Journal of Asian Concrete Federation
Vol. 5, No. 1, pp. 48-55, June 2019
ISSN 2465-7964 / eISSN 2465-7972
https://doi.org/10.18702/acf.2019.06.30.48

Technical Paper

The critical freeze-thaw cycle considering moisture content increase in the accelerated freeze-thaw test

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(Received January 7, 2019; Revised May 10, 2019; Accepted June 11, 2019; Published online June 30, 2019)

Abstract: In the paper, specimens with 2 kinds of water to cement ratios were conducted with the accelerated freeze-thaw test and the critical degree of saturation test. In the accelerated freeze-thaw test, the trend of the relative dynamic modulus of elasticity (RDM) can be divided into two parts. In the first part, RDM decreased slightly, while it dropped significantly in the next part. It is believed that with the conduction of the freeze-thaw, the mass moisture content increases. Consequently, severe deterioration would occur when the critical mass moisture content is reached. The freeze-thaw cycle where RDM decreased significantly is defined as N_f. The mass moisture content (W_{cr}) at N_f is calculated and compared with that of the critical moisture degree (S_{cr}). According to the results, W_{cr} almost equals to the mass moisture content of S_{cr}. Besides, no clear relationship can be found out between the durability factor DF in the accelerated freeze-thaw test and T_{pl} calculated from the critical degree of saturation test. However, N_f is in good accordance with DF. The freeze-thaw function in the real environment can be converted into a portion of the new defined evaluation criterion N_f to evaluate the situation of the deterioration by the real environment.

Keywords: Mass moisture content, relative dynamic modulus of elasticity, critical degree of saturation, freeze-thaw.

1. Introduction

Concrete frost resistance can be evaluated by both the accelerated freeze-thaw test according to JIS A 1148 and the critical degree of saturation test based on the RILEM CDC3, respectively. However, in some cases, different frost resistance results have been reported among the two methods even with the same concrete [1]. Moreover, the relationship between the accelerated freeze-thaw test and the critical degree of saturation test is also not clear. The evaluation criterions calculated by the two test methods can only be used to compare the frost resistance of different concrete materials. This paper aims at clarifying the relationship between the two frost resistance evaluation methods and figuring out an appropriate and universal concrete frost resistance evaluation criterion to evaluate its frost resistance in the actual environment. Besides, different drying conditions in the actual environment may affect the frost resistance of concrete in different ways. The frost resistance can be enhanced by adequate drying [2,3]. However, on the other hand, the dry-moisture repetition in real environment is reported to degrade concrete frost resistance [4]. By adopting different drying conditions during the curing period, the concrete even in the same batch can have different frost resistance. Therefore, it is important to figure out the effect of different drying conditions on changing concrete frost resistance. It will also help to understand how the real environment affect the concrete frost resistance.

Three different kinds of concrete specimens with different water to cement ratios were fabricated and cured in water for 2 weeks. The specimens suffered various drying conditions to reach different frost resistance. Then the accelerated freeze-thaw test and the critical degree of saturation test were conducted, respectively. The parameter of the non-destructive method, that is, the relative dynamic modulus of elasticity (RDM), of the specimens changing with freeze-thaw cycles was investigated in this experiment. The number of cycle where the RDM decreased dramatically in the accelerated freeze-thaw test is defined as the critical freeze-thaw cycle N_f. Besides, the moisture content at the N_f has

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been compared with the mass moisture content of the critical degree of saturation (S_{cr}) in the critical degree of saturation test. In addition, the conventional concrete frost resistance criterion DF calculated by the accelerated freeze-thaw test and the T_{pl} calculated by the critical degree of saturation test have been compared with the novel defined criterion N_f.

2. Experimental Outline

2.1 Experimental plan

Table 1 exhibits the experimental plan. The specimens with different water to cement ratios and air contents were cast. All the specimens were cured in tap water for 2 weeks. Two drying conditions, drying condition 1 (50℃ drying for 1 week) and drying condition 2 (20℃ drying for 2 weeks) have been applied to the specimens. According to the results by Baba [3], drying condition 1 may degrade the frost resistance of W/C35%-1%, while condition 2 may improve the frost resistance of W/C55%-1% and 4.5% specimens. The results can also be used to verify the correctness of the drying effect on frost resistance in Baba's paper [3]. The universal application of the N_f can be proved not only in the specimens with better frost resistance but also with worse frost resistance.

