Effectiveness of air entraining agent and defoamer on the bubble distribution of fresh mortar under different mixing methods

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Abstract. The purpose of this study was to elucidate the distribution of fine bubbles and coarse bubbles in the mortar system of cement-superfine slag powder mortar after multiple mixing of air-entraining agent and defoaming agent. The experimental study used a fresh concrete pore structure analyzer (AVA instrument) to measure the bubble size distribution and related bubble parameters of fresh mortar. The critical size of the bubbles was defined as the size that the air bubbles can maintain the volume stability over time. Based on the time-dependent change in the volume content of the larger bubbles within two hours after mixing, it was found that 500 μm was the critical dimension of bubbles. By adjusting the mortar mixing methods, a lower mortar viscosity was achieved while the total volume of fine and coarse bubbles were also reduced. In addition, when the blending ratio of defoaming agent and air-entraining agent was within the range of 0.50-0.54, the bubble structure of the cement-superfine slag powder mortar had been significantly improved, and more fine bubbles and fewer coarse bubbles had been distributed in the mortar.

1. Introduction
Hardened concrete contains a large amount of voids, which was mainly formed in the process of mixing, vibrating and hardening[1]. The voids in hardened concrete can be divided into several types according to their size, including gel pores, capillary pores and air pores. Among them, air pores were subdivided into well-dispersed spherical entrainment voids with a diameter of less than 1000μm and large irregular entrainment voids with a diameter of more than 1000μm, which were mainly caused by poor compaction and agitation. Obviously, pore structure characteristics, such as air content, size distribution, spacing and pore shape have important influence on the mechanical properties, volume stability and durability of hardened concrete[2-3]. Previous research[4] found that each 1% increase in air content reduces the compressive strength of concrete by 4-6%. Research had shown that to ensure the introduction of a suitable number of small bubbles in concrete, the spacing and distribution of air bubbles was the key to deal with durability issues such as freeze-thaw. The fully introduced air system provided space inside the concrete to ease the swell, which was caused by freezing the internal hydrostatic pressure (volume expansion of about 9%)[5-7].

Air-entraining agent and defoaming agent were also common air bubble distribution control
measures. The air-entraining agent reduced the air-liquid interfacial tension of the gelling material, so that a large number of uniform, stable, and closed bubbles were introduced during the mixing methods, which significantly improved the workability and cohesion of the paste. Defoaming agent eliminate large bubbles in the concrete. The influence of defoaming agent and air-entraining agent alone on the performance of concrete had been studied at home and abroad, but the research on the influence of defoaming agent and air-entraining agent on the composite use was still relatively rare. The compounding of defoaming agent and air-entraining agent could not only improve the overall performance of concrete, but also effectively improved the structure of concrete bubbles, which was of great benefit in enhancing the appearance quality of concrete[8-9]. At the same time, mixing was also one of the effective way to adjust the distribution of concrete bubbles and improve the compactness of concrete. The bubble distribution was adjusted by setting different agitation rates, times and agitator blades. In order to improve the distribution of bubbles, this paper selected three different mixing methods, various proportions of air-entraining agent and defoaming agent to carry out experimental research on the parameters of mortar air content and bubble characteristic parameters. The effect of air-entraining agent and defoaming agent was added to further guide the popularization and application of air-entraining agent and defoaming agent in the concrete.

2. Experimental

2.1. Raw materials and mix proportion

The cement is Beijing Jinyu P·O42.5 with a specific surface area of 342m²/kg. The fly ash is Class I which is produced by the Yuan Baoshan with a fineness (mass fraction) of 11.8%; the fine aggregate is produced by Hebei; grading river sand, according to 10 to 20 heads, 20 to 40 heads, 40 to 70 heads and 70 to 140 heads of four fineness formulated into a fineness modulus of 2.6 II sand, mud content of 0.6% (mass fraction), apparent density is 2640kg/m³; Hebei Sankai Polycarboxylate high-performance water-reducing agent is used, defoaming agent is Japan Tuoyuan QH-AF, air-entraining agent is Clariant; mixing water was from the laboratory. Mix ratios are: cement dosage 400 kg/m³; ultra fine slag powder dosage 80 kg/m³; sand 665 kg/m³; water 145 kg/m³, water reducing agent dosage 1.7 kg/m³.

