Dry Shrinkage and Frost Resistance Performance of Cement Stabilized Aggregate Materials

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Abstract. In this paper, the mechanical, dry shrinkage and frost resistance performance of cement stabilized aggregate materials were investigated. Furthermore, the nonlinear analysis between dry shrinkage strain and frost resistance performance was conducted. The results showed that the mechanical performance of cement stabilized aggregate with different cement content showed a linear improving relationship. A higher cement content owns a correspondingly better mechanical performance. With the different moisture content, the dry shrinkage and frost resistance performance of cement stabilized aggregate material showed a similar improving and deteriorating trend. With the increasing moisture content, the dry shrinkage strain and dry shrinkage coefficient were improved and reached to the optimum values, and then started to deteriorate. The optimum dry shrinkage and frost resistance performance were obtained with 6.0% moisture content. The nonlinear relationship between dry shrinkage strain and frost resistance coefficient was gained and the correspondingly optimum dry shrinkage strain and frost resistance was discerned, i.e. 94.3×10-6 and 47.6, respectively. The moisture content significantly influence the pore parameters of prepared samples, whose trend followed those of dry shrinkage and frost resistance performance. The pore size distribution of these composites shifted toward smaller pore size scope with a proper moisture content. In addition, scanning electron micrographs (SEM) showed that the denser microstructure of prepared cement stabilized aggregate materials.

Keywords: cement stabilized aggregate; mechanical performance; frost resistance performance; dry shrinkage; nonlinear relationship; pore parameter; microstructure.

1. Introduction

With the highly development of highway in China, the semi-rigid base asphalt pavement has been considered as a promising candidate in high-level asphalt pavement for its high bearing capacity and low cost [1~4]. However, the crack formation limits the development of semi-rigid based materials, and finally increases the risk of deterioration of the correspondingly mixes [5~8].

In the latest decade, the researchers found that the thorough method to prevent the formation of crack reflection is to decrease the cement content and apply the low dosage cement stabilized aggregate. Yu [9] found that when the cement content is under 3%, the shrinkage coefficient of 28 days is sensitive to cement content. Temperature contraction coefficient of both 7 and 28 days raise with the increasing of cement content and temperature contraction coefficient of 7 and 28 days in high temperature is remarkably bigger than that in common and low temperature. Zeng[10] got that both the non-cracking ultimate temperature drops and the moisture losses decreases with the increase of cement content. The ultimate temperature drops and the moisture losses of frameworking materials are 19.9%~24.3% and 3.6%~6.8% high than those of floating materials, respectively. Motohiro [11] produced the crushed cement-stabilized constrtion sludges (CCSS) and the correspondingly CBR of 8% CCSS changed remarkably by moisture contents. Disfani [12] researched the flexural beam fatigue strength evaluation of crushed brick in cement stabilized recycled concrete aggregates. The cement-stabilized blends with crushed brick as a supplementary material with up to 50% brick content and 3% cement were found to have physical and strength properties, which would comply with road authority requirements.

In this paper, the mechanical, dry shrinkage and frost resistance of cement stabilized aggregate materials with cement content of 3%, 4%, 5%, 6% and 7% were investigated. Further research about the nonlinear relationship between dry shrinkage strain and frost resistance coefficient was analyzed, which provided a scientific basis of design of pavement base. Furthermore, Mercury intrusion porosimetry (MIP) and SEM analysis provided a nano-scale analysis for the influence of moisture content in cement stabilized aggregate materials.

2. Experimental Procedures

2.1 Materials

Ordinary Portland 32.5R cement was used in this study, which produced from Shandong Shanshui Cement Co., Ltd., China; its physical properties are shown in Tab.1. Coarse aggregate was limestone gravel, which provided by Nanjing Quanshui Quarry. The correspondingly gradation was shown in Tab.2.

Table 1. Physical properties of the purchased P·O 42.5R cement

Specific surface area (m^2/kg)	Loss on ignition (%)	Setting time (min)		Flexural strength (MPa)		Compressive strength (MPa)	
		Initial setting	Final setting	3d	28d	3d	28d
330	2.85	215	360	3.9	6.8	26.5	47.1

Table 2. Gradation of coarse aggregate

Gradation	Sieve size/ mm								
	31.5	19	9.5	4.75	2.36	0.6	0.3	0.15	0.075
Rate/ %	100	95.4	72.1	41.6	27.5	17.8	9.5	3.2	1.1

2.2 Preparation and testing

According to the Chinese Standard JTG E51-2009, the optimum moisture content and maximum dry density were confirmed by the method of compaction test of cement stabilized aggregate materials. Then the samples were prepared with 98% compaction degree, the resulted optimum moisture content and maximum dry density. Dry shrinkage test were performed on prism specimen (100×100×400 mm^3) and 7 days unconfined compressive strength (JTG E51 T0805-1994), 90 days indirect tensile strength (JTG E51 T0806-1994) and 90 days elastic modulus (JTG E51 T0852-2009) were all performed on cylindrical specimens with 150 mm height and 150 mm diameter. These tests were all performed on six replicated specimens.

