

LEACHING OF CONCRETE UNDER THERMAL INFLUENCE

TEMPERATURABHÄNGIGKEIT DER AUSLAUGUNG VON BETON

LA RELATION ENTRE LA TEMPERATURE ET LA LIXIVATION

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SUMMARY

Leaching represents the main mechanism for self-healing in concrete. Up to present the relationship between temperature and leaching is unclear. In the present work the influence of temperature was therefore examined at 20, 50 and 80 °C. The results show that an increase of temperature is not always accompanied by an acceleration of the transport processes. For this purpose, high performance concretes with different types of cement and aggregates, and the French BPR were examined and compared with a reference concrete and among themselves.

ZUSAMMENFASSUNG

Die Auslaugung stellt den hauptverantwortlichen Selbstheilungsprozess in Beton dar. Unklar ist hierbei noch der Zusammenhang zwischen der Temperatur und der Auslaugung. In der vorliegenden Arbeit wurde deshalb der Einfluss der Temperatur bei 20, 50 und 80 °C untersucht. Die Ergebnisse zeigen, dass mit der Erhöhung der Temperatur nicht immer auch eine Beschleunigung der Auslaugung einher geht. Dabei wurden hochfeste Betone mit unterschiedlichen Zementarten und Zuschlägen, sowie der französische BPR untersucht und mit einem Nullbeton bzw. untereinander verglichen.

RESUME

La lixivation est le principal mécanisme d'autorégénération du béton. Jusqu'à présent, la relation entre la température et la lixivation n'est pas encore déterminée. Dans l'étude présentée, l'influence de la température a été examinée à 20, 50 et 80 °C. Les résultats montrent qu'une augmentation de température n'est pas toujours accompagnée d'une accélération de la lixivation. Les bétons examinés sont des bétons à hautes performances avec différents ciments et

granulats, ainsi que le BPR français. Les résultats pour les différents bétons sont comparés entre eux, ainsi qu'avec un béton de référence.

KEYWORDS: leaching, self-healing, high performance concrete, self compacting concrete

1. INTRODUCTION

The leaching of concrete and reinforced concrete is a phenomenon that is known for ages. It also has already found entry into literature as sintering or autogenous healing a long time ago. Essentially, the self-healing process is caused by a reduction of the flow rate for chemical and physical reasons. Basically, three self-healing processes are distinguished: physical, chemical and mechanical.

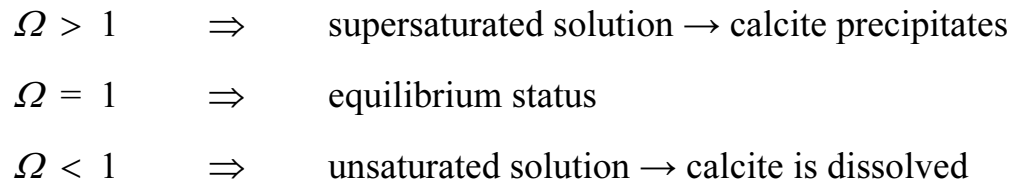
In regard to self-healing, the chemical processes seem to be the most important ones. Especially further hydration and formation of carbonate have to be mentioned here. After an extensive literature enquiry it can be suggested that the formation of calcium carbonate in fact plays the decisive part in the self-healing of cracks. The formation of calcium carbonate is affected by the leaching of the concrete. A more detailed description is found in [1].

2. THEORETICAL PRINCIPLES OF LEACHING/SELF-HEALING

Above all, the laws of mass action form the basis for the discussion of the self-healing of cracks i.e. primarily for the precipitation resp. disintegration of CaCO_3 . The occurrence of a chemical reaction depends on the concentration of the educts resp. the products (left and right side of the chemical equation). If educt and product are in equilibrium, the forward and the backward reaction have the same velocity. This equilibrium is described by so-called equilibrium constants K , that are dependent on temperature. In case of an involvement of pure solids or pure liquids in a reaction, their value is integrated into the equilibrium constants and is called solubility product K_L . Calcium carbonate mostly crystallises in the modifications calcite, aragonite and vaterite. In his tests, Plummer and Busenberg [4] detects that calcite is the thermodynamically most stable modification of the three types of crystals mentioned above, up to temperatures of 100 °C and pressures of 1 bar. It depends on the so-called supersaturation index Ω if calcium carbonate precipitates or is dissolved. The supersaturation index is a proportional value of the solubility product:

