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Freeze-Thaw Deicing salt Attack on Concrete: Towards Engineering Modelling

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Abstract. *In order to reduce carbon dioxide emissions, the construction sector is increasingly using clinker-reduced concretes. Concrete with high amounts of ground granulated blast furnace slag (GGBFS) show increased durability performance in certain environmental conditions compared to concrete with Ordinary Portland cement (OPC). However, in laboratory studies, concrete with GGBFS show an increased initial scaling rate in comparison with OPC concrete. Changing weather conditions and high relative humidities > 70 % in field lead to less carbonation and thus, to a better freeze-thaw deicing salt resistance (FTDSR). Therefore, the current lab performance test penalizes GGBFS concrete. This contribution studied the impact of carbonation and intermittent dry periods on FTDSR of GGBFS concrete compared to that of OPC concrete. It could be shown that for concrete with OPC, intermittent dry periods lead to an accumulation of salt in the near surface, which causes a reduction of the scaling. GGBFS concrete, on the other hand, shows an opposite behavior: carbonation causes an increase in scaling after the dry period. GGBFS concrete samples with* CO_2*-free pre-storage show the lowest scaling of all samples investigated. A better simulation of the behavior of GGBFS concrete could lead to economical as well as optimized solutions for the local environment conditions and enable the large-scale use of more environmentally-friendly concretes. Therefore, by taking the effect of carbonation and intermittent dry periods into account in a simple engineering model, the prediction of concrete scaling under consideration of real climatic conditions is possible in an eco-efficient manner.*

Keywords: *Freeze-Thaw Deicing Salt Attack, Carbonation, Durability, Service Life Prediction, Combined Attack.*

1 Introduction

With the increased use of clinker-reduced concretes, the evaluation of durability design methods remains a topical issue. There are mainly three design strategies:

- Deemed to satisfy rules: Avoidance of damage by complying with the requirements from the exposure classes, and
- Lab-Performance concept: Compliance tests with accepted limit criteria provide a yes/no decision on the suitability of a material for a certain environment; see (Thiel and Gehlen, 2018).
- Performance based durability design: The condition of a structure remains with a certain reliability below selected limit states (Gehlen, Schießl and Schießl-Pecka, 2008.; fib, 2006).

Performance based durability design can be further divided into fully probabilistic approaches, and the partial safety approach derived from the probabilistic concept. Probabilistic service life design require deterioration-time-laws that account for statistical uncertainty. An important input parameter is the material resistance, which is usually determined in compliance tests in the laboratory, (Thiel and Gehlen, 2018). Those engineering models should be as simple as possible in order to promote acceptance. In addition, different attack intensities, i.e. different climatic conditions should be realistically displayed. They should also be flexible so that new materials, new knowledge or new situations (*e.g.* climate change) can be accounted for. The use of performance based design concepts offer the following advantages

- Quantification of deviations between execution and planning
- Optimized maintenance strategies through service life prediction
- Optimized choice of material as well as curing time and type
- Simplified use of new building materials through proof of performance

Djuric successfully combined the critical saturation model from Fagerlund (Fagerlund, 2004) with the transport mechanism of the micro ice lens pump (Setzer, 1999) for pure freeze-thaw attack, thus developing a model for the evolution of internal structural damage (Djuric, 2017). For the durability assessment of structural components under combined freeze-thaw deicing salt attack (FTDSA), different approaches exist. For example, a simple performance concept based on the CDF method was proposed by Guse (Guse, 2009). The glue-spall model developed by Valenza (Valenza and Scherer, 2007) is able to explain most observations during FTDSA. An easy and structured factor approach was proposed by Sarja *et al.* (Sarja and Vesikari, 1996) and further developed by Lowke *et al.* (Lowke and Brandes, 2008). Currently, Müller *et al.* (Mueller; Ludwig and Erhardt, 2019) are combining the cryogenic suction model with frost suction in order to model the scaling evolution under FTDSA.

However, evidence of large-scale practical applications of these concepts is still missing. The aim of this contribution is to demonstrate, by means of practical experiments, how the strongly varying attack intensity under field conditions can be taken into account.