2.2 Specimen preparation and mix proportion

The mix proportion is listed in Table 2. Type 1 Portland cement was used as the cementitious material and fine aggregate was silica sand with the fineness modulus of 2.68. Coarse aggregate was the crushed stone with the maximum dimension 20mm. Water to cement ratio (W/C) 35% and 55%, were adopted in this experiment. In addition, to set up different frost resistances in the W/C55% specimens, air-entraining agent and defoamer were used to obtain the air contents 1% and 4.5%, respectively. In the specimen of W/C35%, the air content 1% was reached by using the defoamer. Besides, to guarantee the workability of W/C35%-1% specimens, the superplasticizer with a dosage of 0.47g/kg was also used. Since the amount of concrete in each type was way much for the concrete mixer, concrete mixing has been divided into two batches.

For the mix denotation (e.g. 35-1-D/N1), the first two values represent the water to cement ratio and the air content, respectively. D1 means specimen 1 experienced 20℃ drying while N1 represents the specimen 1 without drying.

Table 1 – Experimental plan

W/C (%)	Air Content (%)	Drying condition	Experimental Method	Dimension (mm)
35	1	1 week50℃	The accelerated freeze-thaw test (JIS A 1148 Method A)	75×75×400
		No drying		
55	1 4.5	2 weeks 20℃	The critical degree of saturation test (RILEM CDC3)	Scr:100φ×200 Scap:100φ×30
		No drying		

Table 2 – Mix proportion of concrete

W/C (%)	Target Air Content (%)	Fine Aggregate Ratio (%)	Unit Amount (kg/m^3)				Admixture* (c×%)			Actual Air content (%)	Slump (cm)
			W	C	S	G	303A	404	SP8SVX2		
35	1	46.0	165	471	827	978		0.011	0.47	①1.3* ②1.9*	①23.0 ②22.5
55	1	49.9	180	327	936	949		0.05		①1.2 ②1.1	①17.5 ②20.5
	4.5	47.2	172	313	843	949	①0.014 ②0.010			①4.5 ②4.9	①21.5 ②22.5

2.3 Test procedure

2.3.1 The accelerated freeze-thaw test

The fresh concrete was put into the prismatic molds with a size of 75mm×75mm×400mm and the cylindrical molds with a dimension of 100mm×200mm, respectively. Then covered the mold with the preservative film and stored the specimens in a room with constant temperature and humidity (20℃, RH 60%) for one day. The specimens were removed from the mold on the second day and submerged in tap water for another 2 weeks. For the prism specimens, half of the W/C 35%-1% specimens were then dried at 50℃ for 1 week while the other half were conducted with the accelerated freeze-thaw test immediately. A similar procedure was also performed to the W/C 55%-1% and -4.5% specimens, where half of each type were dried at 20℃ in a temperature-controlled room with the temperature 20℃ and the relative humidity 60% for 2 weeks and the other half were conducted with the accelerated freeze-thaw test immediately.

The accelerated freeze-thaw test was conducted according to the Japanese specification named of JIS A 1148 method A. The temperature of the specimen was set between -18 and 5℃. The length change, the weight in the air and the water, the RDM were measured at suitable intervals. The freeze-thaw test was ceased when the RDM fell to 60% or 300 cycles, whichever came earlier, was reached. RDM is widely used to evaluate concrete frost resistance [5-8]. Calculate the numerical values of relative dynamic modulus of elasticity RDM as follows:

$$P = \left(E_1^2 / E^2 \right) \times 100\% \tag{1}$$

P: relative dynamic modulus of elasticity after c cycles of freeze-thaw, percent
E_1: dynamic modulus of elasticity after c cycles of freeze-thaw (Hz)
E: dynamic modulus of elasticity at 0 cycle (Hz)

The formula Eq. 2 shows the calculation of DF.

$$DF = \frac{P \times N}{M} \tag{2}$$

P: the RDM at N cycles
N: number of cycles at which P reaches the 60% or 300 cycles, whichever is less
M: 300 cycles

All the specimens were then dried in an oven at 105℃ to the constant weight after the freeze-thaw test. The densities of different types of concrete were then calculated.