2.2. Design of recombination ratio of defoaming agent and air-entraining agent

Using an ordinary planetary mortar mixer with non-defoaming agent and air-entraining agent as the control group C1, a high-speed disperser using non-defoaming agent and air-entraining agent as the control group C2, the total amount of the fixed defoaming agent and the air-entraining agent was 0.1 ‰ of the mass of the cementitious material. The compounding ratios of the defoaming agent and the air-entraining agent were respectively 6 ratios of 0.46, 0.48, 0.50, 0.52, 0.54 and 0.56.

2.3. Mixing approach

According to the order of addition of defoaming agent and air-entraining agent and the conditions of mixing equipment, three different mixing processes were designed in this paper, namely MP1, MP2, and MP3. The three mixing processes are shown in Figure 2.

MP1: Planetary mortar mixer is used to add mixing water, water reducing agent, defoaming agent, air-entraining agent, cement and ultra fine slag powder first. Slowly mix for 30 seconds, then adding sand within 30 seconds, for 30 seconds. Then carrying out high-speed mixing for 30 seconds, stop for 15 seconds, then mixing at high speed for 60 seconds.

MP2: Planetary mortar mixer is used to add the water, water reducing agent, defoaming agent, cement and superfine slag powder firstly for 30 seconds. Then adding the sand within 30 seconds, after that mixing for 30 seconds. Finally, adding the air-entraining agent within 15 seconds and mixing for 60 seconds.

MP3: Using a continuously variable high-speed disperser firstly add pre-weighed mixing water, defoaming agent, and water-reducing agent to the mixing tank, and then mix the cement at low speed (200rpm, line speed about 1.0m/s) for 30 seconds. The superfine slag powder is added to the mixing
tank, the mixer is suspended for 15 seconds, the powder adhered to the bottom of the bucket and the wall of the bucket is scraped off, and then the mixing speed is adjusted to a predetermined mixing speed (1500 rpm, line speed of about 7.9 m/s). After the paste is continuously mixed for 90 seconds, the air-entraining agent is added to continue mixing for 60 seconds. The mixed cement paste is added to the mixing barrel of a planetary mortar speed-variable mixer, and sand is mixed. When the compounding ratio is 0.54, the number for 30 seconds.

2.4. Testing method
According to JGJ/T 70-2009 *Basic Performance Test Method for Building Mortar*, the equipment used is SANYO mortar air content measuring instrument. AVA-2000 (Air Void Analyzer) fresh concrete pore structure analyzer was used to test the bubble structure parameters of fresh mortar prepared by different mixing processes. The specific steps were as follows: pre-prepared airless water and standard buffer. Place the liquid in 20°C environment for 12 hours. Turn on the balance switch 30 minutes before the test to ensure the stability of the balance. Connect the instrument and the computer's data cable and turn on the computer. Connect the temperature sensor to the instrument. Pour in a cylindrical container. Into the airless water, use a brush to drive out the air bubbles adhering to the cylinder wall; insert the funnel containing the standard buffer into the bottom of the cylindrical container, let the standard buffer slowly flow into the container, and use a special retriever to take in the fresh mortar. Test and obtain the air bubbles parameters of fresh mortar. The test is shown in Figure 1.

![Figure 1. Set-up of equipment for air void analysis](image)

3. Results and discussion

3.1. Influence on air content of mortar
The air content of mortar mixed with different defoaming agent and air-entraining agent is shown in Fig.2. It shows that fixed content of defoaming agent and air-entraining agent, with the proportion of defoaming agent increases, the air content of mortar tends to gradually decrease. However, there is a slight fluctuation near the compounding ratio 0.50, and the air content of mortar is reduced. It can be seen that under three different mixing processes, the MP2 produces the lowest air content of mortar, followed by the MP3 and MP1. This is because that the process of “first elimination and then introduction” fully exerts the defoaming and foam breaking action of the defoaming agent and at the same time reduces the tension of the gas-liquid interface through the air entraining agent, so that the mortar introduces a large number of fine bubbles during the mixing process. Analyzing the air content of the control groups C1 and C2 in the figure shows that the high-speed dispersion mechanism paste process can significantly reduce the air content of the mortar, while comparing the MP3, it found that the air content of the mortar mixed with defoaming agent and air-entraining agent were overall. Obviously, this is because the compound defoaming agent and the air-entraining agent changed the surface tension of the cement paste, and finally caused a large amount of air entrained during the 30 seconds mixing of the cement paste and sand.
3.2. Influence on spacing factor and specific surface of air bubble