Dry shrinkage tested were shown on the prism specimen with a certain size of 100×100×400 mm^3, which were cured in the standard atmosphere for 7 days. Finally, the specimens were dried and the dry shrinkage were measured till equilibrium conditions existed. The dry shrinkage coefficient was calculated as equation (1).

$$\Delta\varepsilon_d = \frac{\sum \Delta l}{L} \tag{1}$$

where $\sum\Delta l$ is the accumulated dry shrinkage and L is the initial length of prepared sample.

Frost resistance performance were performed on 100×100×400 mm^3 specimens for 180 days' curing. The cycle temperature was set as -20~20 °C, and a whole cycle included a 3 hours' drop temperature and 5 hours' raise temperature procedure.

After 90 days curing, these prepared samples were soaked in ethanol to stop the hydration reaction, and then put in an oven at 70 °C for about 1 day for drying procedure. Four samples were tested by using an automated mercury porosimeter (AUTOPORE IV 9500 series, Micromertics Instrument Corp., USA), with two low-pressure stations plus one high-pressure station, and with a maximum pressure of 33,000 psia for pore size measurements. Moreover, morphology study was performed with an ultra-high resolution field emission scanning electron microscopy (FE SEM)

(NOVA NanoSEM 450, FEI Co. Ltd., USA) at an accelerating voltage of 3 kV with 40~400,000 magnification. Prior to this SEM observation, the prepared samples (1×10×10 mm) were coated an about 20 nm thick Au film layer.

3. Results

3.1 Mechanical performance

The mechanical performance of different mixes with different cement content (3.0%, 4.0%, 5.0%, 6.0% and 7.0%) was shown in Tab.3. With the increasing of cement content, we clearly discerned that the tested mechanical performance was improved, including 7 days unconfined compressive strength, 90 days indirect tensile strength, 90 days elastic modulus and 90 days fracture toughness. More added cement could adhesive the aggregates to formed a mix system, which had a better pore parameter.

Table 3. Mechanical performance of different mixes cement stabilized aggregate materials

	Cement content (%)				
	3.0	4.0	5.0	6.0	7.0
Optimum moisture content (%)	4.1	4.7	5.3	5.6	6.1
Maximum dry density (g/cm^3)	2.25	2.33	2.41	2.46	2.50
7 days unconfined compressive strength (MPa)	3.11	3.57	4.33	4.92	5.56
90 days indirect tensile strength (MPa)	0.62	0.71	0.85	0.96	1.12
90 days elastic modulus (MPa)	696	951	1512	1998	2456
90 days fracture toughness (kN·m$^{-3/2}$)	31.2	37.6	44.5	53.2	59.9

3.2 Dry shrinkage test

The gradation of aggregate among the prepared samples were shown in Tab.2 and the added cement content in the mixes was about 3.0%, 4.0%, 5.0%, 6.0% and 7.0%, respectively. The correspondingly dry shrinkage strain and dry shrinkage coefficient were measured and calculated in Tab.4. With the increasing of cement content in mixes, The correspondingly dry shrinkage strain and dry shrinkage coefficient were increased in a certain extent. Especially the cement content exceed 6%, the dry shrinkage strain and dry shrinkage coefficient were significantly deteriorated. Hence, the cement content in a well prepared cement stabilized aggregate material was controlled between 4% to 5%.

Furthermore, the moisture content in this cement stabilized aggregate system was researched. The specimens (sample 0, 1, 2, 3) , with different moisture content (4%, 5%, 6%, 7%) in a 6% cement content, were measured and analyzed in Tab.5. As is shown in Tab.5, for the prepared samples, with the increasing of moisture content, dry shrinkage strain and dry shrinkage coefficient decreases and reaches the minimum value of about 30.48×10^{-6} and 0.14 at the moisture content of 6.0%, and then starts to grow. A sample contained low moisture content has a relatively loose and dry mixtures, which owns a insufficient cement hydration reaction and low strength, finally leads to a weaker dry shrinkage resistance performance. Moreover, the high moisture content sample has a damp and flabby reaction procedure, which has a larger amount of moisture evaporation and further deteriorated dry shrinkage [13~15]. From the perspective of controlling dry shrinkage, the cement stabilized aggregate material needs to have a 6.0% moisture content.

