

The Effect of Initial Water Saturation on Freeze-thaw Damage of Submerged Mortar Materials

SY. Chen, HG. Xiao

School of Civil Engineering, Harbin Institute of Technology, Harbin 150090, China

ABSTRACT

An indispensable condition for the occurrence of the freeze-thaw (F-T) damage in unsaturated cement-based materials is that the specimens absorb water during the F-T process and reach a critical level of water saturation, which is influenced by the initial degree of water saturation (IDWS). However, the fast-freeze method commonly used in the laboratory does not reflect the effect of the IDWS on F-T damage. Thus, this study investigated the effect of different IDWS on the development of F-T damage in specimens to fill the gap in this research area. In this study, compressive strength tests were used to characterize the development of damage, while SEM and MIP tests were used to identify the damage at a microscopic level. A reasonable explanation for the damage development was suggested using the critical saturation theory as well as the negative-temperature pumping effect. According to the experimental and discussed results, damage developed more rapidly in specimens with a low IDWS compared to specimens with a high IDWS. The rapid development of damage was not only related to the hydrostatic pressure during the F-T process, but also the osmotic pressure generated during the entry of external water. As the IDWS decreased, the negative-temperature pumping effect of the specimens became more pronounced. Therefore, a lower IDWS led to a higher osmotic pressure caused by the entry of external water and faster development of damage. This, in return, created more water transfer paths and accelerated the process of reaching the critical level of water saturation, significantly weakening the F-T resistance of the specimen.

1. INTRODUCTION

Durability is an important influencing factor on the service life of concrete members [1]. The frost resistance of concrete has become the second largest durability problem in addition to steel corrosion in the early 20th century [2]. Therefore, it is necessary to study the frost resistance of concrete. Currently, most laboratory studies on the frost resistance of concretes use the freeze-thaw (F-T) process of the fast-freeze method to investigate the effect of the material's own factors on the frost resistance [3,4,5,6,7]. This method requires specimens to be soaked and saturated before F-T process[8], however, it is difficult to reach the saturated state before F-T cycles in the actual engineering environment. Different degrees of water content not only affect the level of the hydrostatic pressure and infiltration pressure of the specimen during F-T process, but also influence the water absorption to a certain extent [9,10,11,12]. According to the theory of the critical degree of saturation, high rate of water absorption accelerates the process of reaching the critical degree and leads to the occurrence of damage [13]. Therefore, the initial degree of water saturation (IDWS) of the specimen has a huge impact on the development of concrete

freeze-thaw damage, but there is a lack of research in this area.

Concrete can be divided into three parts: matrix (mortar), coarse aggregate and interface transition zone [1]. In concrete, coarse aggregates generally have good frost resistance [14], so the coarse aggregates do not preferentially undergo damage during F-T cycles, the mortar and interface transition zone are often important intrinsic factors affecting the frost durability. Therefore, through the change of compressive strength in different cycles, this study focuses on the damage development process of mortar specimens with different initial degree of water saturation (IDWS) under the condition of external water recharge. The macroscopic compressive strength variation was explained from the microscopic point of view by SEM and MIP tests, and the damage development mechanism based on the critical degree of water saturation and negative temperature extraction was proposed.

2. MATERIALS AND METHODS

2.1 Raw materials

The raw materials in this study were cement with a strength class of 42.5, river sand with a maximum particle size of 4.75, tap water, and a polycarboxylate-based superplasticiser (PS). Table 1

demonstrates the chemical composition of the cement.

Table1. Chemical composition of cement (wt%)

CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	MgO	R ₂ O	LOI*(%)
62.3	21.06	5.5	3.92	2.66	1.71	0.47	1.64

*Loss on ignition

2.2 Mixing proportion and specimen preparation

Mortar with a water-cement ratio of 0.4 were used for all tests, and the specific mix proportions were shown in Table 2. The size of the mortar specimen is 70mm×70mm×70mm. The specimens were poured at room temperature (9°C). After curing for 24h, it was demoulded and put into the standard curing room. The test was conducted after 60 days.