The air bubble spacing factor and specific surface of the mortar mixed with different defoaming agent and air entraining agent are shown in Fig.3. It can be seen from the Fig.3 that the air bubble spacing factor and the specific surface of the mortar in the control group are quite different when no defoaming agent and air entraining agent were added. The air content of mortar obtained by high speed dispersing machine pulping is low, but the bubble structure of the mortar system has obvious deterioration trend. Compared with the three different mixing processes, when the defoaming agent is mixed with the air entraining agent, the air bubble spacing factor and the specific surface of the mortar are improved significantly. When the mortar was prepared by MP1, the structure of the bubble was the best when the ratio of admixture was 0.50. When the mortar was prepared by the MP2, the structure of the bubble was the best when the ratio of admixture was 0.54. This is because that the bubble spacing factor increases with the increase of the air content and the average diameter of the bubbles, and the defoaming agent has a significant effect on reducing the air content of the mortar and reducing the average pore size of the bubbles. The compound use of defoaming agent and air entraining agent can optimize the bubble structure of the mortar. With the increase of defoaming agent ratio, the air bubble spacing factor of mortar gradually decreases, but when the proportion of defoaming agent is higher (0.56), the air bubble spacing factor of mortar increases. This further illustrates that the bubble structure of the mortar is not good when the proportion of defoaming agent is lower or higher. By the comprehensive comparison, it is found that the bubble structure of the mortar is best when the mixing ratio is 0.5 to 0.54 in three different mixing processes.
3.3. Influence on distribution of air void content

The pore size distribution of the mortar under the ratio of different defoaming and air-entraining agents is shown in Figure 4. The fig. 4 shows that when no defoaming agent and air-entraining agent are added, the number of pores with a diameter less than 100 μm in the control group mortar is zero, and the number of pores larger than 500 μm increases significantly. It can be seen that when the defoaming agent and the air-entraining agent are not mixed together, the bubble diameter of the mortar is relatively large, and the bubble structure is not good. When the defoaming agent and the air-entraining agent are mixed, the mortar prepared by the MP1 is found to have higher air content and the number of pores of each bubble is significantly higher than that of the control group C1. When the compounding ratio is 0.50, the number of bubbles with the diameter of 1000 to 2000 μm slightly decreased, but the overall cell pore size distribution did not significantly improve. The mortar prepared by the MP2 has more obvious refinement of the bubble of the mortar. Compared with the control group C1, bubbles with the diameter of less than 100 μm are produced in different mixed proportions, and the number of bubbles with the diameter of 1000-2000 μm is also significantly reduced. When the compounding ratio is 0.54, the number of bubbles less than 100 μm is the largest. When the compounding ratio is 0.50, the number of bubbles from 1000 to 2000 μm are the least. The mortar prepared by the MP3 was compared with the control group C2. It was found that compound defoaming agent and air-entraining agent significantly improved the bubble content of different pore sizes, but did not improve the pore size distribution of the bubbles.
3.4. Influence on stability of air bubbles

3.4.1. Stability of air content

During the mixing process, a large number of bubbles introduced during the mixing process each other during the mixing process and coalesce into larger bubbles. Under the action of buoyancy, the viscous resistance of the paste overcomes the upward discharge, resulting in loss of the air content of mortar, and the longer the static time is, the more likely that the large number of independent bubbles inside meet and coalesce, the greater loss of air content. By compounding defoaming agent and air-entraining agent, the air bubble structure and air bubble distribution in the mortar can be changed, and the viscosity of the paste will be affected. Fig. 5 shows the development of the loss rate of the air content for 1h, 2h, and 3h when the mixing ratio of defoaming agent and air-entraining agent is 0.50. It can be seen from Fig. 4 that the loss rate of air content of mortar prepared by MP1 and MP2 is significantly lower than that of the control group C1. Analysis of the control group C2 found that the paste was prepared by a high-speed disperser and then mixed with sand to prepare mortar. Its initial air content is low, and the loss rate of air content is also lower as the static time increases. On the one hand, defoaming agent can reduce the air content of mortar, on the other hand, defoaming agent can inhibit the formation of unstable large bubbles and enhance the stability of the bubble system. At the same time, when the air content is low, the probability of bubble coalescence is greatly reduced. The loss of air content of the mortar prepared by the MP3 is basically the same as that of the control group C1. It can be seen from the pore diameter of Fig. 4(c) in the previous section that is mainly due to the fact that the high-speed disperser changes the properties of the cement paste and generates the mortar when the paste is prepared. A large number of coarse pores, with the prolonged standing time, making the bubbles can not be stable in the mortar. As can be seen from the figure, the mixing process and compounded defoaming agent and air-entraining agent can both affect the stability of the air content. In this study, how to use different mixing processes to enhance the effect of defoaming agent and air-entraining agent recombination, the focus is to clearly understand the effect of defoaming agent in reducing the surface tension of air-liquid, and to control the large number of fine bubbles introduced by air-entraining agent.
3.4.2. Stability of spacing factor

