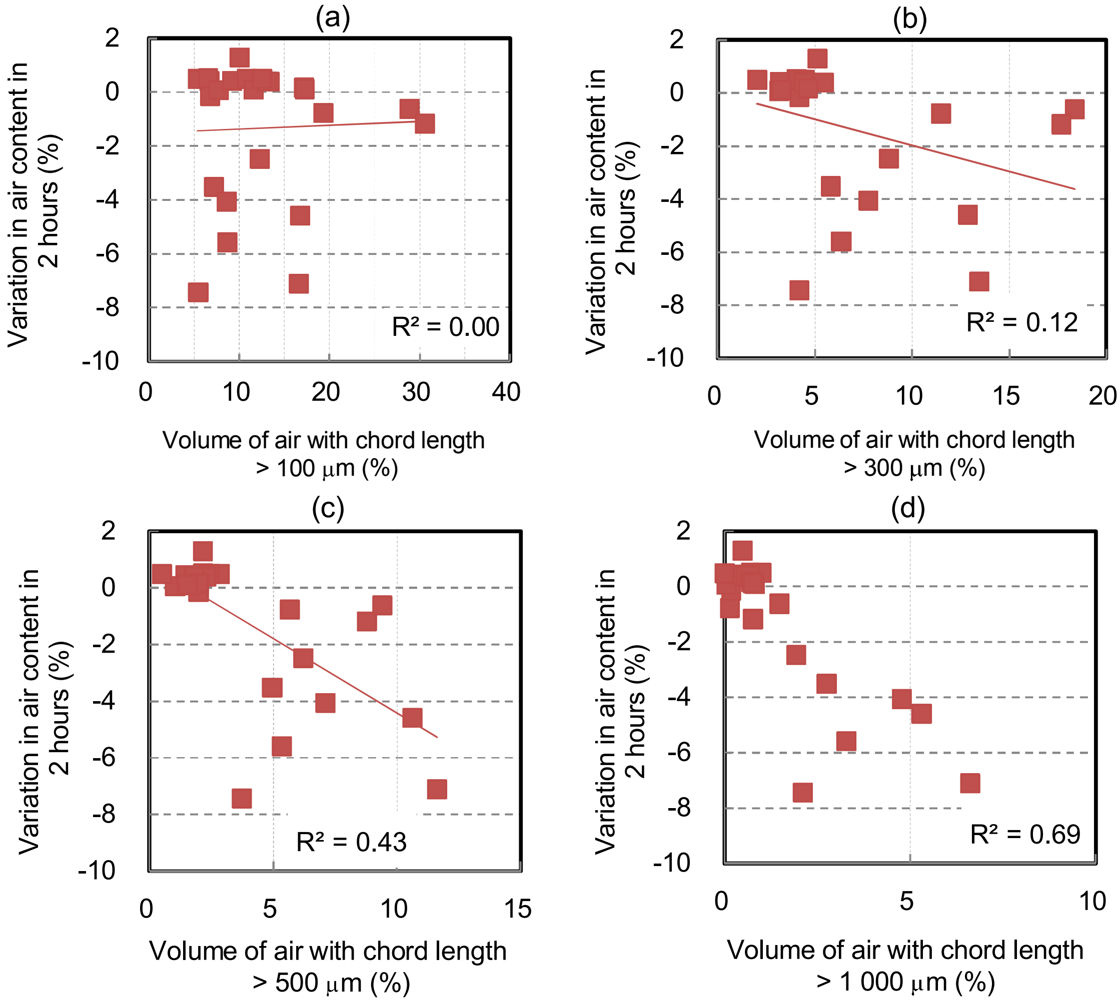

Fig. 7 Relationship between the stability of air in 2 hours and the air volume with chord length (a): >100 μm, (b): >300 μm, (c): >500 μm, and (d): >1 000 μm at fresh stage.

unification between air bubbles occurred in both mixing procedure cases but the increase in air content in 2 hours was rarely occurred with mixing procedure A as the total air content was already high even with a low dosage of AE, comparing to the mixing procedure B case, thus there was not enough amount of AE reserved to stabilize the newly formed bubbles which were usually large in size with poor resistance to collapse. Moreover, the mortar sample was re-mixed for 5 seconds before test which could also generated more air bubbles thus increasing the total air volume which was able to be stabilized in a similar situation as explained earlier. Correlation between the stability of air and the air content at each chord length at fresh stage is shown in **Fig. 8**. It can be seen that the stability of air is more related to the content of coarser air bubbles than the smaller ones. The highest correlation (R^2) of 0.69 may be obtained with the chord length larger than 1000 μm. This result showed that the stability of air influenced mainly by the air volume of bubbles with chord length larger than 1000 μm.