2 Materials and Methods

Two concrete mixes using quarzitic sand and gravel were produced, Table 1. C1 contained Ordinary Portland Cement (OPC) with the European label CEM I 42.5 R, while C2 labelled CEM III/B 42.5 N contained 75 % ground granulated blast furnace slag (GGBFS). For carbonation to occur, a rather high w/b-ratio of 0.55 was used. Therefore, the concrete composition is neither XF2 nor XF4 conform, see (DIN 1045-2:2008-08).

Table 1. Concrete composition, air content and compressive strength.

	C1	C2
Binder type	OPC	GGBFSC
Binder amount (kg/m³)	320	320
w/b-ratio	0.55	0.55
Sand + Gravel (kg/m³)	1732	1724
Air content	1.9	2.0
28d compressive strength	42.5	38.5

Entrained air is the dominant factor in ensuring high FTDSR for OPC based systems (Powers, 1949). However, Auberg showed that the FTDSR of concrete with GGBFS ≥ 58% in the cement was largely unaffected by entrained air voids (Auberg, 1999). Furthermore, entrained air is not required in the exposure class XF2. As this contribution intends to exploit the potential of non-entrained concrete more effectively, no air-entraining agent was used. Carbonation leads to increased initial scaling and is the dominant parameter for concrete with

GGBFS cement. Ludwig justified this observation with the coarsening of the pore structure due to the formation of metastable aragonite and vaterite, which are dissolved by FTDSA. 25 cubes (edge length 150 mm) were cast against PTFE slides, immediately covered with moist cloth and stored at 20±2°C. After demoulding at an age of 1 d, the samples were stored further 6 d under water. Then the samples were cut according to the CDF method (DIN CEN-TS 12390-9:2017-05) and divided in five different series of five samples each, Fig. 1.

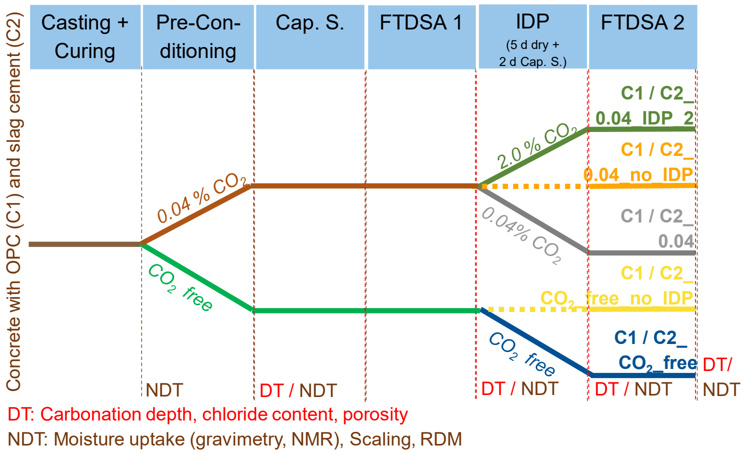

Figure 1. Overview of experimental program (FTDSA: Freeze-thaw deicing salt attack, Cap. S.: Capillary Suction; IDP: intermittent dry period, DT: destructive testing, NDT: non-destructive testing, RDM: relative Young's modulus).

Three series were stored according to the CDF method at 20±2°C and 65±2 % RH and natural CO_2 content of 0.04±0.005 % by vol. Two series were preconditioned under the same temperature and humidity conditions but in argon flooded chambers creating a CO_2 free atmosphere. All samples were exposed to a seven days period of capillary suction followed by freeze-thaw cycles in 3 % NaCl solution. The setup followed the guidelines of the CDF method, however the temperature cycle was changed. In order to reduce damage, the minimum temperature was increased to -10°C, which is closer to field situation in Germany, see (Siebel *et al.*, 2005). Here, the holding times remained constant but cooling and heating rate were lowered from 10 K/h to 7.5 K/h. Before, during and after FTDSA nondestructive tests as well as destructive tests characterized the evolution of damage, Table 2. For porosity measurements, the samples were manually broken, immediately exposed to isopropanol for 12 - 24 h and then oven dried at 40°C for 24 ± 2 h.

Table 2. Experimental methods.