$$\Omega = \frac{[Ca^{2+}] \cdot [CO_3^{2-}]}{K_L}$$

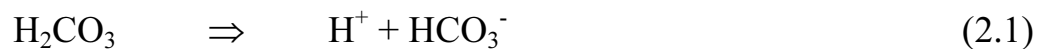
The following constellations can occur:



In a first step it shall be explained which types of chemical reactions go off during the precipitation of calcium carbonate. The following events basically take place:

The CO₂, that is available in the atmosphere below the dome of the storage, diffuses into the water and, together with the water, it is converted into carbonic acid (H₂CO₃). However, this process only takes place in very small amounts (0.1% of the whole CO₂), (H₂O + CO₂ ⇌ H₂CO₃). In literature, the share of dissolved CO₂ that did not convert into H₂CO₃ is described as CO_{2(aq)}.

The carbonic acid dissociates in two steps under formation of bicarbonate (HCO₃⁻) and carbonate ions (CO₃²⁻). In case of chemical equilibrium, the following reaction equations are valid:



Equation (2.1) describes the first, equation (2.2) the second grade of dissociation of the carbonic acid. The distribution of the carbonate species H₂CO₃, HCO₃⁻ and CO₃²⁻ in a solution containing CO₂ is determined by the level of the pH value. With the help of ionisation coefficients the single parts can be calculated. Such calculations are described in detail in [2]. The results of these calculations are pictured in the following diagram.

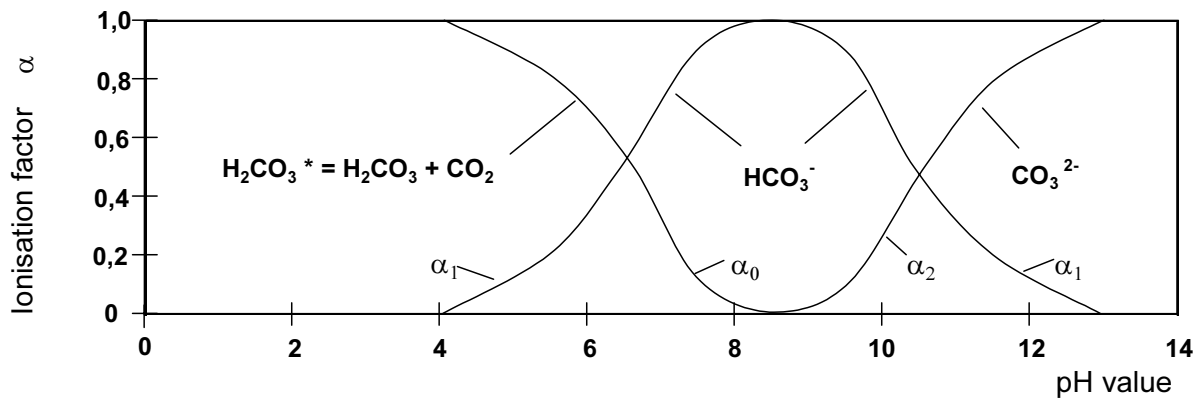
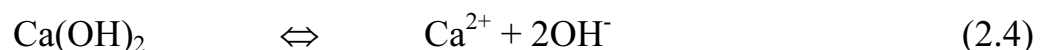


Fig. 0.1: Distribution of the various carbonate species H_2CO_3 , HCO_3^- and CO_3^{2-} as a function of the pH value at a temperature of 25 °C, according to [2]

From this chart it can be recognized that CO_3^{2-} ions can not be found in the solution until a pH value of approx. 8.3 the maximum quantity of HCO_3^- ions is available at this pH value the share of CO_3^{2-} ions surpasses that of HCO_3^- ions at a pH value of approx. 10.3 only CO_3^{2-} ions are left at a pH value of approx. > 12.3.