2.3.2 The critical degree of saturation test

The critical degree of saturation test is conducted according to the RILEM CDC3. In the critical degree of saturation test, it is believed that a critical degree of saturation (S_{cr}) exists [9-11]. Once the moisture content of the specimen exceeds the S_{cr}, concrete will be destroyed by frost. The critical degree of saturation test has been divided into two parts. The S_{cr} is determined by a test in which the cylindrical specimens were sealed with different moisture degrees and then conducted with freeze-thaw test for more than 6 cycles. RDM is measured before and after the freeze-thaw test. The S_{cr} is defined as the moisture degree where RDM decreases rapidly after the freeze-thaw cycles.

The other test is the water absorption test by the bottom surface at room temperature. The water absorption ability is measured and the potential capillary degree of saturation (Scap) at room temperature has also been calculated. In this test, all the samples with a dimension of φ100×30mm were cut from the cylindrical specimens which had suffered from the same drying processes. The samples were then dried at 50℃ for 3 days before commencing the test. Then, the samples were set in a stainless container filled with water. A preservative film was covered upon the samples to prevent water from evaporating. The samples were taken out to measure the weights in the air at suitable intervals and the moisture absorption curve can be drawn by the weight change.

The time (T_{pl}) when the Scap arrives at the S_{cr} is defined as the concrete frost resistance in the critical degree of saturation test [12]. When the T_{pl} is large, it is thought that concrete has excellent frost resistance. Therefore, the service life of concrete can be evaluated by T_{pl}.

3. Experiment Results

3.1 The accelerated freeze-thaw test

In the critical degree of saturation test, the specimens have been artificially set to different moisture contents. Once the moisture content exceeds the S_{cr}, specimens will be damaged by freeze-thaw dramatically. In the accelerated freeze-thaw test, with the increase of the freeze-thaw cycles, water will be pushed into the specimens and thus the moisture content will also increase gradually. Therefore, similar to the critical degree of saturation test, a significant decline in the RDM, which represents the deterioration of concrete by freezing and thawing, should also occur when the specimen moisture content reaches to the S_{cr} in the accelerated freeze-thaw test. The cycle at the nick point is defined as N_f in this paper. To clarify the relationship between the two test methods, the mass moisture contents (M_{cr}) of the S_{cr} in the critical degree of saturation test and the moisture content during the accelerated freeze-thaw test have been calculated, respectively. Especially, the moisture content at the start and the end of the freeze-thaw cycles and the moisture content where the RDM drops obviously (W_{cr}) have also been compared with M_{cr}.

The M_{cr} can be calculated by Eq. 3 below.

$$M_{cr} = \frac{m_{cr} - V \times \rho_{105°C}}{V \times \rho_{105°C}} \tag{3}$$

M_{cr}: the weight moisture content of S_{cr}
m_{cr}: the weight of specimen at the S_{cr}
V: the volume of the specimen
$\rho_{105°C}$: the oven dry density of the specimen

The mass moisture content (W_N) in the accelerated freeze-thaw test is determined by Eq. 4.

$$W_N = \frac{m_{N,air} - (m_{N,air} - m_{N,water}) \times \rho_{105°C}}{(m_{N,air} - m_{N,water}) \times \rho_{105°C}} \times 100\% \tag{4}$$

W_N: the mass moisture content at N cycle
$m_{N,air}$: specimen weight in the air at N cycle
$m_{N,water}$: specimen weight in the water at N cycle

$\rho_{105℃}$: the oven dry density of the specimen

The oven-dry density $\rho_{105℃}$ of the specimen can be calculated by the formula below.

$$\rho_{105°C} = \frac{m_{105°C,air}}{m_{105°C,air} - m_{105°C,water}} \tag{5}$$

$m_{105℃,air}$: the oven dry weight in the air

$m_{105℃,water}$: the oven dry weight in the water

3.1.1 Results of W/C35%-1% specimens

Fig. 1 to 3 exhibit the change of length, RDM and the mass moisture content of W/C35%-1% specimens, respectively. For the specimens without drying, the length change remains the same before the 46th cycles while a significant increase can be figured out after that. Besides, as can be seen from the change of RDM, a nick point appears at the 54th cycles for the specimens without drying. The RDM decreases slightly before the 54th cycles, while it drops significantly afterward. Besides, the curve of mass moisture content also shows a similar trend and appears a nick point at the same 54th cycles. Similar to the critical degree of saturation test, the nick point where RDM decreases dramatically is regarded as concrete damaged by freeze-thaw and the cycle at the nick point is defined as N_f.