To reveal the effect of different IDWS on the development of F-T damage, the specific working conditions in the test are shown in Table 3. Water-saturated specimens in the F-T test were obtained when the specimens were soaked to a stable mass, while dry specimens were obtained when the specimens were dried at 60°C to a stable mass. The other degrees of water saturation are obtained by linear interpolation of the water absorption mass of the saturated specimen and the water loss mass of the dried specimen.

Table 2. Substrate mortar mixture proportions(kg/m³)

cement	water	fine aggregate	PS
488	195	533	1.464

Table 3. Classification of the specimens.

Test	Working condition
F-T cycles	water-saturated (S)
	75% natural water-saturated (SN)
	50% natural water-saturated (DN)
	Dry (D)

2.3 Test method

The temperature change regime of the fast freezing method was used in the F-T test. Compressive strength tests and mass tests were performed every 5 F-T cycles. scanning electron microscopy (SEM) and mercury intrusion porosimetry (MIP) tests were conducted on the surface and center of the specimens before F-T process and after 5 cycles. The results of these tests were used to illustrate the effect of IDWS on internal and external damage under the same F-T cycles.

3. Result and discussion

3.1 Mass loss

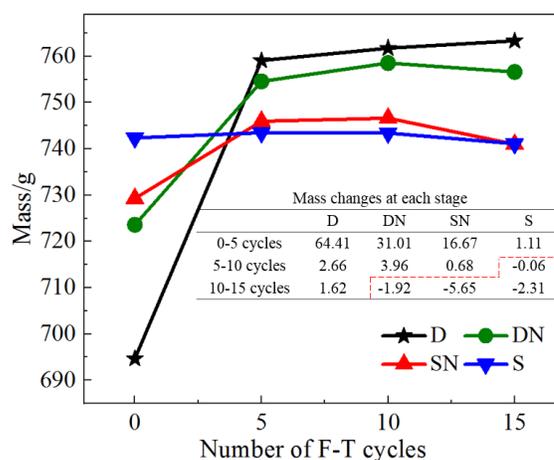


Figure 1. Mass change in terms of F-T cycles

Figure1. shows the mass change of the specimen during F-T process for each working condition. The mass increase in Figure1. represents the water absorption of the specimen during F-T cycles, which is mainly due to the specimen not reaching the critical degree of water saturation before F-T process and the microcrack cracking during F-T cycles to provide more water storage space. The decrease of mass indicates that the water absorption mass in the freeze-thaw process was less than the surface peeling mass.

The magnitude of the increase in the specimens' mass decreased with higher IDWS and more F-T cycles, and it takes more cycles for lower IDWS specimen to begin to lose mass. The higher the water content in the specimen, the closer to the critical degree of water content. According to the critical saturation theory [15], the lower IDWS specimens with water supply were damaged more quickly because they reached the critical degree of water saturation faster. The compressive strength test in section 3.2 can well prove this point.

3.2 Compressive strength

Compressive strength is a physical quantity that is often used in engineering to indicate the change in component damage. The degree of damage of mortar specimens in this study was represented by the relative compressive strength. The relative compressive strength was calculated according to Eq.1. The results are shown in Figure 2., in which lower relative compressive strength means greater damage of the specimen. Figure 2. shows that the relative compressive strength decreased with the increase in the number of F-T cycles and the magnitude decreased as the IDWS of the specimens increased. This indicates that the specimens with high initial water content have less damage when they undergo the same number of F-T cycles.

$$RCS = \frac{f_{ci}}{f_{c0}} \times 100 \quad \text{Eq.1}$$

$$v_{reduction} = \frac{f_{ci} - f_{c(i+5)}}{5} \quad \text{Eq.2}$$

Where RCS denotes the relative compressive strength, f_{ci} and f_{c0} denote the compressive strength before F-T process and after i th F-T cycles. $v_{reduction}$ represents the compressive strength reduction rate.