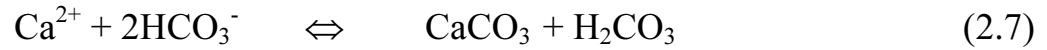
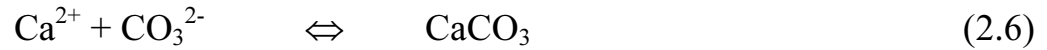
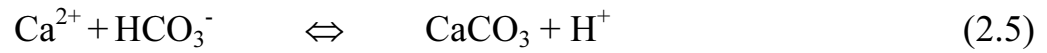
On a contact area of the concrete Ca^{2+} ions out of the calcium hydroxide ($\text{Ca}(\text{OH})_2$) go into solution. It is presumed that the water, that flows through, is in equilibrium with the CO_2 . The water can contain both bicarbonate ions and a certain quantity of Ca^{2+} ions. However, it is undersaturated ($\Omega < 1$) in regard to calcium carbonate. As cited above, in case of a permeation of the water, that contains CO_2 , into the cement stone it solutes Ca^{2+} ions from the calcium hydroxide ($\text{Ca}(\text{OH})_2$) and the CSH phases of the concrete.



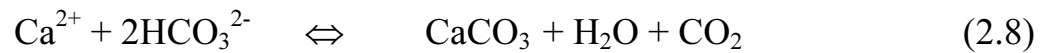
An increase of the pH value arises in this. In addition, the pore water of the concrete also mixes with the water that flows through, which also causes an increase of the pH value, because of the alkalis (KOH and NaOH) in the pore water. Due to the increase of the pH value to more than 8.3 the bicarbonates, that can be found in the water, are transformed into carbonates, c.f. equation 2.2 and fig. 2.9.

The high concentration of Ca^{2+} ions and carbonate ions CO_3^{2-} now leads to a precipitation of calcium carbonate. In principle, various reactions are involved

in the formation of CaCO_3 in the ternary system that is available now. Depending on the grade of dissociation the reactions are:



From tests, that were carried out till now, it follows that the formation of calcium carbonate is mainly due to the equation 2.6. The addition of step 1 to 4 results in the simplified sum equation of the formation of calcium carbonate:



It may be summarized in table 2.1, which external realities influence the precipitation resp. the disintegration of calcite.

Table 0.1: Influence of the content of CO_2 , the temperature and the pH value on the formation / dissolution of calcium carbonate

Precipitation of CaCO_3	Dissolve of CaCO_3
<ul style="list-style-type: none"> - falling content of CO_2 in the water - rising water temperature - rising pH value 	<ul style="list-style-type: none"> - rising content of CO_2 in the water - falling water temperature - reducing pH value

3. EXPERIMENTAL EXAMINATIONS AND RESULTS OF HIGH PERFORMANCE CONCRETE

Tested types of concrete

Various types of concrete mixings have been produced and tested for processing ability, strength as well as impermeability. Main goal was the improvement of impermeability. A reference concrete, that complied with a conventional waterproof concrete according to DIN 1045, numeral 6.5.7.2, was used by means of comparison.

Reference concrete B 35

As already emphasized in the heading, this concrete merely was used for comparison with the other types of high performance concrete. The mixing meets the requirements of a waterproof concrete according to DIN 1045.

High performance concrete (HPC)

A standard mixing, that was optimized under the aspects of impermeability, processing ability and costs in the course of the tests, was the starting point. As in other types of concrete, several components (sort of cement, additives, supplement) were varied during the tests. Altogether, five different mixings of high performance concrete were produced for the test program.

Reactive powder concrete (RPC)

Reactive powder concrete (RPC) is a concrete equipped with fiber, that is characterized by an extreme strength and impermeability. With a maximum grain of approx. 250 μm , a cement content of about 700 to 800 kg/m^3 and a w/c-ratio of < 0.20 , it already plainly differs from conventional types of concrete in regard to compound. The processing as well is completely different to that of conventional types of concrete. RPC can not be compacted and has a honey-like consistency at the end of the mixing.