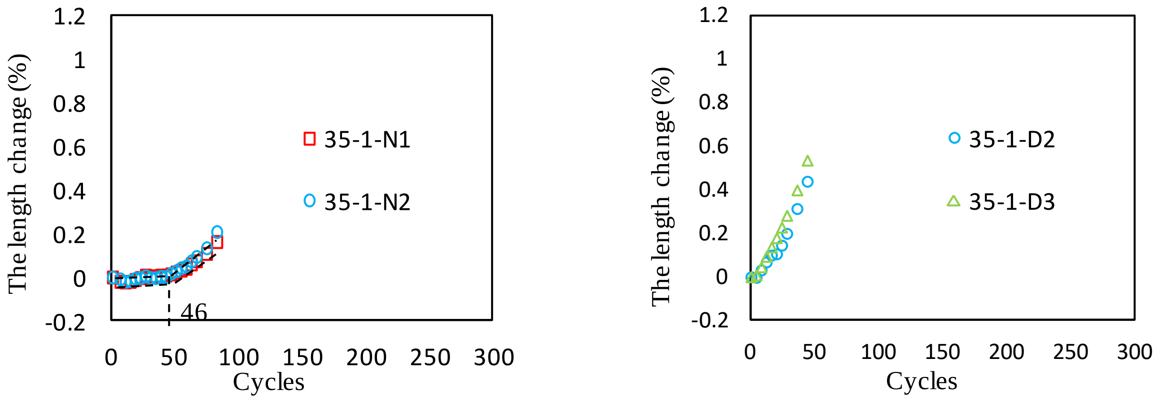

Fig. 1 – The length change in the W/C35%-1%

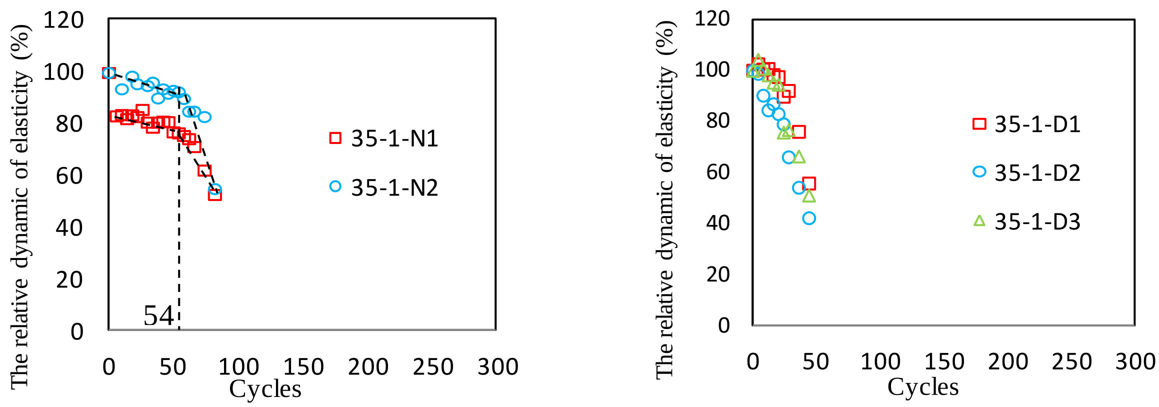

Fig. 2 – The change of RDM in the W/C35%-1%

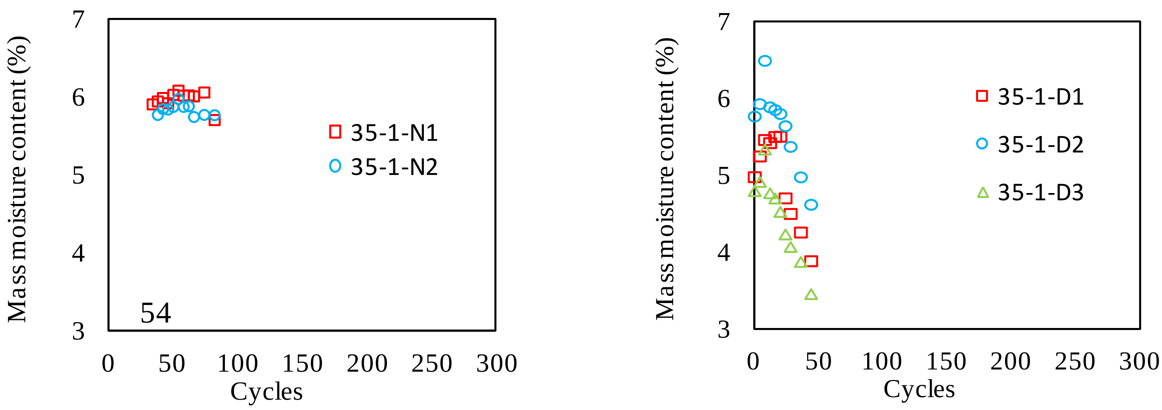

Fig. 3 – The change of mass moisture content in the W/C35%-1%

Table 3 – The mass moisture content in the W/C 35%-1% specimens

Specimen	the critical degree of saturation test	Moisture content in the accelerated freeze-thaw test		
		0 cycle	Nf	Final cycle
	M_{cr}	W_0	W_{cr}	W_{final}
35-1-N	6.10	5.87	6.06(54cycles)	5.73
35-1-D		7.35	—	6.48

With the increase of the freeze-thaw cycles, the external moisture would penetrate the specimens, which will result in an increase of the mass moisture content. Consequently, when the mass moisture content reaches to the maximum, the specimens are damaged by freeze-thaw fiercely. Besides, due to the emergence of large numbers of cracks, the specimen may peel off, and consequently, the mass moisture content may decrease. As can be seen from Fig. 2 and 3, both the nick point in RDM and the maximum mass moisture content occurs at the same cycle.

Table 3 shows the mass moisture content of W/C35%-1% specimens in the two freeze-thaw methods. The average value of W_{cr} at the 54th cycles is 6.06, while the value for M_{cr} is 6.10. Therefore, the existence of a critical mass moisture content (W_{cr}) in the accelerated freeze-thaw test has been verified. Since the critical mass moisture content W_{cr} is not affected by the different test methods, it can be regarded as a concrete characteristic value representing the point where severe damage occurs. Concrete will be deteriorated by freeze and thaw when the critical mass moisture content (W_{cr}) is achieved regardless of the test methods.