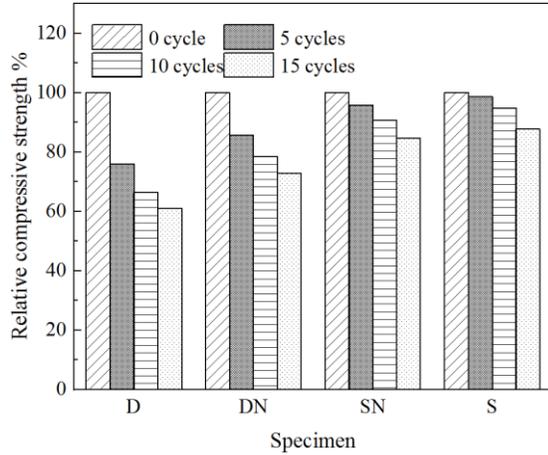


Figure 2. Relative compressive strength change in terms of F-T cycles

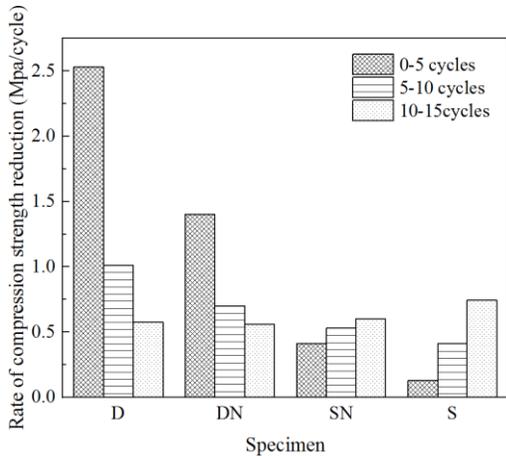


Figure 3. Rate of compression strength reduction change in terms of F-T cycles

In order to compare the development of the damage process, the compressive strength reduction rate was calculated according to Eq. 2 for each test phase, and the results are shown in Figure 3. Figure 3. Exhibits two different patterns of change in compressive strength reduction rates, one such as the D and DN specimens, where the compressive strength loss rate gradually decreased. The other one, such as SN and S specimens, has a gradually increasing rate of compressive strength loss.

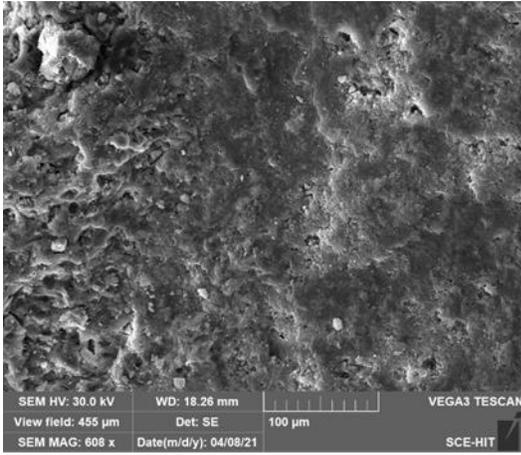
Generally speaking the damage rate in the early stage (0-10 cycles) decreased with the increase of the IDWS of the specimens, while the damage rate in the later stage gradually increased.

The above phenomenon may be due to the fact that in the early stage of freezing and thawing, the moisture in the specimens with high initial saturation degree occupied more space, which hindered the entry of external moisture and delayed the occurrence of damage. Meanwhile, because of the negative temperature suction effect[16,17], the external water quickly entered the specimen with low initial saturation, which not only caused additional osmotic pressure, but also made the specimen quickly reach the critical saturation and then damaged. In the later stage, the osmotic pressure caused by the ingress of the external moisture is reduced as the dry specimens were gradually saturated, and the damage was slowed down compared to the early stage. With the gradual development of microcracks in the early stage of the saturated specimen, the water content inside the specimen increased, and the osmotic and hydrostatic pressures increased during the F-T process, so the damage was accelerated and the compressive strength loss rate increased.