Test	Goal	Device	Reference
Modified CDF (-10°C): Scaling, Relative Young's Modulus (RDM), solution uptake	Determination of scaling rate and possible relation to moisture uptake	Chamber, Balance, US-equipment (Pundit)	(DIN CEN-TS 12390-9:2017-05)
Carbonation depth (colour indicator spray test)	Determination of the effect of carbonation	Spray bottle with 1% phenolphthalein in 70% ethanol	(RILEM CPC-18, 1988)
Mercury Intrusion Porosimetry (MIP)	Determination the effect of carbonation and FTDSA on pore structure	AutoPore III, hammer for crushing	(Lomakovych, V., 2018)

3 Results and Discussion

Fig. 2 shows scaling, moisture uptake and RDM of the concretes. Especially the accelerated carbonation leads to a five times higher scaling rate for C2. The orange line (C1/2_0.04_no_IDP) represents the standardized procedure (DIN CEN-TS 12390-9:2017-05), which is quite in the middle of the different series. Despite the significant effect of the different storage conditions, the carbonation depth in general was very low. Thus, Table 3 shows only the carbonation depth of the series with which a color change occurred at all. For the C1 samples, for example, only the surface was usually uncolored, while the carbonation depth for the C2 was 0.1 mm, which disappeared in the course of the 14 freeze-thaw-cycles (FTCs). No carbonation was detected before the intermittent dry period (IDP) indicating that the carbonated layer scaled off.

Table 3. Carbonation depth (mm).

Series	Exposure time (d)			
	0 (before FTC)	7 (after 14 FTC)	14 (after either 28 FTC or after IDP)	21 (after 28 FTC
C1_0.04_IDP_2	0.0*	0.0	0.1	0.0
C2_0.04_IDP_2	0.1	0.0	0.4	0.0
C2_0.04	0.1	0.0	0.0*	0.0
C2_0.04%_no IDP	0.1	0.0	0.1	0.0

*Surface was colorless (carbonation depth < 0.1 mm)

The solution uptake shown in Fig. 2 middle correlates neither with the external damage nor with the drop of the RDM (bottom). After an IDP, the samples very quickly reabsorb moisture; however, for all series the total solution uptake remains below the level of the continuously exposed series. The RDM of the C1 only decreases for samples that have not been dried in the meantime ("no_IDP"). The high scatter of the reference series (C1/2_0.04_no_IDP) is most likely due to detaching of the sealing of two samples. This is also reflected in the RDM.

C2 did not show any significant changes in RDM. This could be due to the finer pore structure,

Fig. 3. C1 has a lower total porosity before and after the FTDSA compared to C2.