However, for the dried specimens, no clear nick point can be figured out during the whole freeze-thaw process. RDM and the mass moisture content fell even from initial state. As can be seen from Table 3, even the mass moisture content at the beginning of the freeze-thaw cycles has already exceeded the M_{cr}. Thus, the dried specimen had been not frosting resistant when the freeze-thaw test started. It is supposed to be that more micro cracks emerged on the specimen resulting from the drying process and thus the mass moisture content even from the 0th cycle became higher than W_{cr}. Consequently, RDM of the dried specimens decreased rapidly even from the 0th cycle.

3.1.2 Results of W/C55%-1% specimens

The change of RDM in W/C55%-1% specimens during freeze-thaw cycles is shown in Fig. 4. As can be seen from the figure on the left, RDM has decreased rapidly right after the freeze-thaw cycle begins and no nick point can be found during the whole freeze-thaw cycles. For the dried specimen, 55-1-D-1 and 2 specimens show a similar trend as the 55-1-N specimen. However, 55-1-D-3 specimen exhibits a different trend. A nick point shows up at 16 cycles during the freeze-thaw cycles. Table 4 shows the mass moisture content of W/C55%-1% specimens. As shown in the table, the W_{cr} of 55-1-D-3 is 6.77 and the M_{cr} of the W/C55%-1% specimens is 7.10. The two values also match well with each other, which can be also regarded as a validation of the existence of the critical mass moisture content M_{cr}. However, for the other specimens in the W/C55%-1%, even the mass moisture content at the 0th cycle is much larger than the M_{cr}, which means that specimens have already been damaged by frost at the start of the cycles.

Even though only one specimen in the dried group shows a better concrete frost resistance, it can be still regarded as a sign that an adequate drying process could improve concrete frost resistance. Besides, the trend also corresponds to the results by Tomita [2] that concrete frost resistance is improved when it is subjected to minor drying.

