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Abstract

This Chapter provides a detailed better understanding of the freeze/thaw effect on concrete, it is discussing the attack mechanism for both types of freeze/thaw deterioration: Internal frost damage and Surface scaling. Freeze/thaw attack is a serious problem for concrete but the most common physical deterioration type that shortening the life of concrete in cold environments. An Air-entraining agent is one of the solutions for reducing the effect of freeze/thaw cycles on concrete. Meanwhile Using supplementary cementitious materials in the production of concrete has different effects on the behavior of concrete exposed to freeze/thaw cycles. This chapter is discussing five of the common supplementary cementitious materials and their effect on concrete resistance to freeze/thaw cycles.

Keywords: freeze/thaw cycles, supplementary cementitious materials, internal frost damage, surface scaling

1. Introduction

The effect of freezing/thawing is one of the most common physical deteriorations of concrete in cold environments, which causes serious damages and induces cracks in concrete structures. This effect is clear in such European countries where the temperature drops below 0°C and ice start to form. The freezing of water inside the capillary pore structure causes an increase in the volume of approximately 9%, thus causing severe cracking and disruption of the concrete, especially if the pores in the concrete are close to saturation [1, 2]. Using high-quality and adequate amount of supplementary cementitious materials can enhance the concrete resistance to such issues. Thus, sustainable solutions can be useful not only for environmental aspect6s, but also for enhancing the concrete resistance to freeze/thaw cycles. Fly ash, silica fume, blast furnace slag, and Metakaolin are good examples for waste materials that enhance concrete's resistance to freeze/thaw cycles if used in appropriate amount.

2. Theoretical explanations of freeze/thaw attack

It was originally thought that the small, entrained air voids (deliberately introduced concrete to provide frost resistance) worked by providing sinks for the water
displaced by the volume change associated with the transformation of water into ice, but it was subsequently realized that the voids serve as nucleation sites where ice crystals can grow without constraint. During the freezing process ice crystals in the air voids suck liquid from the small pores of the paste, this suction creates negative pressure in the pore liquid which puts the entire solid matrix into compression; thereby inducing compressive stress into the concrete, Figure 1. The shown figures have been presented at the concrete & cast stone conference in Boston, USA (2008) as well fib bulletin 53 [3].

Pore system characteristics plays an essential role in the transport properties of concrete as well as the behavior of concrete when it exposes to frost action. Capillary forces determine the absorbed water in concrete and by ice expanding hydraulic pressure increases and damage occurs. The process is directly proportional to the rate of temperature decrease, in addition, forming crystals of ice could interact with the walls of the capillary pores [4–6].

3. Types of freeze/thaw deteriorations

The cement paste combines several types of voids which directly affect its properties. The typical scales of both the voids and the solid phases in the hydrated cement paste are shown in Figure 2. While the hydration reaction progresses, the spaces that are initially filled with water, are replaced by the hydration product this residual space is called capillary pore [8]. According to Table 1, capillary pores are divided into three groups: small, medium, and large capillary pores.

Since the pores which are smaller than 10 μm in diameter have less influence on permeability, capillary pores are defined as medium and large capillaries, with diameters from 10 nm to 10 μm. The pores in this range would mostly affect the permeability and diffusivity of the cement paste. The hydrated product occupies more volume than the cement particles, and with the development of the hydration reaction, in the gel form continues to expand into the capillary system. Physical deterioration and damage-inducing processes causing cracking and other effects in concrete structures can arise from various causes including freeze/thaw effects. Concrete structures are periodically exposed to the deteriorating effect of freezing/thawing damage and that comes in two types [7, 11, 12]: internal frost damage and surface scaling. The former is caused by the freezing water inside the
Concrete body, as a result of internal frost damage; weight change and compressive strength loss can occur. When the concrete surface comes into contact with weak saline solutions, surface scaling occurs and causes small flakes or chips of concrete on the surface.

The two types of freezing/thawing damages are visualized in Figure 3. The adoption of air-entraining admixtures is one of the best solutions for enhancing the freeze/thaw resistance of concrete [11]; the addition of a specific amount of appropriately sized air voids allows for the accommodation of any increase in water volume in case of freezing. As shown in Figure 4, air-entraining agent enhances the concrete's resistance to freeze/thaw cycles, as sample A (with air-entraining) experience less deterioration after 300 freeze/thaw cycles than sample B (without air-entraining) [14].