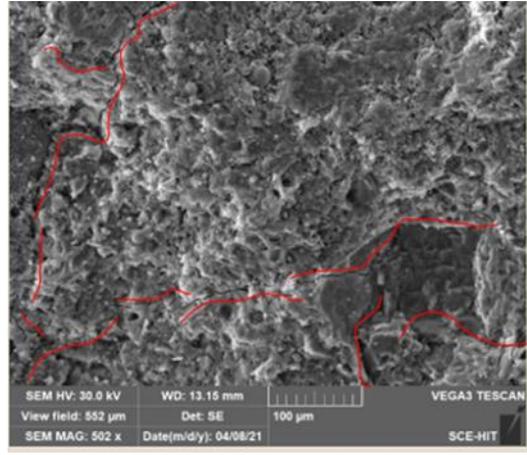
3.3 Microstructure

3.3.1 SEM test

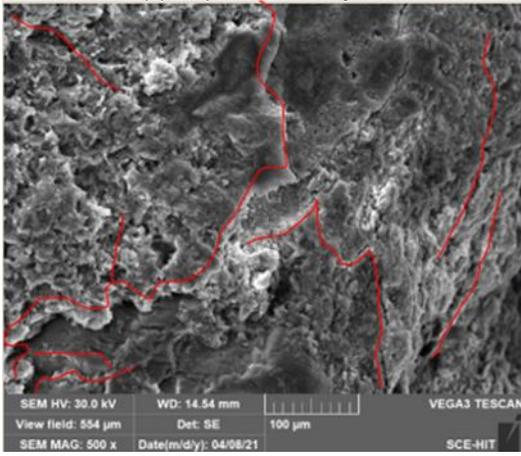
The crack observation of the samples taken from the specimens' surface by electron microscopy was conducted to explain why the low IDWS specimens with moisture supply are more susceptible to damage during freeze-thaw. Figure 4. shows the development of microcracks on the specimen surface before freeze-thaw and after 5 cycles, respectively. Comparing Figure 4.(a)(c)(e)(g), it can be seen that the water saturation or drying treatment of the specimens before freeze-thawing did not cause large differences in the microscopic morphology of the specimens. Comparing Figure 4. (b)(d)(f)(h), the surface microstructure of the specimens is significantly different after being subjected to 5cycles, and the length and width of the surface microcracks cracks increased as the IDWS of the specimens decreased. This may be due to the more obvious negative temperature extraction of the dry specimens, and the rapid entry of external moisture made the surface microstructure locally critically saturated and damaged. The higher the penetration of the microcracks on the specimen surface, the higher the penetration would provide more transmission paths for external water to enter the specimen, which in turn would help freeze-thaw damage occur faster inside the specimen. The evolution of the central pore structure of the specimen in Section 3.3.2 is a good proof of this.



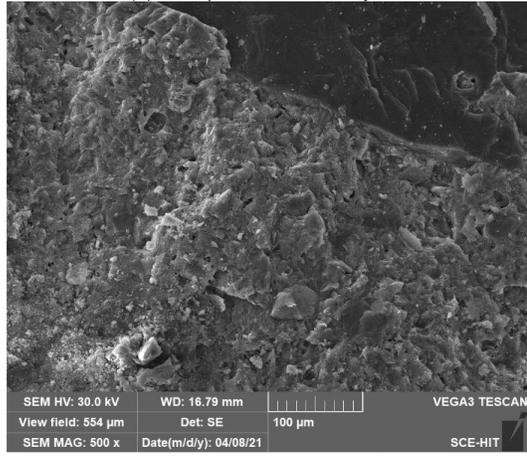
(a) D specimen at 0 cycle.



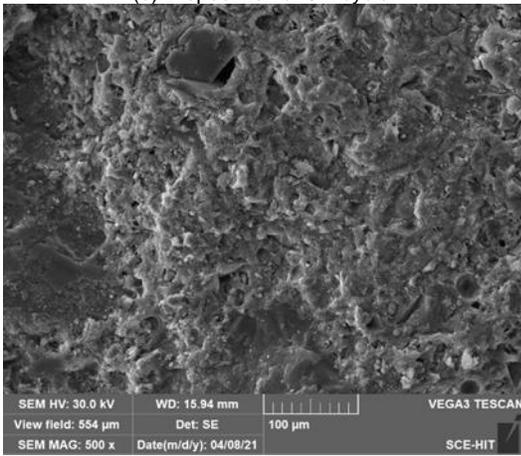
(d) DN specimen at 5th cycle.