3.1.3 Results of W/C55%-4.5% specimens

Fig. 5 exhibits the change of RDM in the W/C55%-4.5% specimens during the freeze-thaw cycles. As shown in the figure, nick points show up in the 55-4.5-N specimens. The average value of the W_{cr} is 6.68, while the M_{cr} for the W/C55%-4.5% is 7.08. Despite the deviations, the W_{cr} can also be regarded as similar to the M_{cr}. However, as shown in the figure on the right, for the 55-4.5-D specimens, the RDM decreases continuously and slowly until 300 cycles and no clear nick points appear during the whole freeze-thaw process.

Table 5 shows the mass moisture content of 55-4.5-D specimens. From the table, for the two 55-4.5-D specimens, the mass moisture content at 300 cycles (final cycle) are 6.49 and 5.84 respectively, which are still lower than the M_{cr}. It reveals that no notable deterioration emerged before 300 cycles for the dried specimens. Concrete frost resistance is raised owing to the 20℃ drying process. It is believed that the 2 weeks drying has dried the moisture in the capillary and these empty capillaries may work as air void. Therefore, the spacing factor of the air void has been shortened and consequently a higher frost resistance can be achieved.

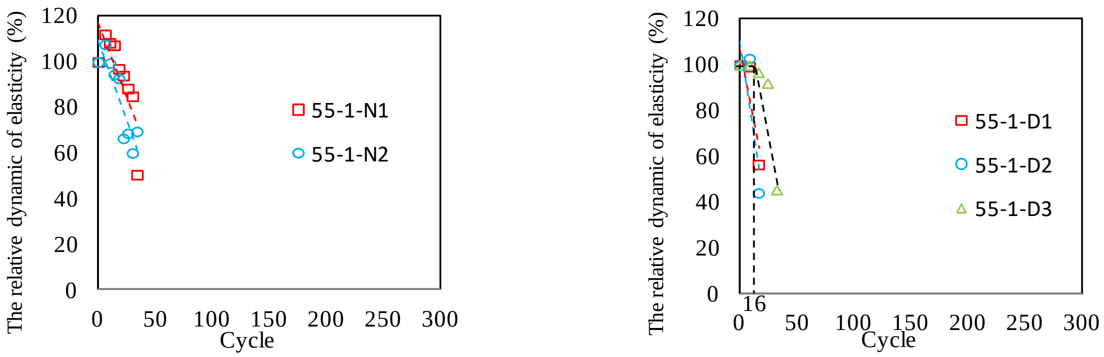

Fig. 4 – The change of RDM in the W/C55%-1%

Table 4 – The mass moisture content in the W/C 55%-1% specimens

Specimen	the critical degree of saturation test	W of the accelerated freeze-thaw test		
		0 cycle	Nf	Final cycle
	Mcr	W0	Wcr	Wfinal
55-1-N	7.10	8.23	—	5.73
55-1-D		10.09	—	2.70
		9.90		
		6.68	6.77(16cycles)	7.00

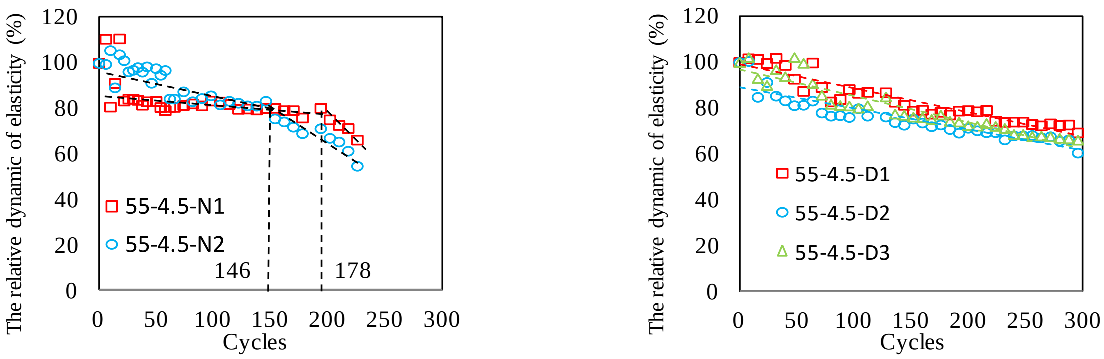

Fig. 5 – The change of RDM in the W/C55%-4.5%

Table 5 – The mass moisture content in the W/C 55%-4.5% specimens

Specimen	the critical degree of saturation test	W of the accelerated freeze-thaw test		
		0 cycle	Nf	300 cycles (Final cycle)
	Mcr	W0	Wcr	Wfinal
55%-4.5%(No drying)	7.08	5.63 6.08	6.48(186 cycles) 6.88(146 cycles) average 6.68	6.34 6.45
55%-4.5%-d20(Drying)		4.98 4.37	N/A	6.49 5.84