4.2 Adjustment the total coarse air volume

The air volume was also measured with weight method prior to sampling process for air diameter distribution measuring with AVA machine. The correlation between air volume measured with weight and the air volume measured with AVA is shown in **Fig. 9**. The relationship between air volume and the stability of air in 2 hours is shown in **Fig. 10**. From these two figures, it can be observed that there were some cases where the measurement of air volume with AVA was considerably lower than that measured with weight. All the cases that the gap of air content measured with weight and with AVA was high were the mortar samples produced with mixing procedure A at lower dosage of AE (0.005% and 0.010% by cement weight). This explained that the mortar samples with poor stability of air entrainment could easily cause a reduction in air volume during sampling mortar for AVA test. Such a reduction in air content was due to the existence of coarse size air bubbles as it escaped faster than that of the smaller ones and will be more critical to be lost if there were large content of air with that coarse size. On the other hand, there is no considerable decrease in air content during sampling the mortar samples produced with the mixing procedure B as there was only a slight difference between air content measured with weight method and AVA method. To avoid miss-counting the air loss during sampling mortar for AVA test, for all cases, the air content measured with weight method was considered as the exact initial air content of each mortar sample.

From the consideration above, the total air content measured with AVA was adjusted to be equal to that measured with weight method. Since the coarse air bubbles were easily to be disturbed, then the adjustment of air content was made to the coarse air bubbles only. To improve the reliability of the determination on the critical size of air bubbles to the stability of air, adjustment on the total coarse air volume was made as shown in **Fig. 11**. In this assumption, firstly the air bubbles with chord length of larger than 1000μm was considered as the critical value to the air loss based on the highest correlation resulted in **Fig. 8**. Thus, the difference between air volume measured with weight and that measured with AVA was assumed as the coarse air with chord length larger than 1000 μm, which were lost during the sampling of mortar. Secondly, the size of air bubbles changed with time passed and there would be unification between air bubbles occurred before the measurement was done in 2 hours. Thus, by considering that during that time small air bubbles would be unified together forming larger size ones, part of the air volume

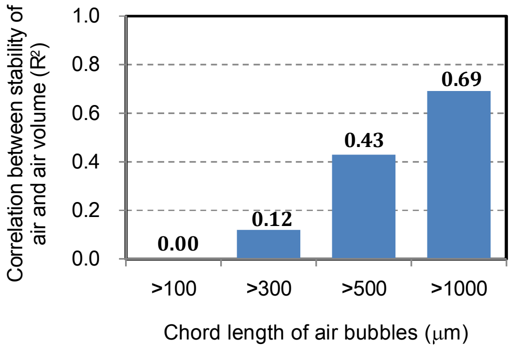

Fig. 8 Correlation between the stability of air in 2 hours and the air volume (measured with AVA) of each chord length range at fresh stage.

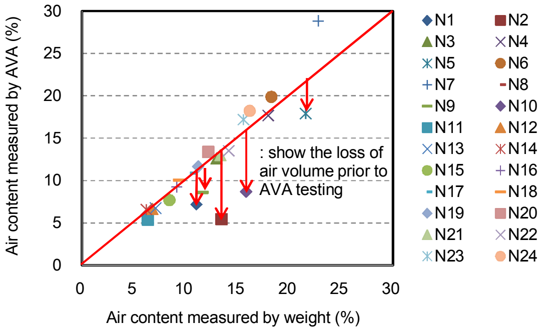

Fig. 9 Correlation air volume measured between with weight and AVA.

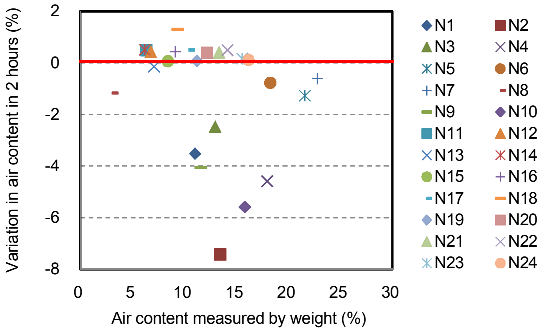

Fig. 10 Relationship between air content and the stability of air in 2 hours.

with chord length of ranging from 500 μm to 1000 μm was also unified to form bubbles with chord length of larger than 1000 μm.