Self-compacting concrete (SCC)

Self-compacting concrete was generated in Japan in 1988. In comparison to conventional concrete it distinguishes by ventilating below its self-weight and by being fluid until the levelling. It therefore does not need to be compacted by vibrax. Furthermore, SCC has three significant properties: a reduced content of

coarse additive in comparison to conventional concrete, a lower water cement ratio and an extreme yielding ability caused by highly effective yielding agents (e.g. polycarboxylates).

4. DETERMINATION OF THE LEACHED OUT QUANTITY

For the estimation of the leached out quantity of superficially dissolved compounds of Ca and Mg at contact of the concrete with hot water, cubes with a lateral length of 10 cm were leached out for 84 days for HPC no.41 and RPC in an desiccator (volume: approx. 4 liter.) with dragged-in cover. For both self-compacting types of concrete SCC 1 and SCC 2 a cylinder ($\varnothing = 10\text{cm}$, $h = 10\text{cm}$) was chosen for reasons of production engineering. The eluent was distilled water.

For the temperation, the desiccator was put in a temperature cubicle at 40, 60 and 80 °C \pm 2 °C. These temperatures were chosen, because it can be assumed that the leaching processes are accelerated by a rising temperature. A mechanical movement, caused e.g. by agitation, does not take place, yet it can be assumed that a sufficient mixing was achieved through the thermal movement in the eluent. Since it was not clear at first, if the future reservoirs should be provided with a nitrogen pad between the water surface and the roof, in order to reduce the leaching processes, the leaching tests were executed on all types of concrete both in atmospheric conditions (air) and nitrogen. During the tests in nitrogen atmosphere the nitrogen pad was regularly regenerated (all 2 to 4 days) through an addition of nitrogen. Eluent and sample were in a ratio of 3:1. For the determination of the soluble total volume of Ca and Mg, each eluent (all in all 7 eluates) was changed 1, 3, 7, 14, 21, 42, and 57 resp. 84 days after the beginning of the test and the concentration in the eluates was determined according to DIN 38406 E 22.

5. RESULTS

5.1 Leaching of HPC

It becomes plainly apparent from Fig. 5.1 that the leaching rates are highly dependent on time. With time advancing, less and less calcium is leached out. This is naturally true for magnesium as well. For better comparison the leaching rates of a conventional standard concrete (B 35) are also drawn in Fig. 5.1.