However, the result of such treatment is not always satisfactory, and the debate is ongoing. Thus, standards have recommended limitations regarding air void parameters, such as the spacing factor between voids and minimum air content of the fresh mixture [15, 16]. The recommended level of air-entraining is about 5%, which already causes a reduction in mechanical properties. It is necessary to compensate for this loss by technological steps to retain the required class of concrete.

<table>
<thead>
<tr>
<th>Name</th>
<th>Type of pores</th>
<th>Diameter</th>
<th>Paste properties affected</th>
</tr>
</thead>
<tbody>
<tr>
<td>Micro pores “inter layer”</td>
<td>Gel pores</td>
<td>Up to 0.5 nm</td>
<td>Shrinkage, creep at all RH</td>
</tr>
<tr>
<td>Micro pores “inter layer”</td>
<td></td>
<td>0.5 nm to 2.5 nm</td>
<td>Shrinkage, creep at all RH</td>
</tr>
<tr>
<td>Small (gel) capillaries</td>
<td></td>
<td>2.5 nm to 10 nm</td>
<td>Shrinkage between 50% and 80% RH</td>
</tr>
<tr>
<td>Medium capillaries</td>
<td></td>
<td>10 nm to 50 nm</td>
<td>Strength, permeability, shrinkage at high RH, &gt;80%</td>
</tr>
<tr>
<td>Large capillaries</td>
<td></td>
<td>50 nm to 10 μm</td>
<td>Strength, permeability</td>
</tr>
<tr>
<td>Entrained air</td>
<td>Capillary pores</td>
<td>0.01 mm to 1 mm</td>
<td>Strength</td>
</tr>
</tbody>
</table>

Table 1. Classification of pores in hydrated cement paste [9, 10].
Susceptibility to freeze/thaw damage is affected by the concrete’s composition, permeability, porosity, type, moisture content, age, air-entraining admixtures, workability, exposure environment, and aggregate type. Where it observed that the key factors relevant to freeze/thaw durability are the air void characteristics especially in the case of using admixtures, they should compatible, in concrete and high quality of aggregate. As well as mix design limitations according to BS EN 206:2013 + A1:2016 [17] are to be considered necessary [18].

Yet not sufficient conditions for achieving high frost resistance of concrete, especially when exposed to severe frost, wet conditions, and high levels of de-icing agents. Thus, following the principles transpiring from the literature’s observations during the design of freeze/thaw resistance concrete can be highly beneficial. Some of these observations have been concluded as [11]:

- High-quality aggregate should be used featuring appropriate frost resistance tested in NaCl solution and water, high resistance to fragmentation, continuous grading, and minimal water requirement.
When specifying admixtures like Supplementary cementing materials, to concrete it is necessary to check their compatibility by valuating the air void characteristics in hardened concrete, and this concerns in particular air-entraining and water-reducing admixtures.

The severity of freezing (minimum temperature); rate of fall of air temperature (rate of freezing); change of air temperature during periods of freezing; the number of freeze/thaw cycles and the presence of de-icing salts is the major environmental aspects and critical factors that are participated in the developing of freeze/thaw damage.

5. Evaluation of the freeze/thaw deterioration

There are many test methods by which the internal damage and surface scaling of concrete can be evaluated. ASTM C 666 [19] and ASTM C 672 [20] are the typical tests by which the internal damage of the concrete specimen and the scaling of the specimen surface in the presence of de-icer salts can be assessed prospectively. However, numerous national freeze/thaw tests resemble the previous direct tests performed by using actual freezing and thawing loads. In the following two sections the used procedure for testing the internal frost damage and surface scaling test is explained.

5.1 Internal frost damage

The following procedure is usually used to apply the internal frost damage test:

1. Submerging concrete cubes in water until full saturation.
2. Lifting reference concrete cubes in water and placing the rest in a laboratory freezer until a specific number of freeze/thaw cycles.

According to CEN/TR 15177 [21] each freeze/thaw cycle includes (two hours of cooling, two hours of freezing at −20°C, two hours of thawing, and 2 hours at +20°C). Concrete cubes are surrounded by air in the freezing phase, while surrounded by water in thawing phase.