Following the adjustment process mentioned above, the correlation between the air volume of bubbles with chord length of larger than 1000 μm or 500μm to the stability of air is shown in **Fig. 12**. To precisely consider the unification of air bubbles, different share of air volume with chord length of 500 μm to 1000 μm adding to that the air volume with chord length of larger than 1000 μm was analyzed to correlate with the stability of air. The correlation between the stability of air to the air volume of bubbles with chord length of larger than 1000 μm plus partially of chord length 500 μm to 1000 μm is shown in **Fig. 13**. It can be seen that by including 25% of air volume at chord length 500 μm to 1000 μm to the volume of coarse air with chord length of larger than 1000 μm, the highest correlation of 0.94 could be obtained.

Finally, the existence of air volume with chord length of larger than 1000 μm and the partial air volume of chord length 500 μm to 1000 μm due to the unification caused the instability of air in fresh mortar of self-compacting concrete as seen in **Fig. 14**.

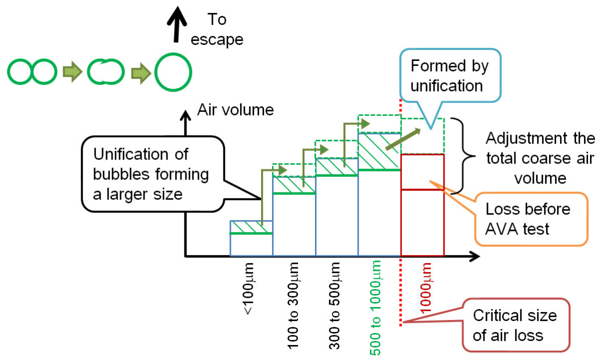

Fig. 11 Adjustment of total coarse air volume considering unification of smaller air bubbles.

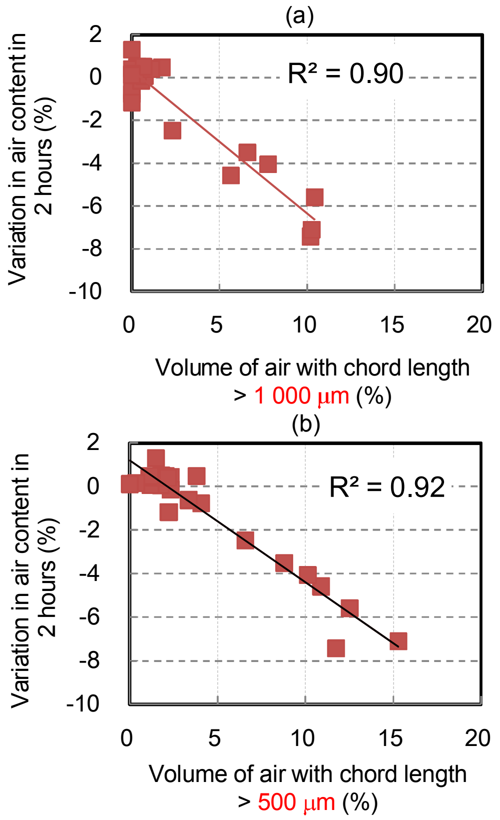

Fig. 12 Correlation between the stability of air and the air volume after adjustment with chord length (a) larger than 1000μm or (b) larger than 500μm.