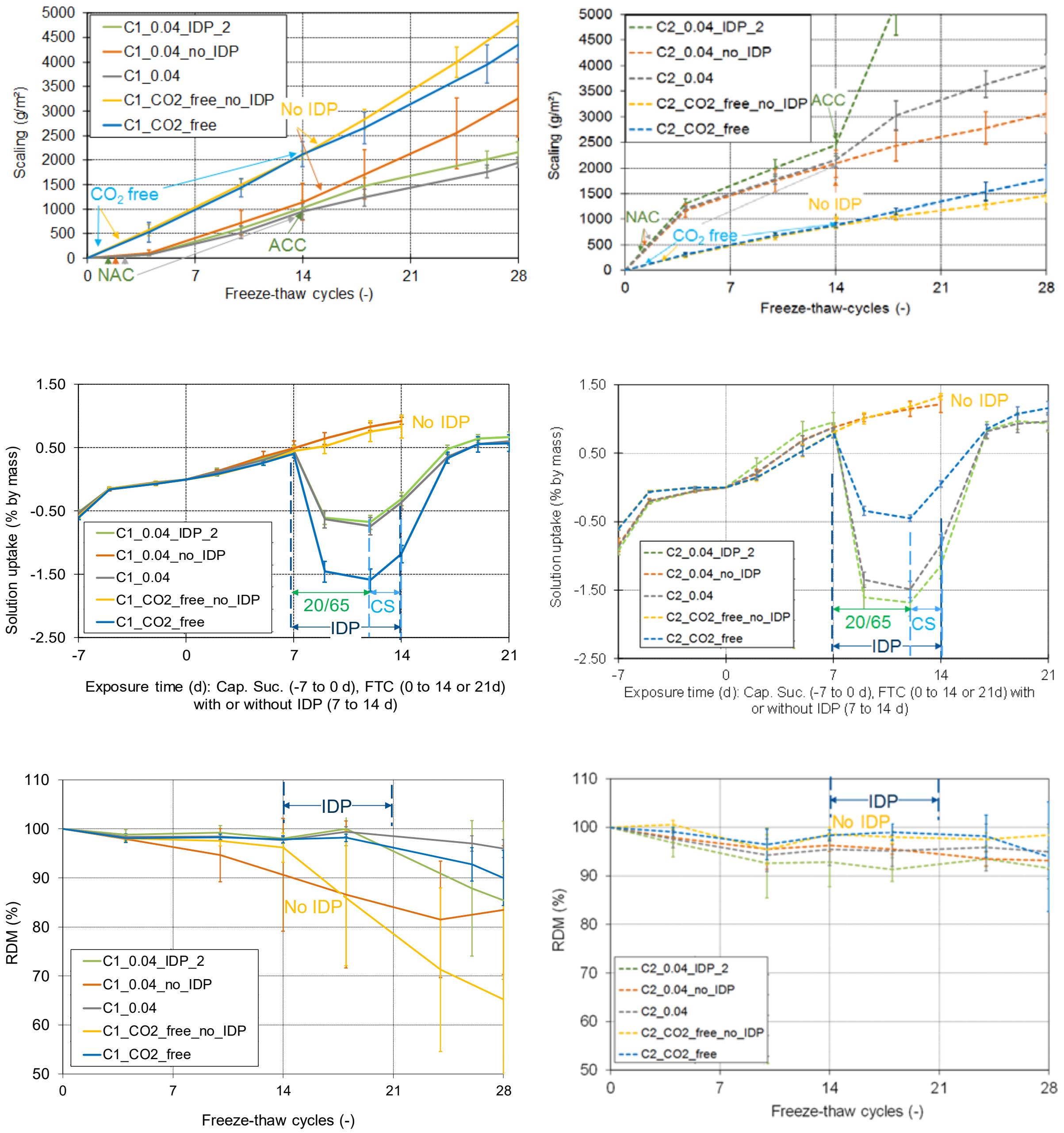

Figure 2. Top: Scaling of C1 (left) and C2 (right), middle: solution uptake of C1 (left) and C2 (right) (note: x-axis starts with dry state before capillary suction and represents the exposure time); bottom: Relative Young’s modulus of C1 (left) and C2 (right). (NAC: Natural Carbonation; ACC: Accelerated Carbonation, CS: Capillary suction).

In all cases, FTDSA led to an increased total porosity. Note that only concrete exposed to three weeks of natural carbonation was measured as “before FTDSA”. The carbonation front was already scaled off which explains why the well described coarsening of the pore structure due to carbonation in C2 was not found here. C1_0.04_no_IDP had the highest total porosity and a clear increase in the capillary pore range. This clearly indicates micro-cracking which is also confirmed by the RDM (Fig. 2 middle left).While a coarsening of the pore structure occurred for C1, the pore structure became finer in all C2 series, Fig. 3. This is most likely due to ongoing hydration. To simplify matters, if the carbonated surface layer is scaled off, GGBFS concrete show higher FTDSR than OPC concretes with otherwise identical composition. However, IDPs can cause the exposed surface layer to carbonate again, resulting in high initial scaling, corresponding to the carbonation depth. With OPC the behaviour is reversed. This shows that a test according to 28d is not sufficient to describe the damage behaviour in practice and that it is absolutely necessary to consider the age and the degree of carbonation and hydration.

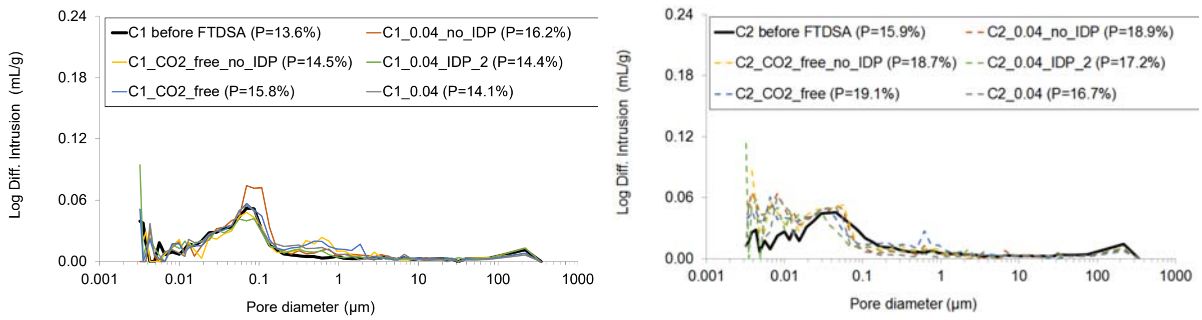

Figure 3. Pore size distribution and total porosity (P) of C1 (left) and C2 (right) before and after FTDSA.