3. Lifting the concrete cubes in water for three days after specified number of freeze/thaw cycles, which are determined based on the purpose of the study. This is to prevent any residual freezing from occurring inside the concrete cubes.
4. Finally, testing the residual compressive strength and/or determining the weight change.

Figure 5 shows the time–temperature curve in the centre of the concrete sample, where: 1 / freeze/thaw cycle; 2 / temperature range in the reference; Y / temperature in °C; and X / time in h.

5.2 Surface scaling

In accordance with CEN/TS 12390–9 [22] (slab test) surface scaling is evaluated. The main idea the test is to determine the scaled materials amount after exposing the concrete surface to 56 freeze/thaw cycles, where the tested concrete surface should be in contact with a 3% NaCl solution. The testing procedure begins by
cutting concrete cubes in half and using the sawn surface to come into contact with 3% NaCl solution, while the other surfaces must be isolated. For each specimen (half concrete cube), 5 mm thickness of 3% NaCl solution should be placed in contact with the sawn surface sand placed in freezer to start the freeze/thaw cycles. Each freeze/thaw cycle includes (six hours of cooling, six hours of freezing at $-20^\circ$C, six hours of thawing, and six hours at $+20^\circ$C). After a specified number of freeze/thaw cycles, up to 56 cycles, the scaled material is collected from the tested surface and weighed, the higher the weight, the higher the scaling.

Figure 6 shows the time–temperature curve in the freezing medium at the centre of the test surface, where: 1 / temperature range at the centre of the test surface, whereby the time of temperature $> 0^\circ$C is 7 to 9 h; Y / temperature in $^\circ$C; and X / time in h.

6. Internal frost damage mechanisms

The durability of concrete refers to its ability to withstand deterioration due to harsh environmental conditions. These conditions can act alone or together and
include heating and cooling, freezing and thawing, wetting and drying, chemical attacks, and abrasion. Deterioration due to freezing/thawing causes D-cracking to occur [23]. Under harsh service conditions, the durability of reinforced concrete structures is related to concrete frost resistance. Frost resistance tests are accompanied by the accumulation of residual dilation deformations affected by temperature-humidity stresses, ice formation and other factors. It is affected directly by the porosity, which is an integral part of the concrete structure which is formed as a result of cement hydration [24]. If the porous material is so wet that the theory of hydraulic over-pressure governs the freezing phenomenon, pore water is squeezed into the larger air-filled pores and the external environment surrounding the sample and causes there an abrupt increase of relative humidity. However, if the pore system is not filled with pore water to the extent that hydraulic pressures are induced into the material then after the first freezing of the pore water an under-pressure is formed in the pore system and the sample contracts [25], Figure 7.

D-cracking is a type of freeze/thaw damage in concrete pavements, it occurs due to the poor-quality coarse aggregates. By increasing the wet level of coarse aggregate reaching saturation, it becomes more susceptible to damage during freezing/thawing cycles. Pressure builds up inside of the coarse aggregate as a result of water freezing inside its pores. If the pressure due to the expansion of the water within the pores of the coarse aggregate is higher than its internal strength, the coarse aggregate will crack. Then the deterioration process accelerated due to the increased potential water availability, where the interfacial transition zone between the coarse aggregate and cement matrix is supposed to be slightly thicker. Its porosity can be 100% and that increases and accelerates the freeze/thaw deterioration [23, 26, 27].

Sicat et al. [28], investigated experimentally the real-time deformational behavior of the interfacial transition zone in concrete during freeze/thaw cycles. They observed that due to its high porosity and weak strength of the interfacial transition zone, its deformation is higher than that of cement matrix and aggregate. The deformation has been experimentally proven by taking a closer look at the unbroken sections of the interfacial transition zone of the specimens by electron microscope after the freeze/thaw cycles in Figure 8. This deformation is clearer in the case of wet specimens as well as much significant in higher water to cement ratios.

It is observed that the deterioration due to freeze/thaw cycles increases by increasing the number of cycles, and sometimes a certain number of freeze/thaw cycles is required before the deterioration occurs, and that could be the effect

![Figure 7](image-url)  
*Figure 7. Water transfer mechanisms in the freezing theories of wet porous materials [25].*
of fatigue. As each freeze/thaw cycle is added to cumulative internal deterioration this is similar to the normal mechanical fatigue with constant load cycles. However, it considers as low-cycle fatigue, where normally the freeze/thaw cycles are less than 300.

