7 th International Conference on the Durability of Concrete Structures 20-22 May 2021 University of Jinan, Jinan, Shandong, China

Frost resistance and pore structure properties of mortar containing air entraining agent combined with superabsorbent polymer

Yanqun Xu^{1,2}, Qiang Yuan^{1,2}

1. School of Civil Engineering, Central South University, Changsha, 410075, China 2. National Engineering Research Center of High-Speed Railway Construction Technology, Changsha, 410075, China

ABSTRACT

The air entraining effect of air entraining agent (AEA) is deeply affected by environment, which will further influence its function of improving frost resistance. To solve this problem, a strategy of using AEA combined with superabsorbent polymer(SAP) to improve the frost resistance of mortar was proposed in this study. Mass loss and relative dynamic elastic modulus of mortar were used to characterize frost resistance of mortar. Air void density, spacing factor, air void distribution and air content were measured to analyze its pore structure properties. The results verify that the combined use of AEA and SAP can improve the frost resistance of mortar in comparison with AEA alone. The air content and the porosity of mortar increased under the combination of AEA and SAP.

1. INTRODUCTION

A large number of micro-bubbles are often deliberately introduced in concrete by adding AEA to improve the frost resistance [1]. However, the use of AEA is not an ideal way to improve the frost resistance of cement-based material as the increase of pores will greatly reduce the strength and fracture toughness of materials [2]. In addition, the air entraining effect of AEA is greatly affected by external environmental factors [3-5]. SAP has often been used as an internal curing agent[6] to reduce self-shrinkage, also showing a good performance in improving the frost resistance of concrete [7]. The air voids produced by SAP are less affected by the environment and are more stable in the materials in comparison with AEA. However, when no additional water is added to the mixture, SAP particles will absorb moisture in the

mixture, resulting in a decrease in the w/c ratio and flowability of the mixture [8, 9]. The addition of AEA can improve the flow performance of the mixture mixed with SAP. Therefore, it may be a good choice to improve the frost resistance of cement-based materials by mixing AEA and SAP. However, there is no detailed research about this in the existing literature.

In this study, the mortar was used to explore the influence of different combinations of SAP and AEA on the frost resistance of mortar. Air void density, spacing factor, air void distribution and air content were measured and analyzed to explain the phenomenon. Mass loss and relative dynamic elastic modulus of mortar were used to characterize its frost resistance.

2. MATERIALS AND METHODS

2.1. Materials

The materials used in this study were ordinary

Portland cement(P·O 42.5) , tap water, river sand, air entrained agent of sodium dodecybenzene sulfonate, and superabsorbent polymer. The fineness modulus of sand was 2.47. The particle size distribution of sand and SAP are presented in Fig. 1. The free swelling capacity of SAP is illustrated in Fig. 2, which was measured by gravimetric "tea-bag" method [10-12]. The mixing proportion of mortar is shown in Table 1 and the water to binder ratio of 0.4 was chosen in this study to avoid the effect of self-shrinkage of mortar. It should be noted that the amount of SAP and AEA were calculated by the mass fraction of binder materials.

Figure 1. Particle size distribution of sand and SAP

Figure 2. Absorption and desorption of SAP in water

2.2. Methods

2.2.1. Sample preparation

The mortar mixing process was as follows: The cement, AEA, and SAP powders were mixed in the mixing pot and were stirred for 30s at a low speed (140±5rpm). Subsequently, the sand was added in the pot and was mixed totally with other powers for 30s at a low speed. Then, water was added into the mixing pot to mix them for 1.5min at a low speed. Afterward, stopped for 90s to scrape the mortar on the blade and pot wall into the pot. Finally, the mixture was mixed at a high speed (285±10rpm) for 1.5min. It should be noted that no extra water was added to dissolve SAP powder in this study. All specimens were cured in curing chamber with 98% RH and 20±2℃. Specimens used in the air void property test and BSEM test were cut from mortar cubes with the size of 100mm×100mm×100mm that was cured for 28d.

2.2.2. Air-void properties of hardened mortar

The air voids structure analyzer CABR-457 was used to measure the air void properties of hardened mortar. The preparation process of tested sample was according to [4]. The air void properties were measured and calculated by the gray value difference of tested surface.

2.2.3. Freeze-thaw test

In this study, the rapid freeze-thaw method was used for testing according to GB/T 50082-2009. The mortar cuboid (40mm×40mm×160mm) were used in the rapid freeze-thaw test. The damage of mortar could be showed by its mass loss (ML) and the relative dynamic elastic modulus (RDEM). The initial mass and the fundamental transverse

frequency of specimens were firstly obtained before the rapid freeze-thaw test. The measurements were carried out every 25 freeze-thaw cycles. Three specimens were produced from each group.