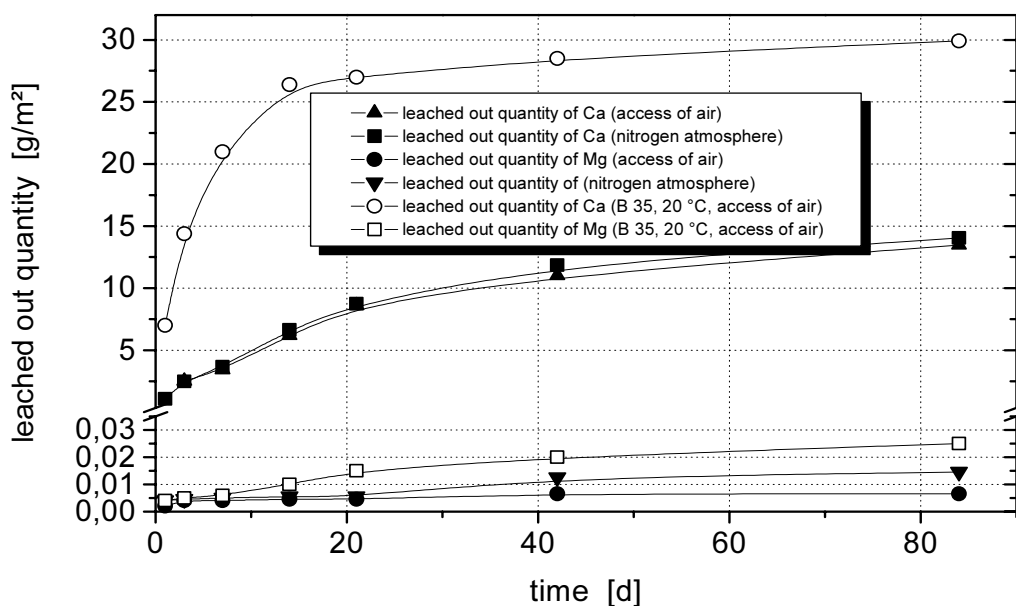


Fig. 5.1: Comparison of the leached out quantity of Ca and Mg for HPC 41 and reference concrete in case of access of air and in nitrogen atmosphere in dependency on time

The reference concrete consists of 288 kg/m³ of CEM III/A 32.5 R, 1969 kg/m³ of additive AB 16, 182 kg/m³ of water and had a thrust strength of 43.0 N/mm² after 28 days. A more precise analysis can be found in [5]. After about one month the leaching rate of Ca in high performance concrete is less than 10 g/m², the reference concrete on the other hand already has a leached out quantity of 27 g/m² - more than twice as much as the high performance concrete. Considering that the leaching tests for high performance concrete were executed at 60 °C and the comparative tests of Schießl [5] were only carried out at 20 °C, this difference becomes even more obvious. The difference concerning the precipitation rates in case of access of air resp. exclusion of air, that was expected before the beginning of the tests, could not be recognized. Therefore the differences between air and nitrogen atmosphere can be regarded as natural dispersion.

5.2 Leaching of RPC

Beneath the leaching rates for RPC in case of access of air and in nitrogen atmosphere the reference concrete has likewise been drawn in again in Fig. 2.3 for reasons of comparison.

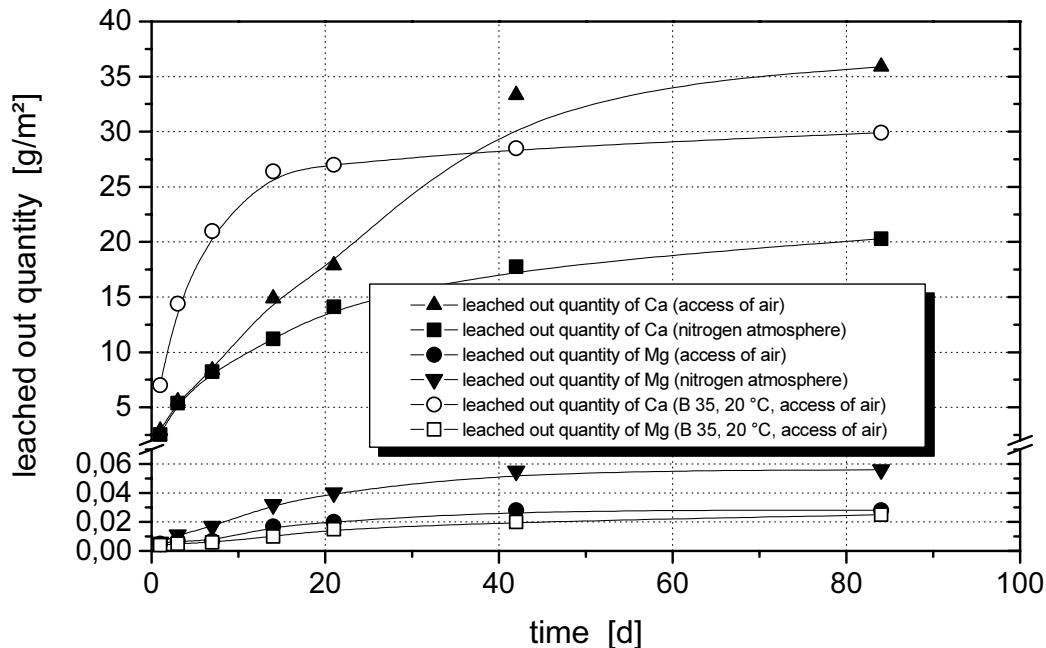


Fig. 5.2: Comparison of the leached out quantity of Ca and Mg in RPC and a reference concrete under access of air and in nitrogen atmosphere in dependency on time