Table 4. Dry shrinkage strain and dry shrinkage coefficient of cement stabilized aggregate materials with different cement contents

Cement content (%)	Dry shrinkage strain ×10^{-6}	Dry shrinkage coefficient
3.0	36.77	0.13

4.0	54.13	0.22
5.0	63.85	0.28
6.0	82.34	0.38
7.0	102.59	0.55

Table 5. Dry shrinkage strain and dry shrinkage coefficient of cement stabilized aggregate materials with different moisture contents

Sample	Moisture content (%)	Dry shrinkage strain $\times 10^{-6}$	Dry shrinkage coefficient
0	4.0	94.13	0.86
1	5.0	43.53	0.29
2	6.0	30.48	0.14
3	7.0	132.62	0.47

3.3 Frost resistance test

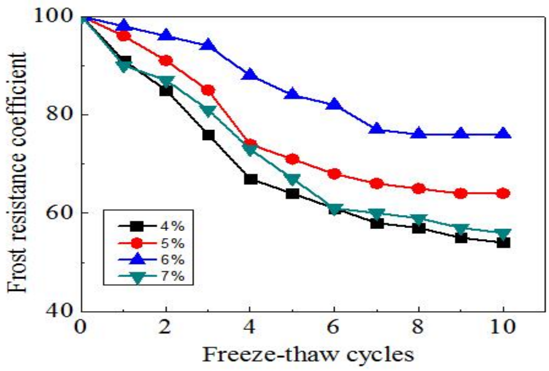

Fig.1 Frost resistance performance of cement stabilized aggregate material with different moisture content

After the procedure of freeze-thaw cycle, the cement stabilized aggregate material was destroyed by the formed expansion pressure and osmotic pressure in the process of water congeals into ice. The detailed frost resistance performance of prepared cement stabilized aggregate material was shown in Fig.1. With the increasing number of freeze-thaw cycles, the frost resistance coefficient was deteriorated, which attributed to the correspondingly pore parameters. In the initial period, the frost resistance coefficient decreased significantly till the eighth freeze-thaw cycle, which reached to a stable condition. After 10 freeze-thaw cycles, the frost resistance coefficient of each mixes was 54%, 64%, 76% and 56%, respectively. The sample with 6.0% moisture content owns the optimum frost resistance coefficient for its appropriate moisture content [16~18].

3.4 Analysis and Discussion

Recorded the dry shrinkage strain *N* and frost resistance coefficient *Y*, we finally got the equation between *N* and *Y* by the method of nonlinear analysis. The nonlinear curve equation of samples were shown as follows:

$$Y = 93.0 + \frac{-3483.2}{60.6 \cdot \sqrt{\frac{\pi}{2}}} \cdot e^{-\frac{(N-95.1)^2}{60.6^2}} \tag{6}$$

Analyzed the calculated equation, we found that with the improving of dry shrinkage strain *N*, the frost resistance coefficient *Y* was deteriorated and then improved, which was shown in Fig.2. When the dry shrinkage strain stayed in 94.3×10^{-6}, the optimum frost resistance coefficient was obtained, was 47.6.

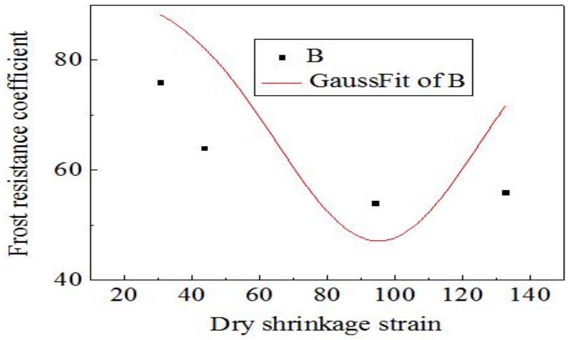

Fig.2 Nonlinear analysis between dry shrinkage strain and frost resistance coefficient

3.5 MIP analysis test

MIP was widely applied in concrete science to characterize the pore parameters, e.g., porosity, pore volume and pore size distribution [19~20]. Fig.3 shows the variation of dV/dlogD pore volume with pore diameter, and the total pore characterization is summarized in Table 6. Obviously, it can be seen that moisture significantly influence the pore parameters of cement stabilized aggregate materials. This trend is similar with those of the dry shrinkage and frost resistance performance. The average pore diameter (APD) of sample with 4.0% moisture content is about 66.9 nm, while the APD values of composites are much lower, about 45.7, 28.3 and 36.7 nm, respectively.

For sample “0”, with the decrease of pore size, dV/dlogD pore volume increases and reaches the maximum value of about 0.253 mL/g at the pore size diameter of 83.8 nm, and then starts to drop. For other mixes, they have the same trends and these maximum values of dV/dlogD pore volume appear in the much finer region of pores, which indicates that the proper moisture content concentrations the pore size. As a kind of porous material, hardened cement stabilized aggregate materials owns a lower property (mechanical and durability properties) for its weaker pore characterization. The moisture content influences the pore characterization of these composites. Ultimately, the dry shrinkage and frost resistance performance of the composites are enhanced.