3. Results and Discussions

3.1. Air void density and spacing factor

Fig. 3 illustrates that the air void density ascended slightly with the increase of SAP. Specifically, when the mortar contained 0.2wt‰ AEA and the content of SAP increased from 0wt% to 0.3wt%, the air void density increased by 41.46%. However, when SAP remained unchanged in 0.3wt%, and AEA increased from 0 wt‰ to 0.2wt‰, the air void density increased significantly with a rate of 410.7%. It suggested that the air entraining effect of SAP was weaker than that of AEA.

Figure 3. Air void density

Figure 4. Spacing factor

The spacing factor showed in Fig. 4 was calculated based on the mean half-distance between air voids [13]. When the SAP remained as 0.3wt% and that of AEA increased from 0wt‰ to 0.2wt‰, the spacing factor reduced from 0.308mm to 0.122mm.

Figure 5. Pore diameter distribution and air content with the same content of AEA

However, the spacing factor only decreased from 0.162mm to 0.122mm when the SAP increased from 0wt% to 0.3wt% and the content of AEA was

retained as 0.2wt‰. This indicated that both AEA and SAP will reduce the spacing factor, but the spacing factor decreased faster after adding AEA than adding SAP.

3.2. Air void distribution and air content

The air void diameter distribution and air content of mortar with 0.1wt‰ AEA is illustrated in Fig. 5. As showed in Fig. 5(a)-(d), the addition of SAP increased the average pore diameter of mortar and made the distribution of pore size more even. Furthermore, when the content of SAP increased from 0wt% to 0.3wt%, the pores with a diameter larger than 200μm increased gradually from 18.65% to 33.48%, while the pores whose diameters were smaller than 100μm decreased from 67.18% to 48.1%, and the air content increased from 3.4% to 6.4%.

When the SAP content was 0.3wt%, the air void diameter distribution and air content are shown in Fig. 6. The air void diameter of hardened mortar without AEA (G1-3) was mainly concentrated in 10-20μm and the air content was low as 0.53%. After adding AEA, the air voids with diameters greater than 200μm increased, and the air void diameter gradually concentrated in the range of 10-20μm and 500-1000μm. From Figs. 5 and 6, it can be found that the addition of SAP lead to the redistribution of air void size in mortar, whereas the addition of AEA increased the large air void. The air content of G3-3 reached 6.4%, which was more than the sum of the air contents of G1-3 and G3-0, i.e., 3.93%. This indicated that a combined use of AEA and SAP would increase the air content of mortar.

3.3. ML and RDEM after F-T cycles

Fig. 7 shows ML of specimens under different F-T cycles. In Fig. 7(a), when F-T cycles was more than 150 times, ML increased rapidly. Meanwhile, the ML of the specimen exceeded or approached 5% when the F-T cycles reached 250 times. Similar law had been shown in Fig. 7(b). As shown in Fig. 7(c) and (d), ML increased first with the increase of F-T cycles, then fluctuated for a period of time, and finally accelerated. Fig. 8 shows the variation of RDEM with the increase of F-T cycles.

Figure 6. Pore diameter distribution and air content with the same content of SAP

Figure 7. Mass loss of specimens during F-T cycles **Figure 8.** RDEM of specimens during F-T cycles

Figure 9. The BSEM pictures of mortar at 28d (a) 0‰AEA, 0.3%SAP; (b) 0.1‰AEA, 0.3%SAP; (c) 0.1‰AEA,0% SAP; (d) 0.1‰AEA, 0.3%SAP; (e) 0.05‰AEA, 0.2%SAP; (f) 0.2‰AEA, 0%SAP.

When F-T cycles exceeded 250 times, the RDEM of G1 dropped below 60%, implying that the specimens were destroyed (Fig. 8 (a)). From Fig. 8(c) and (d), the RDEM of G3 and G4 decreased rapidly after 175 and 200 F-T cycles, respectively. As presented in Figs. 7 and 8 that when F-T cycles reached 300 times, the ML of G4-0 was about 2.4% while that of G2-2 was about 2.3%. Meanwhile, the RDEM of G4-0 and G2-2 decreased to 84.6% and 83%, respectively. It can be considered that the frost resistance of G4-0 is basically the same as that of G2-2. Compared to adding AEA alone, the frost resistance of mortar can be effectively improved by adding SAP combined with AEA.