RPC exceeds the value of the reference concrete, because of an extremely high leaching rate on the 42nd day. It can be assumed that without the very high 42-day-value the graph of RPC would be below that of the reference concrete. The leached out quantities of Ca in case of access of air are clearly higher than those in nitrogen atmosphere. The leaching reaction of Mg completely differs from that of Ca. In this case, the highest leaching rate for RPC can be found in nitrogen atmosphere. However, the leaching rates for Mg in case of access of air do scarcely differ in RPC and the reference concrete. Compared to standard concrete a higher leaching of Mg in RPC can be noticed. It is 0.02 g/m² for the reference concrete on the 42nd day in case of access of air, but 0.055 g/m² for RPC. As already mentioned above, the leaching tests with the reference concrete were carried out at a temperature of 20 °C. Thus, distinctly higher values should be expected for the reference concrete at 60 °C.

5.3 Leaching of SCC

Since distinctly more specimen were available for the leaching tests with self-compacting concrete and were planned on being used for the test procedure from the beginning, leaching tests were executed at various temperatures for both types of self-compacting concrete (SCC 1 and SCC 2). The test temperatures were 40, 60 and 80 °C. From the following diagram it is clearly visible that an increase of the leaching of calcium takes place in SCC 1 between 40 and 60 °C. However, between 60 and 80 °C the leached out quantity does not increase any more, but instead drops to values that are below those at 60 °C. This process can be noticed both in case of access of air and as well in case of an exclusion of air, thus in nitrogen atmosphere.

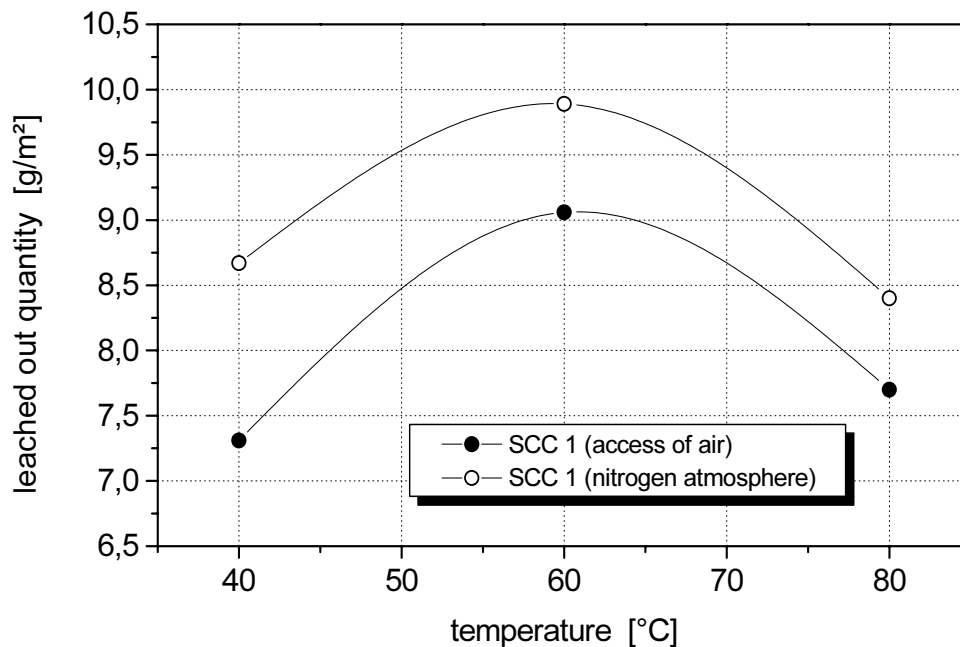


Fig. 5.3: Influence of temperature on the leached out quantity of calcium after 57 days in g/m² concrete surface in case of access of air and in nitrogen atmosphere