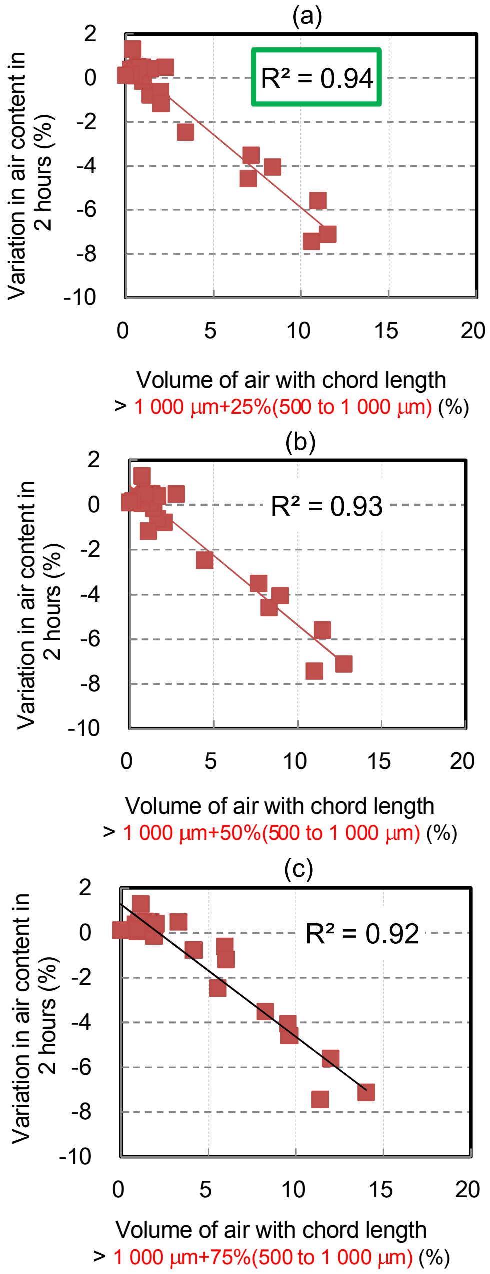

Fig. 13 Correlation between the stability of air and the air volume after adjustment at chord length larger than 1 000 μm plus (a) 25%, (b) 50% or (c) 75% of volume of air with chord length 500 μm to 1 000 μm.

4.3 Repeatability of test results

To strengthen the test results above, three mortar samples with the same mix-proportion as the mortar sample N°1, N°4, and N°5 shown in **Table 3**, were chosen for repeatability test of air content measured with weight method and that measured with AVA. These three samples are the mortar produced with mixing procedure A having a poor air stability as well as a large difference between air content measured with weight and that measured with AVA. The target of this experiment is to show that the air distribution of mortar samples obtained from AVA were reliable even with a single value test for each case. The total entrained air content of these cases may be different to that same mix presented in **Table 3**. This is due to the difference of the fine aggregate material available for the tests which were conducted several months later from the ones presented in **Table 3**.

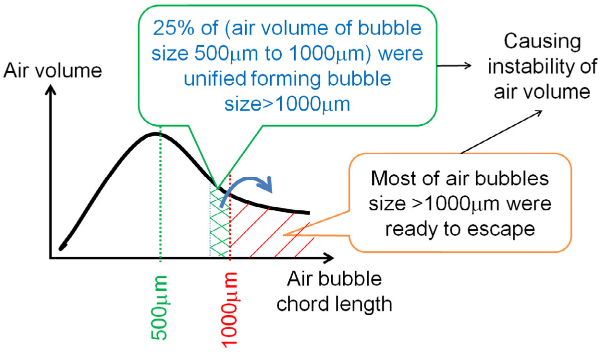

Fig. 14 Summarizing the border of air bubbles size causing instability of air volume.

Air distribution of each mortar mix-proportion produced with three different batches are shown in **Fig. 15**, **Fig. 16** and **Fig. 17** for mortar sample having the same mix-proportion as sample N°1, N°4, and N°5 respectively. It can be observed that the air distribution of mortar samples for each case was considerably repeatable especially the content of air bubble of the finer size. A slight difference in air content was occurred at coarser size of air bubbles but this difference was minimized after adjustment the gap between air content measured with weight method and that measured with AVA. This result ensured the reliability of AVA test results.

5. Conclusion

With the results and discussion in this study, conclusion can be written as following:

(1) The volume of air measured with AVA was often lower than that measured with weight. The gap of the difference in the volume of air between these