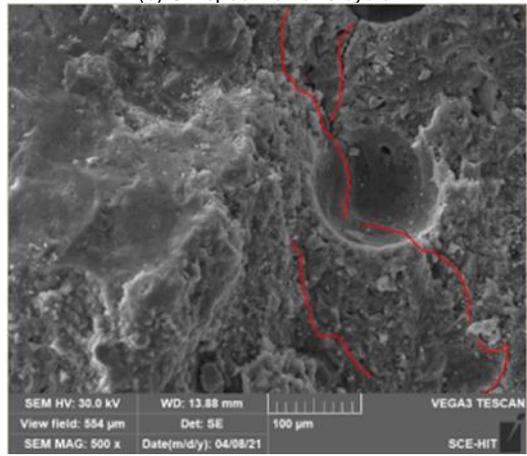
(b) D specimen at 5th cycle.



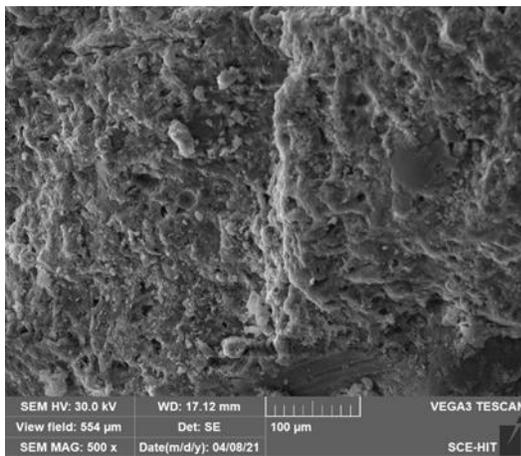
(e) SN specimen at 0 cycle.



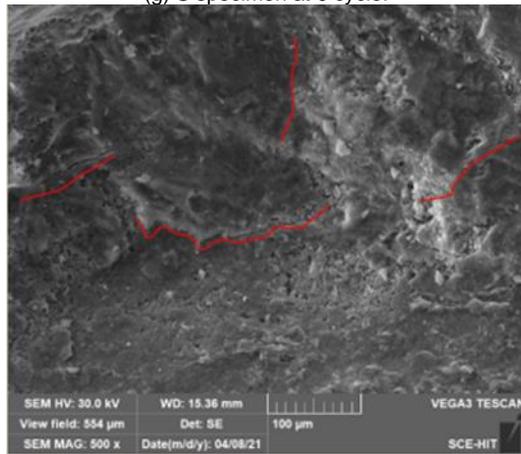
(c) DN specimen at 0 cycle.



(f) SN specimen at 5th cycle.



(g) S specimen at 0 cycle.



(h) S specimen at 5th cycle.

Figure 4. SEM images.

3.3.2 MIP test

The MIP test was used on the pore structure in the center of the specimens to characterize the degree of damage, and the results are shown in Figure 5. The change of pore structure in the center of the specimen was mainly reflected in the pore size distribution rather than the change of porosity[18], and the F-T process made the multi-harm pores in the center of the specimen increase. Figure 5. shows that the increase of multi-harm pores in the center of the specimen decreases before and after the F-T process with the increase of the initial fullness of the specimen. This indicates that as the IDWS of the specimen decreases, it may have a facilitating effect on the entry of external moisture and thus it would be faster for the center of the specimens to reach the critical saturation, causing damage to the microscopic pore structure.