Furthermore it can be recognized that all values, that were measured under exclusion of air, lie above the comparable values in case of access of air. This effect could already be observed in the leaching tests with HPC no. 41. The increase of the leaching between 40 and 60 °C is approx. 25 % (57-day-value) in case of air access. At 80 °C the leaching is approx. 18 % lower than that at 60 °C and only just about 5% higher than it was at 40 °C. In a nitrogen atmosphere the final value of the measurement (57 days) for 40 °C is approx. 18 % higher than the comparable value in case of access of air. At 60 and 80 °C the final

value of the leaching in nitrogen atmosphere is yet approx. 10 % higher than the value in case of access of air. Parallel to the leaching of calcium, the leaching reaction of magnesium was tested, too. The temperature-sensitive leaching of magnesium is depicted in the following diagram. In the case of magnesium a continuous decrease of the leaching process depending on a rising temperature can be recognized. This goes for both SCC 1 and SCC 2. An influence of the nitrogen atmosphere can not be stated. It is clearly noticeable, in contrast, that at 40 °C the leaching rates in SCC 2 are approx. 1.75 times as high as those in SCC 1. Both graphs converge depending on an increase of temperature. They are nearly congruent at 80 °C. Because of the very small portions (quantum ratio Ca : Mg; > 250 : 1) and the slight pertinence for heat reservoirs other aspects concerning the leaching of Mg are not further discussed.

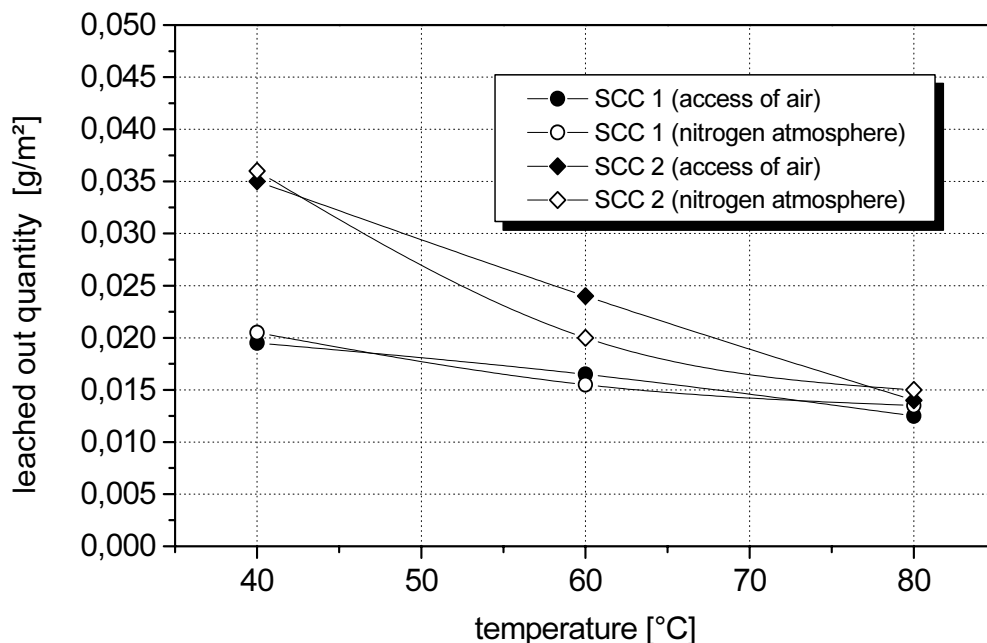


Fig. 5.4: Influence of temperature on the leached out quantity of magnesium after 57 days for SCC 1 and SCC 2 in g/m² concrete surface in case of an access of air and in nitrogen atmosphere

In the following diagram the sum graph of the leaching rates is depicted in dependency on temperature and atmosphere for SCC 1. Again clearly recognizable is the leaching maximum at 60 °C in nitrogen atmosphere. For SCC 2 a fairly similar progression appears. However, barring the leaching graph at 60 °C in nitrogen atmosphere the leached out quantity of Ca is distinctly smaller in SCC 2. Depending on the conditions it only amounts to almost 50 to 80 % of the quantum of leached out calcium in SCC 1.