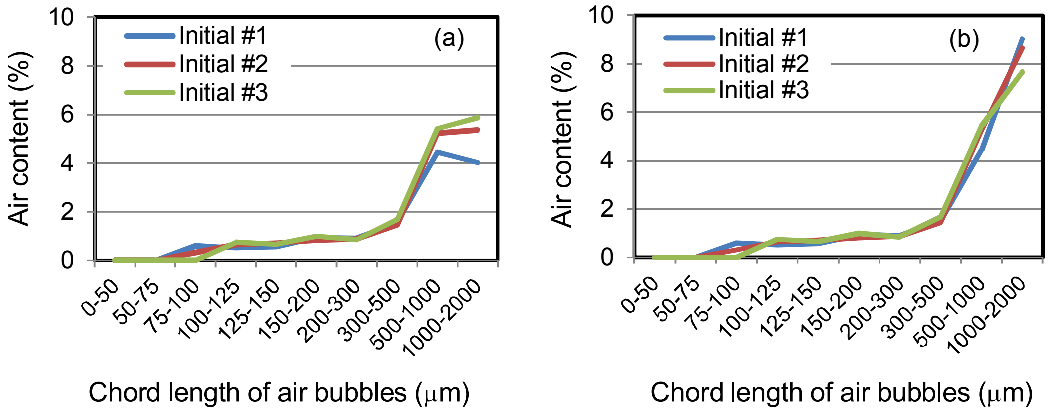

Fig. 15 Air distribution of Sample N°1 repeated by 3 batches; (a) initial air content with no adjustment and (b) initial air content with adjustment to the air content measured with weight method.

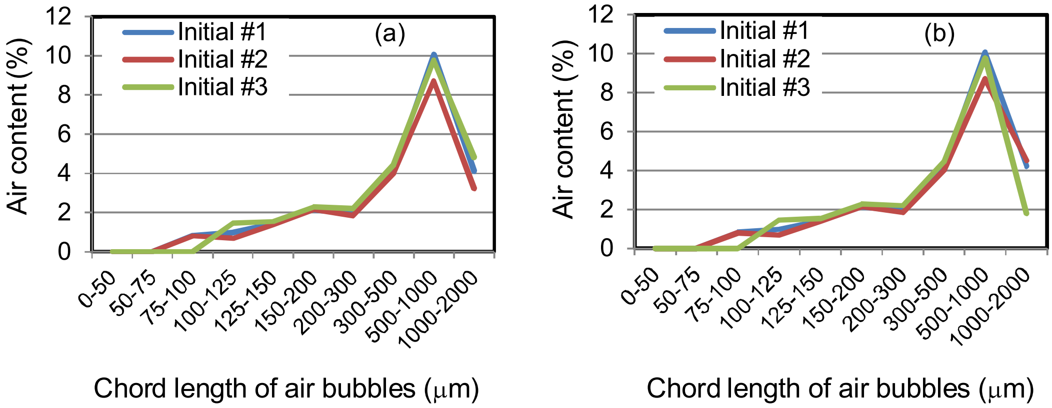

Fig. 16 Air distribution of Sample N°4 repeated by 3 batches; (a) initial air content with no adjustment and (b) initial air content with adjustment to the air content measured with weight method.

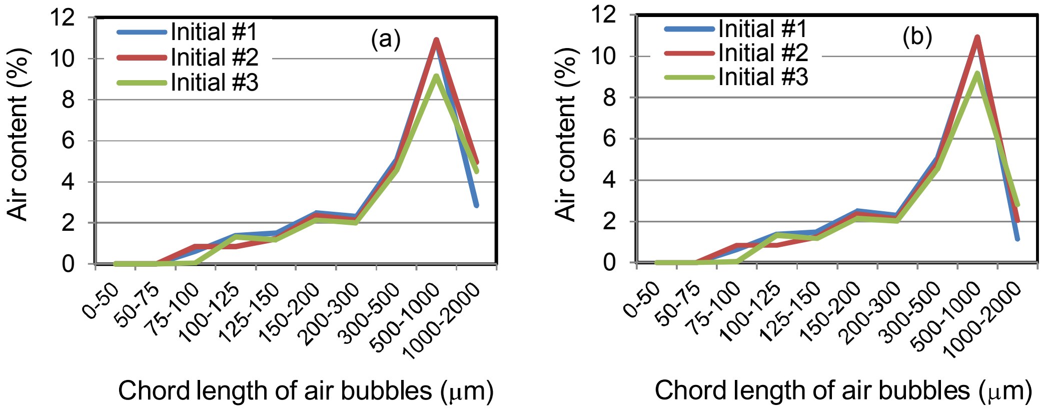

Fig. 17 Air distribution of Sample N°5 repeated by 3 batches; (a) initial air content with no adjustment and (b) initial air content with adjustment to the air content measured with weight method.

measurements was high when the mortar contained higher volume of coarse air.

(2) Adjustment the total coarse air volume of the result obtained with AVA was necessarily needed to avoid miscounting the volume of coarse air loss before AVA test was conducted.

(3) Instability in volume of air in fresh mortar of SCC was caused mainly by the existence of air bubbles with chord length of over 1000 μm. However, due to the unification between air bubbles, the air volume of bubbles with size over 500 μm should be minimized.

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