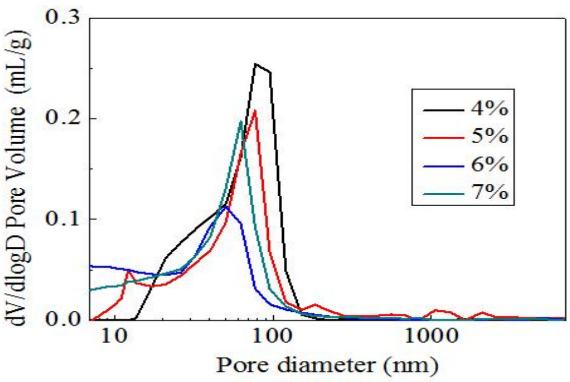

Fig. 3 MIP analysis of pore parameters of cement stabilized aggregate materials with different moisture contents

Table 6. Pore parameters of cement stabilized aggregate materials with different moisture contents

Sample	Moisture content/ (%)	Total intrusion volume/ (mL/g)	Total pore area/ (m^2/g)	Median pore diameter (volume)/ nm	Average pore diameter/ nm	Porosity/ %
0	4.0	0.1433	8.95	83.8	66.9	23.12
1	5.0	0.1123	9.96	63.4	45.7	19.51
2	6.0	0.0867	14.12	42.7	28.3	16.75
3	7.0	0.0994	11.7	57.4	36.7	18.24

3.6 SEM analysis test

It's believed that the higher dry shrinkage and frost resistance performance of these samples attributes to the correspondingly denser microstructure. FE SEM micrographs of sample "0" and "2" at curing periods of 28 days are shown in Fig.4. Compared with the SEM micrograph of sample "0", the microstructure of sample with 6.0% moisture content is more concentrated and less cracks and pores are seen. More C-S-H structure was formed in cement stabilized aggregate material with 6.0% moisture content, which improved the correspondingly performance.

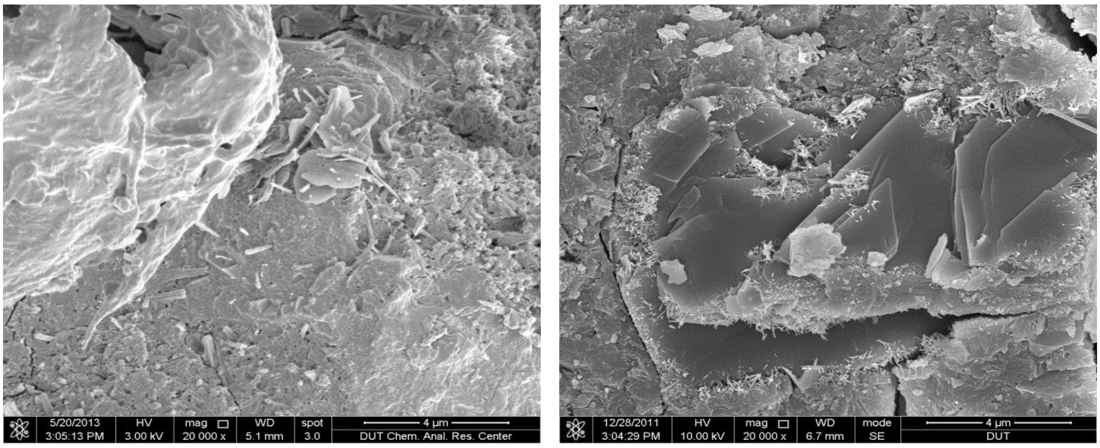

Fig. 4 SEM analysis of cement stabilized aggregate materials

4. Conclusion

In this paper, the mechanical, dry shrinkage and frost resistance performance of cement stabilized aggregate material were investigated. Furthermore, the nonlinear relationship between dry shrinkage strain and frost resistance coefficient were discussed. With 7.0% cement content, the cement stabilized aggregate material has the optimum mechanical and the worst dry shrinkage performance. With different moisture content, the dry shrinkage strain and dry shrinkage coefficient were improved with the increasing moisture content, and then obtained the minimum dry shrinkage with 6.0% moisture content, and finally dropped. Compared each frost resistance performance, the same trend was obtained. The optimum frost resistance performance was obtained within 6.0% moisture content. The nonlinear relationship between dry shrinkage strain and frost resistance coefficient was gained and the correspondingly optimum dry shrinkage strain and frost resistance was discerned, i.e. 94.3×10^{-6} and 47.6, respectively.

Furthermore, the MIP analysis of cement stabilized aggregate materials were obtained and the results showed that the moisture content significantly influence the pore parameters of prepared samples. The sample has a lower porosity and pore diameter with a proper moisture content, i.e. 6.0%. The microstructure of composites shows a nano-scale analysis, which has a concentrated structure with 6.0% moisture content.

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