The reason behind the fatigue hypothesis is that one often notices a development of damage of the type shown in Figure 9 when the material is tested in so-called “open” freeze/thaw, i.e. in a test where the specimen has access to water, during freezing and/or during thawing. In some cases -curves D, P, C in Figure 9 damage increases progressively with the number of freeze/thaw cycles already from the first cycle, in other cases, all other curves a certain number of freeze/thaw cycles are needed to initiate.

The residual compressive strength of concrete after the effect of the freeze/thaw cycle is tested by Lu et al. [30] as shown in Figure 10, Left. It shows the changing of compressive strength after 0, 25, 50, and 75, freeze/thaw cycles respectively when the strain rate remained constant. They observed that the compressive strength decreased linearly with an increasing number of freeze/thaw cycles, where after

![Figure 8](image1.png)

**Figure 8.**
Interfacial transition zone after freeze/thaw cycles (from left to right 30%, 50%, and 70% w/c); (a) dry conditions, (b) saturated conditions [28].

![Figure 9](image2.png)

**Figure 9.**
Reduction in E-modulus of cement mortar specimens repeatedly frozen in the air to −15°C and thawed at water +5°C, 2 cycles per day [29].
repeated freeze/thaw cycles, the cracks in the concrete will pass through each other, and their strength will gradually decrease, and finally even completely lost. However, the compressive strength increases with strain rate in a nearly linear progression as shown in Figure 10, Right. Under freeze/thaw cycles, crack propagation becomes faster with a higher stress rate, however, enhancement of concrete strength could happen due to the coarse aggregate that stops the crack extension. They find a decrease in the residual compressive strength of concrete if the concrete was subjected to a fatigue compression loading prior to freeze/thaw cycling and this reduction was increased by increasing the fatigue cycles. Fatigue cycles cause microcracks and that increase in the irreversible tensile strain due to freeze/thaw cycling afterwards. Han and Tian [31], presented the stress–strain relationships after different freeze/thaw cycles as plotted in Figure 11. They concluded that the concrete deformation experienced four deformation stages: compaction, elastic deformation, plastic deformation, and finally fracture. The peak load decreases as the freeze/thaw cycling increase where microcracks occurs as a result of freeze/thaw cycling, its number affects the microcrack number and width.

7. Mechanism of action of the air-entraining admixtures

For adequate resistance of concrete to the freezing/thawing cycles; air voids should not have a spacing factor (the maximum distance between two air voids in
concrete) larger than 0.2 mm or 0.008 in. For a given air content, the size of the air voids cannot be too large if the proper spacing factor is to be achieved. The specific surface should be greater than 24 mm²/mm³ [32], where the air void system consists mainly of three parameters: the void's size, the space factor, and the void size distribution [33, 34]. As previously mentioned, to produce freeze/thaw resistant concrete; a proper air-void system is required in the concrete to accommodate the volume extension of water when it freezes without causing damages.

Artificially, the use of effective air-entraining admixtures can ensure the stabilization of the air-void system and produce air-entrained concrete which is one of the greatest advancements in concrete technology [35]. The term “air-entrainment” refers to the air deliberately introduced into concrete by adding air-entraining admixture. In the case of using air-entraining admixtures, the entrained air voids reduce the hydraulic pressure by behaving as expansion chambers despite the volume increment when water turns into ice [36, 37]. The schematic mechanism of the performance of concrete (with and without air-entraining admixture) exposed to freezing is shown in Figure 12. Šelih [39] and Wang et al. [40], investigated the freeze/thaw resistance of concrete with air-entraining admixture and low water content and they experienced a better performance of 10 times lower mass loss comparing to the normal one.