3.4. Microstructure

Fig. 9 (a)-(d) shows the magnification of BSEM photos was 500 times, and (e)-(f) are photos magnified by 200 times. From Fig. 9 (a) and (c), the air voids introduced by AEA are regular spherical, while the air voids introduced by SAP are irregular in shape. As shown in Fig. 9 (c), (d), (a) and (b), both AEA and SAP can increase the pore content of mortar. Furthermore, when AEA was mixed with SAP into the mortar, many pore groups and connected pores generated in the mortar, as shown in Fig.9.

4. Conclusions

Both AEA and SAP can increase the air void density of hardened mortar and reduce its spacing factor, but the air entraining effect of AEA is better than that of the SAP. The addition of SAP will increase the average pore diameter and lead to redistribution of air void that the pores with a diameter larger than 200μm increase and those with a diameter less than 100μm reduce. A combined use of AEA and SAP will increase the air content, and can improve the frost resistance of mortar in comparison with AEA alone. This is because on one hand, SAP will decrease the w/c ratio and release retained water in the later cement hydration process. On the other hand, the combined use of AEA and SAP changes the pore structure of mortar, which will improve the frost resistance of mortar.

References

- 1.X.H. Zeng, L. Chen, K.R. Zheng, C.B. Ling, H.S. Zhu, H.C. Liu, P. Wang, K.P. Li, Z.C. Liu, M. Wang, 2020. Electrical resistivity and capillary absorption in mortar with styrene-acrylic emulsion and air-entrained agent: improvement and correlation with pore structure, Constr. Build. Mater., 255.
- 2.J.-C.C. ZDENEK P. B AZ A N T, ARNOLD M. ROSENBERG, JAMES M. GAIDIS, 1988. Mathematical model for freeze-thaw durability of concrete, Journal of the American ceramic society, 71 776-783.
- 3.G.J. Ke, J. Zhang, B. Tian, J.L. Wang, 2020. Characteristic analysis of concrete air entraining agents in different media, Cem. Concr. Res., 135.
- 4.X.H. Zeng, X.L. Lan, H.S. Zhu, G.C. Long, Y.J. Xie, 2021. Investigation on air-voids structure and compressive strength of concrete at low atmospheric pressure, Cem. Concr. Comp., 122.
- 5.J. Holan, J. Novak, P. Muller, R. Stefan, 2020. Experimental investigation of the compressive strength of normal-strength air-entrained concrete at high temperatures, Constr. Build. Mater., 248.
- 6.J.H. Liu, N. Farzadnia, C.J. Shi, 2021. Microstructural and micromechanical characteristics of ultra-high performance concrete with superabsorbent polymer (SAP), Cem. Concr. Res., 149.
- 7.Z.M. He, A.Q. Shen, Y.C. Guo, Z.H. Lyu, D.S. Li, X. Qin, M. Zhao, Z.L. Wang, 2019. Cement-based materials modified with superabsorbent polymers: A review, Constr. Build. Mater., 225 569-590.
- 8.B. Craeye, M. Geirnaert, G. De Schutter, 2011. Super absorbing polymers as an internal curing agent for mitigation of early-age cracking of high-performance concrete bridge decks, Constr. Build. Mater., 25 1-13.
- 9.D. Snoeck, D. Schaubroeck, P. Dubruel, N. De Belie, 2014. Effect of high amounts of superabsorbent polymers and additional water on the workability, microstructure and strength of mortars with a water-to-cement ratio of 0.50, Constr. Build. Mater., 72 148-157.
- 10.C. Schrofl, D. Snoeck, V. Mechtcherine, 2017. A review of characterisation methods for superabsorbent polymer (SAP) samples to be used in cement-based construction materials: report of the RILEM TC 260-RSC, Mater. Struct., 50.
- 11.V. Mechtcherine, E. Secrieru, C. Schrofl, 2015. Effect of superabsorbent polymers (SAPs) on rheological properties of fresh cement-based mortars - Development of yield stress and plastic viscosity over time, Cem. Concr. Res., 67 52-65.
- 12.C. Schrofl, V. Mechtcherine, M. Gorges, 2009. Relation between the molecular structure and the efficiency of superabsorbent polymers (SAP) as concrete admixture to mitigate autogenous shrinkage, Cem. Concr. Res., 42 (2012) 865-873.
- 13.M.o.H.a.U.-R. Development, Chinese Industrial Standard for test methods of long-term performance and durability of ordinary concrete. , Beijing, China.