The following options are available for considering our findings when designing or maintaining structural components exposed to FTDSA:

- Deemed to satisfy rules: Further subdivision of the exposure classes XF2 and XF4 according to the expected time in which a critical saturation occurs.
- Performance based durability design: Implementing the effect of IDPs and carbonation on scaling rate in an engineering models

4 Engineering Model

For chloride-induced corrosion as well as for carbonation validated probabilistic models exist (Gehlen, 2000). For carbonation induced corrosion, the effect of changing moisture conditions is taken into account by a parameter for considering the effect of micro-climatic conditions (relative humidity) and a parameter for considering the macro-climatic conditions (time of wetness and probability of wind-driven rain for vertical components. A similar procedure could be used for scaling due to FTDSA: The scaling rate over time $s_r(t)$ can be described by including the initial scaling rate and a later scaling rate determined in a lab performance test. The initial scaling rate $s_{r,ini}$ accounts for the effect of carbonation while the later scaling rate $s_{r,prog}$ represents the evolution of scaling of the uncarbonated material under damage-relevant environment. The environment only leads to scaling when sufficient moisture, rather low chloride content and a minimum Temperature below - 5°C are present at the same time. This is included in the environment factor f_e, which depends on the number of damage-relevant freeze-thaw-cycles in one winter period as well as on the outer salt concentration and the minimum temperature. As the resistance of the concrete is affected by curing, the curing factor f_c is also introduced, Equation 1.

$$s_r(t) = (s_{r,ini} + t \cdot s_{r,prog}) \cdot f_c \cdot f_e \cdot f_a \tag{1}$$

This approach allows a weighting of the different scaling rates. The exact statistical distributions as well as dependencies need to be quantified in future. Moreover, suitable limit states need to be defined. Scaling not only leads to visual defects, it also reduces the concrete cover. Cracking reduces mechanical performance and might accelerate other mechanisms relevant to durability. Therefore, a much lower scaling depth than the concrete cover is certainly tolerable.

5 Conclusions and Outlook

FTDSA is very versatile. It strongly depends not only on the material resistance but also on the macro- and micro-climatic conditions. Recent investigations (Aßbrock *et al.*, 2019) showed that for most European locations, freeze-thaw deicing salt attack (FTDSA) is milder than displayed in laboratory tests. Consequently, there is a need for eco-efficient and economic methods to design and maintain structures under FTDSA despite the current deemed to satisfy rules. The most important findings of this contribution are summarized as follows:

- Damage evolution strongly depends on the climatic conditions. The scaling rate can change considerably for structural components that can dry in field during periods without precipitation.
- For concrete with OPC, the scaling rate was reduced by intermittent dry periods with and without carbonation. The main reason for the reduction of the scaling rate is probably salt crystallization, which should be further investigated.
- Carbonation was the dominant influencing parameter for concrete with GGBFS. Here, after an IDP concrete with GGBFS will always exhibit a new increased initial scaling rate until the carbonated layer is scaled off. Afterwards, the scaling rate is lower than for concrete with OPC. Concretes with high amount of GGBFS could be used in building components that are exposed to low carbonation (few dry periods, high relative humidity).
- Palecki (Palecki, 2005) and others found a correlation between inner damage and moisture uptake for freeze-thaw attack without deicing agents. This correlation could not be confirmed for FTDSA. Although concrete with GGBFS absorbed more solution than concrete with OPC, the finer pore structure caused only minor changes in the RDM while concrete with OPC showed a significant drop in RDM.
- IDPs significantly reduce the occurrence of inner damage.
- A new concept was proposed to consider the effect of real weather data and include the effect of carbonation. With the help of this, a weighting of the different scaling rates of the carbonated and non-carbonated layers would be possible.
- Taking into account the actual performance of the material as well as the actual expected intensity of attack would enable more economic and eco-efficient design of concrete structures.

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