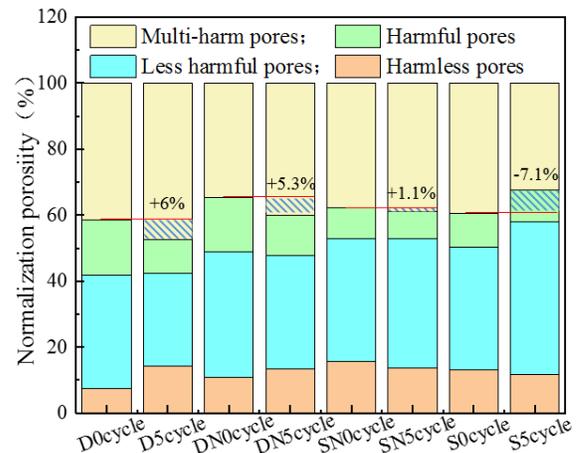


Figure 5. Evolution of pore content with different diameters

4. Conclusions

In this study, the effects of different initial DNWS on the damage development of mortar specimens during F-T cycles were evaluated and the mechanism was explained. The conclusions of this study can be summarized as follows.

Under the condition of external moisture supply, the compressive strength of mortar specimens decreases more significantly during the F-T process as the IDWS decreases.

The reduction rate of compressive strength of mortar specimens showed two opposite patterns according to the different IDWS. The strength reduction rate of the high initial water content specimens showed an increasing trend with the increase of the number of F-T cycles, while the low initial water content specimens showed the opposite pattern.

Under the same number of freeze-thaw cycles, the microcracking of the surface layer of the low-saturation specimens was more obvious. The interconnection of microcracks provided more transmission paths for external moisture to enter the specimens, which in turn made the increase of multi-harmful pores in the center of the low initial saturation specimens more obvious.

Reference

1. Mehta, P.K., Monteiro, P., 2013. Concrete microstructure, properties, and materials[M]. Prentice-Hall.
2. Metha, P.K., 1991. Durability of concrete-fifty years of progress. ACI SP, 126:1-31.
3. Khan, M.I., Siddique, R., 2011. Utilization of silica fume in concrete: Review of durability properties. Resour Conserv Recy, 57: 30-35.
4. Tan, K.F., 2006. Effect of water-cement ratio and mineral admixture on the frost of concrete. Journal of Wuhan University of Technology, 03: 58-60.
5. Knutsson, A., 2010. Freeze/Thaw durability of concrete with fly ash.

6. Shang, H.S., Cao, W.Q., Wang, B., 2014. Effect of fast freeze-thaw cycles on mechanical properties of ordinary-air-entrained concrete. *Sci. World J.*
7. Sabir, B., Kouyiali, K., 1991. Freeze-thaw durability of air-entrained CSF concrete. *Cement and Concrete Composites*, 13(3): 203–208.
8. GB/T 50082-2009, Standard for test methods of long-term performance and durability of ordinary concrete [S]. 2009.
9. Press C. 1995. *Durability of Concrete in Cold Climates*[J]. Crc Press.
10. Powers T.C. 1945. A working hypothesis for further studies of frost resistance of concrete[J]. *Journal of the American Concrete Institute*, 16(4):245-272.
11. Powers T.C, 1953. Helmut R.A. Theory of Volume Changes in Hardened Portland Cement Paste During Freezing[J]. *Highway Research Board Proceedings*.
12. Beaudoin, J.J. Tamtsia, B.T., 2004. Effect of drying methods on microstructural changes in hardened cement paste: an A. C. impedance spectroscopy evaluation. *Journal of advanced concrete technology*, 2(1): 113–120.
13. Li, J., Wu, Z., Shi, C., 2020. Durability of ultra-high performance concrete—A review. *Construction and Building Materials*, 255.
14. Xu, D.H., Xu. M., 2002. *Introduction to Concrete Materials Science* [M]. China Standard Press.
15. Fagerlund, G., 1975. Significance of critical degrees of saturation at freezing of porous and brittle materials. *Aci. Structural. J.*
16. Liu, Z., Hansen, W., F. Wang, Pumping effect to accelerate liquid uptake in concrete and its implications on salt frost durability. *Construction and Building Materials*, 158: 181–188.
17. Smith, S.H., Qiao, C., Suraneni, P., Service-life of concrete in freeze-thaw environments: Critical degree of saturation and calcium oxychloride formation. *Cement and Concrete Research*, 122: 93-106.
18. Jin, S., Zheng, G., Yu J. A micro freeze-thaw damage model of concrete with fractal dimension[J]. *Construction and Building Materials*, 257:119434.