3.2 The accelerated freeze-thaw test

In the critical degree of saturation test, concrete frost resistance is defined by T_{pl}. From Fig. 6, T_{pl} is determined when the moisture content of the specimen reaches the critical moisture content S_{cr}. The results of the critical degree of saturation test are shown in Table 6. Durability factor (DF) and the critical freeze-thaw cycle N_f calculated from the accelerated freeze-thaw test has also been listed.

Fig. 7 exhibits the relationship between T_{pl} and DF. DF changes in a broader range than T_{pl} among the specimens. Besides, DF is not in good accordance with T_{pl}, especially in the W/C55%-4.5% specimens. Therefore, even though the same specimens are used in the accelerated freeze-thaw test and the critical degree of saturation test, conventional evaluation parameter DF does not agree with T_{pl} well.

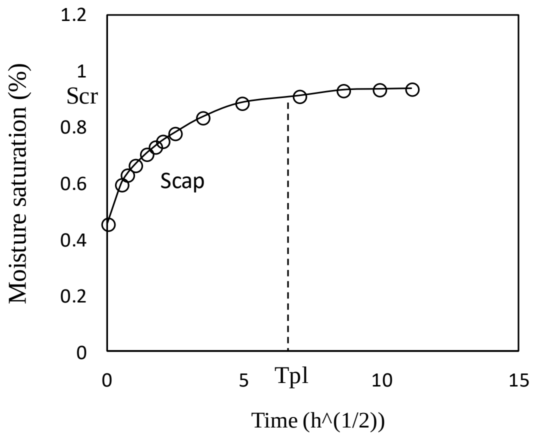

Fig. 6 – The schematic diagram of T_{pl}

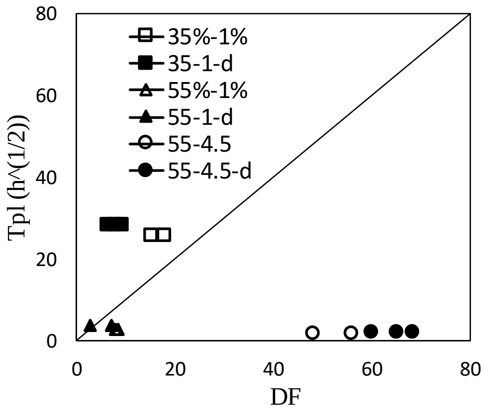

Fig. 7 – The relation between N_f and DF

Table 6 – S_{cr}, T_{pl}, DF and N_f of 3 types of specimens

Type	S_{cr}	T_{pl} ($h^{1/2}$)	DF	N_f
W/C35%-1%-no	0.98	16.6	25.72	54
W/C35%-1%-d50		7.8	28.43	0
W/C55%-1%-no	0.86	8.2	2.73	0
W/C55%-1%-d20		4.1	3.80	0/24
W/C55%-4.5%-no	0.68	52.1	1.91	146/178
W/C55%-4.5%-d20		64.5	2.23	Over 300

3.3 The accelerated freeze-thaw test

The freeze-thaw cycle (N_f) at the nick point in the accelerated freeze-thaw test is regarded as a new concrete frost resistance evaluation criterion. N_f, as a new concrete frost resistance evaluation criterion, is the freeze and thaw cycle when a dramatic decrease of the RDM occurs. Concrete can be regarded as frost resistant when the cumulative freeze-thaw cycle calculated by the freeze-thaw function in real environment has not reached the N_f. Thus, the service life of concrete can be evaluated by N_f.