Air-entraining admixture is either surfactant that reduces the surface tension of water, or substances that produces a water-repellent precipitate which is required to produce the air voids dispersed throughout the concrete and that ultimately provides durability in freezing/thawing situations. Surfactant air-entraining admixture secured the best overall air void characteristics, followed by salt-type air-entraining admixture containing tall oil, and then, salt type air-entraining admixture containing Vinsol resin and wood rosin. However, some types of air-entraining admixtures, including vinsol resin, sodium adipate, sodium oleate, do not reduce the surface tension of water [16, 41]. The fresh state of concrete seriously affected its freeze/thaw resistance like the flowability and setting time of concrete. Air voids have a lifetime and should not be unstable, where they could be collapsed due to different fundamental physical mechanisms. Such as the diffusion of air from a void to a larger one, the void coalescence due to capillary flow, or the rapid hydrodynamic drainage of liquid between voids [33].

Other types of chemical admixtures are usually used to control the flowability and viscosity of concrete such as superplasticizers and viscosity modifying admixtures, which also affect the hardened properties of concrete and generally they affected the

Figure 12.
*Performance of normal and air-entrained concrete exposed to freezing a) cumulative hydraulic pressure over-saturated voids b) mitigating hydraulic overpressure condition by the distribution of air bubbles [38].*
dosage rate of air-entraining admixtures. Viscosity modifying admixture increases the mix viscosity and its mode of action depends on the type and concentration of the polymer in use, while the superplasticizer causes a reduction in total air void surface areas and increases in air void spacing factors [31, 42]. Meanwhile, polycarboxylate superplasticizers usually have an air-entraining effect, whereas the use of polycarboxylate superplasticizers; the air voids characterize with smaller diameters than voids formed as a result of lignosulphonate or naphthalene superplasticizers [41, 43].

The usage of admixtures such as viscosity modifying admixtures and superplasticizers can reduce the ability of an air-entraining admixture to create a proper air void system. Where the air content seems to be decreased with the increase of viscosity modifying admixture content and that will probably necessitate greater additions of air-entraining admixture to secure a given air volume [44, 45]. However, and generally; because of the complexity of modern air-entraining admixtures and other chemical admixtures, it is impossible to generalize the effects of their interactions with surfactants on the air entrainment.

8. Effect of supplementary cementitious materials on freeze/thaw resistance

Recently, the use of supplementary cementitious materials has dramatically increased due to an increase in environmental awareness. Incorporation of various mineral admixtures, supplementary cementitious materials, into concrete as one of the cement-based composites is a generally well known and frequently used approach. Supplementary cementitious materials are usually employed for partial replacement of cement in the production of concrete, where their use brings significant ecological and economic benefits [12].

Fly Ash, blast furnace slag, silica fume and metakaolin are commonly used supplementary cementitious materials for the purpose of enhancing the performance of concrete from a different point of view, such as enhancing the concrete’s properties, value, and cost. They have a clear effect on the fresh, mechanical and durability performance of concrete, and thus on its Freeze/thaw resistance. Several studies conducted on the effect of supplementary cementitious materials on concrete performance have been reviewed [46–48]. Most of these studies have indicated that adding supplementary cementitious materials helps in resisting deleterious effects, such as alkali-silica reactivity, freeze/thaw deterioration, random cracking, and permeability. Supplementary cementitious materials enhance the frost resistance through reducing the macro capillary porosity of cement matrix and form stable gel-like hydration products [24].

8.1 Cement type

Concrete is rich in alkali and therefore it reacts actively with acidic gases and liquids, freeze/thaw resistance of hardened cement paste plays an important role in the resistance of concrete to the freeze/thaw cycles. Especially in the case of using high-quality coarse aggregate with low porosity. The freeze/thaw resistance of cement paste depends on its porosity, the size of pores, capillaries, and their distribution [49]. Skripkiūnas et al. [50], tested the freeze/thaw resistance of concrete made with different types of cement, the effect of four types of cement on the freeze/thaw resistance of concrete has been investigated by them: CEM I type Portland cement, CEM II/A-S 42.5 N and CEM II/A-LL 42.5 R contained 6–20% of blast furnace and limestone, and CEM III/B 32.5 N-LH contained 66–80% of slag. They concluded that concrete containing slag cement CEM III/B 32.5 N-LH has the highest freeze/
thaw and de-icing salt scaling resistance. While concrete containing Portland cement CEM I 42.5 R has the lowest freeze/thaw and de-icing salt scaling resistance.