Fig. 6 shows the relationship between the stability of the bubble spacing factor of the mortar under different mixing processes when the compounding ratio of defoaming agent and air-entraining agent is 0.5. Compared with the control groups C1 and C2, the stability of the bubble spacing factor of the mortar can be significantly improved when the defoaming agent and the air-entraining agent are compounded. In particular, the bubble spacing factor of the mortar prepared by the MP2 and MP3 has the smallest fluctuation, and after 2 hours of standing, the bubble spacing factor slightly decreased. This shows that under the conditions of fixed ratio of defoaming agent and air-entraining agent, different mixing processes can effectively improve the spacing factor of the mortar and improve the stability of the mortar.

In Fig. 6, it is also found that after adopting high speed dispersing mechanism for cement paste preparation of mortar mixed with sand mixing process, control group C2 and MP3 bubble spacing factor of initial value is definitely high, although mixed defoaming agent and air-entraining agent can significantly reduce the bubble spacing factor, but compared to the MP1 and MP2 of mortar bubble spacing factor is still high. Combined with Fig. 4(c), it can be found that the mortar prepared by MP3 has high content of coarse bubbles and poor pore diameter distribution.

3.4.3. Stability of distribution of air void content

Fig. 7 shows the relationship between the pore size distribution stability of mortar bubbles under different mixing processes when the compounding ratio of defoaming agent and air-entraining agent is 0.5. Fig. 7(a) shows the relationship between the pore diameter change of the mortar prepared by the control group C1, MP1, and MP2 for 2 hours when the defoaming agent and the air-entraining agent are mixed. From Fig. 7(a), it can be seen that the pore diameter of the mortar prepared by the MP1 is basically the same as that of the control group C1, while the pore diameter of the mortar prepared by the MP2 is significantly lower than the baseline group C1. On the one hand, this is because the defoaming agent can reduce the air content of the mortar. When the air content is low, the probability of bubble
coalescence escaping is greatly reduced. Defoaming agent can inhibit the formation of unstable large bubbles and enhance the stability of the bubble system. Fig. 7(b) shows the relationship between the pore diameter change of the mortar prepared by the control group C2 and MP3 for 2 hours when the defoaming agent and the air-entraining agent are mixed. Fig. 6(b) shows that the pore diameter of the mortar prepared from the control group C2 did not change significantly with time, whereas the pore diameter of the mortar prepared by the MP3 was significantly more volatile than the control group C2. After 2 hours, the pore diameter stability of the mortar prepared by MP3 deteriorated. This is due to the high air content of the mortar prepared by the MP3 and the presence of more coarse bubbles. The sensitivity of the bubble system is enhanced, and the bubble of the mortar becomes unstable.

![Figure 7. variation of distribution of air void content with different mixing approach](image)

### 4. Conclusion

By testing the air content, bubble spacing factor, specific surface and pore size distribution of mortar, the effects of compounding defoaming agent and air-entraining agent on the bubble structure of mortar under three different mixing processes were studied, and the following conclusions were obtained:

1. The use of an appropriate proportion of defoaming agent and air-entraining agent could effectively increase the air content and stability of the mortar. The method of “eliminate first and then introduce” could effectively control the stability of the air content of mortar. However, there were great differences in the effect of different mixing processes on the “eliminate first and then introduce”.

2. When compounding defoaming agent and air-entraining agent, the spacing factor and specific surface of mortar had improved to varying degrees. Three different mixing processes were comprehensively analyzed and when the ratio of defoaming agent and air-entraining agent was found to be 0.50 to 0.54, the mortar bubble spacing factor was the smallest.

3. The pore size distribution of mortar bubbles was affected by the mixing process and the addition of defoaming agent and air-entraining agent. When the mixing ratio of defoaming agent and air-entraining agent was 0.50 to 0.54, the coarse bubbles of 1000 to 2000μm were the least.

### Acknowledgements

The authors acknowledge the financial support from the National Key Research and Development Program of China (Grant No. 2017YFB0310000) and Science Foundation of China Academy of Railway Science(Grant No. 2017YJ043 and 2018YJ049).

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