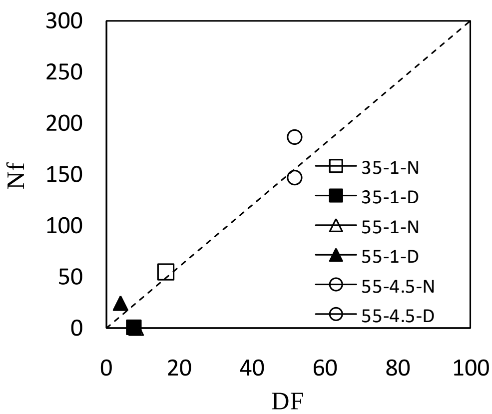

Fig. 8 – The relation between N_f and DF

Similar to the convention parameter DF, N_f can also be used to compare the frost resistance among different specimens. The relationship between N_f and DF is illustrated in Fig. 8. As can be seen from Fig. 8, N_f is almost in good accordance with DF. Therefore, the newly defined evaluation criterion N_f has mutual benefits that it cannot only represent the damage point in the accelerated freeze-thaw test but also be used to compare concrete frost resistance.

4. Conclusion

This study investigates the relationship between the critical degree of saturation test and the accelerated freeze-thaw test by comparing the mass moisture content at different stages. The nick point elucidates the mechanism of concrete frost damage emerged in both tests. The conclusions are listed as follows.

1. In the accelerated freeze and thaw test, there exists a nick point where the relative dynamic modulus of elasticity decreases dramatically. The mass moisture content W_{cr} at the nick point equals to that of the critical degree of saturation M_{cr}.
2. The cycle N_f at the nick point is regarded as a new concrete frost resistance evaluation criterion. Besides, DF is in good accordance with N_f.
3. N_f has physical meaning that it represents the freeze-thaw cycle that concrete occurs significant frost damage.
4. The 50℃ drying for 1 week may decrease concrete frost resistance, while the 20℃ drying for 2 weeks may increase concrete frost resistance.

References

1. Hasegawa, T.; Senbu, O.; and Fukuyama, T. (2014) “Comparison of the service life based on the results of the critical degree of saturation test and the JISA 1148 A test [In Japanese],” Proceedings of the Japan Concrete Institute, Takamatsu, Japan.
2. Tomita, S. (2012) “Effects of drying conditions on frost resistance of concrete using recycled coarse aggregate [in Japanese],” Summaries of technical papers of annual meeting Architectural Institute of Japan, pp. 835−836.
3. Baba, Y. (2002) “Effect of Drying Procedure on Frost Resistance and Water Absorption during Freezing-Thawing of Concrete [in Japanese],” Proceedings of AIJ Hokkaido Architectural Research Conference, Hokkaido, Japan.
4. Senbu, O. (2003) “Influence of repetitive humidity and moisture on water absorption properties and frost damage resistance of concrete [in Japanese],” Proceedings of the Japan Concrete Institute, Kyoto, Japan.
5. Fagerlund, G. (1972) “Critical degrees of saturation at freezing of porous and brittle materials,” Doctoral Thesis, Division of Building Materials, Lund Institute of Technology.
6. Fagerlund, G. (2004) “A service life model for internal frost damage in concrete,” Division of Building Materials, Lund Institute of Technology.
7. Litvan, G. (1988) “The mechanism of frost action in concrete- theory and practical implications.” Workshop on low temperature effects on concrete. National Research Council, pp. 115−134.
8. Shimada, H.; Sakai, K.; and Litvan, G. (1991) “Acoustic emissions of mortar subjected to freezing and thawing.” American Concrete Institute (ACI) Special Publication 126, pp. 263−278.
9. Fagerlund, G. (1975) “The significance of critical degrees of saturation at freezing of porous and brittle materials.” Part of Durability of Concrete, America. Concrete Institution. Publication SP-47, Detroit, 1975, pp. 13−65.
10. Bentz, D. P.; Ehlen, M. A.; Ferraris, C. F.; and Garboczi, E. J. (2001) “Sorptivity-based service life predictions for concrete pavements.” Proceedings 7th international conference on concrete pavements, Orlando FL, US.
11. Beaudoin, J. J.; and Cameron, M. (1972) “Dimensional changes of hydrated Portland cement paste during slow cooling and warming.” Cement and Concrete Research, 2, pp. 225−240.
12. Narita, R. (2015) “Influence of the Mixture Proportion of Concrete on the Various Frost Resistance Indicators using Accelerated Freezing and Thawing Test and the Critical Degree of Saturation Method [in Japanese],” Proceedings of AIJ Hokkaido Architectural Research Conference, Hokkaido, Japan.