The ductility of concrete made of cementitious composites decreased remarkably and the cement containing slag is freeze/thaw resistant [51]. Deja [52] observed a high salt scaling resistance of concrete containing cement rich in granulated blast furnace slag and by using air-entraining admixture even at relatively high values of w/c ratio. Skripkiūnas et al. [53] investigated hardened cement paste made of CEM I 42.5 R cement modified with synthetic zeolite admixture, the mass loss and deformations freezing/thawing cycles were much lower in concrete modified with synthetic zeolite which modifies the morphology of hardened cement paste. However, in other research for Skripkiūnas et al. [54], they investigated the freeze/thaw resistance of concrete made of CEM I 42.5 R cement modified with sodium silicate solution and found that the destruction after 56 freeze–thaw cycles and exposure do de-icing salt solutions is smaller in hardened cement paste modified with sodium silicate solution. It may be used to improve the durability of hardened cement paste and concrete used in road building.

8.2 Fly ash

Fly ash is one of the most popular mineral additives that used in the concrete industry, it helps in improving the consistency of fresh concrete and reducing the hydration heat. The use of fly ash has been investigated extensively in the literature and it is found that it is beneficial for the durability of concrete, and it enhances the permeability properties. Replacement cement by over 40% of fly ash causes a serious reduction in the service life. Fly ash has a low carbon content and very variable properties, thus its effect on freeze/thaw resistance is not clear. Despite of the complexity of defining the influence of fly ash on freeze/thaw resistance, it is favorable due to its positive effect on the long-term durability of concrete [47, 48, 55].

8.3 Blast furnace slag

Blast furnace slag is a by-product formed during pig iron production. The incorporation of ground blast furnace slag into concrete is very beneficial because of its positive effect on the durability properties [56]. Blast furnace slag has a positive effect on many engineering properties, such as bleeding, consistency of the fresh mixture, the heat of hydration and permeability. The relatively high bulk density of a cement matrix modified by slag frequently leads to a decrease in the frost resistance, however, the results of experimental studies are inconsistent [57]. This could be partly a result of the different experimental procedures and also because of the use of modifying agents such as plasticizers, which significantly affect the character of the porous system.

8.4 Silica fume

Using silica fume as a substitution of cement usually does not exceed 5–10%. Therefore, its effect on the frost resistance seems to be marginal, provided the air content is maintained on a level, that is high enough. Besides, the long-term tests indicate, that a negative effect occurs only when the amount of silica fume exceeds 10% [13], Figure 13.

8.5 Metakaolin

Metakaolin has been generally used as a filler or supplementary cementitious material in concrete, and it was proved to be able to enhance its performance.
The introduction of metakaolin into concrete as a supplementary cementitious material can improve its freeze/thaw resistance significantly. Where metakaolin decreases the total porosity and improves the pore structure of concrete which is directly related to the mechanical properties [58]. It was found that the freeze/thaw resistance corresponds to the content of insoluble hydrates in the cement paste. However, the used binder system also significantly affects the character of the concrete capillary system. In general, modern concrete mixtures with reduced water to cement ratio exhibit increased frost resistance; nevertheless, this approach tends to increase the autogenous shrinkage, which could cause propagation of surface cracks during hardening in the case of a poor curing regime, resulting in the reduction of the durability and especially the frost resistance [51, 55].

9. Conclusions

This chapter discusses theoretically the freeze/thaw attack of concrete in terms of its mechanism affected factors. Two types of freeze/thaw attack were discussed, Internal frost damage and Surface scaling. Either type of freeze/thaw attack, increasing the number of freeze/thaw cycles leads to deteriorating the properties of concrete, owning that to the weakened bond between aggregates and paste caused by the development of internal cracks in the cement paste with repeated cycles. Using high-quality aggregate and/or supplementary cementitious materials can enhance the concrete resistance to such issues. However, checking the compatibility of the used materials in terms of evaluating the air void characteristics is mandatory in particularly when the air-entraining agent is used. Fly Ash, blast furnace slag,
silica fume, and metakaolin are commonly used SCMs for the purpose of enhancing the performance of concrete. It is concluded that adding supplementary cementitious materials helps in resisting deleterious effects, such as alkali-silica reactivity, freeze/thaw deterioration, random cracking, and permeability.

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Conflict of interest

No conflict of interest.

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