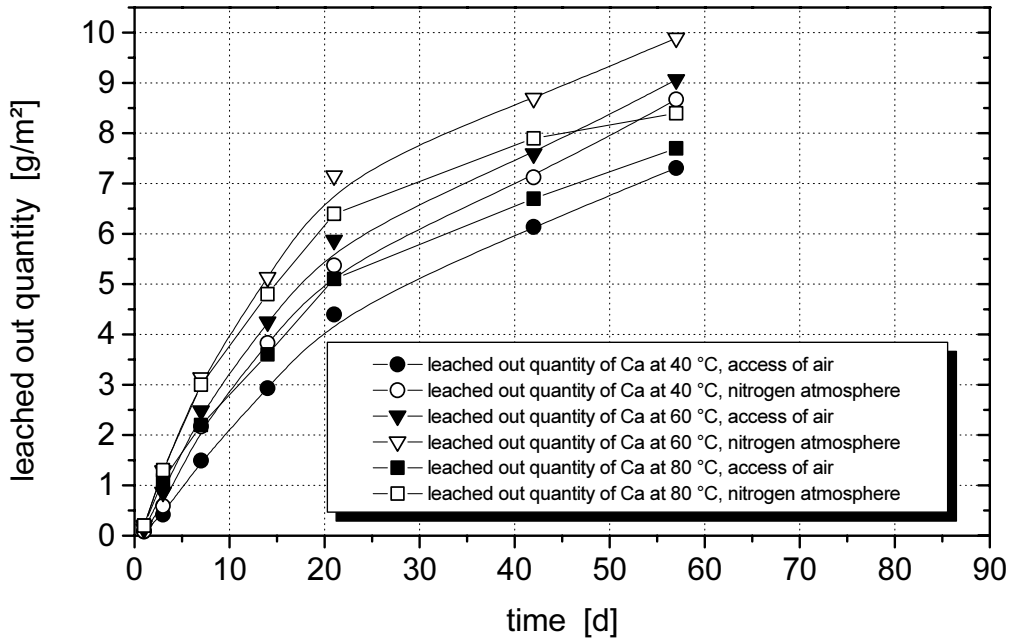


Fig. 5.5: Influence of temperature on the leached out quantity of calcium in SCC 1 in case of an access of air and in nitrogen atmosphere

6. DISCUSSION OF THE TEST RESULTS

From the tests, that were executed, and the comparisons with standard concrete in [5], definite results can be derived. The high performance concrete (HPC no. 41), that was tested at the FMFA, is distinctly less leached out than the standard concrete in [5], that was used by means of comparison. Because of the change of eluents, that was carried out during the tests, a distinctly lower initial leaching rate can be expected in case of a regular operational mode of the heat reservoir (no water exchange). In a longer term, the high performance concrete is distinctly less leached out than standard concrete, since, as time advances, the leaching process must reach deeper shifts of the concrete in which calcium hydroxide ($\text{Ca}(\text{OH})_2$) still exists. This process of diffusion is substantially slower in high performance types of concrete than in standard concrete, because of the smaller diffusion coefficient. Compared to high performance concrete RPC shows a completely different leaching reaction. The leaching of magnesium in nitrogen atmosphere is approximately three times as large as that of standard concrete and in case of access of air it is nearly as large as that of standard concrete. The leached out quantity of calcium in case of access of air, in contrast, is lower than that in nitrogen atmosphere. Altogether, the leached

out quantities of Ca and Mg are distinctly larger than those in standard concrete and in HPC no. 41.

In regard to SCC 1 it is evident that the influence of nitrogen on the total volume (per m²) of the eluated (superficially edulcorated) portions of Ca and Mg is rather small in comparison to the eluation in atmosphere (air-CO₂). However, differences can be recognized in the leaching mechanisms. In the following table the eluated total amounts are again clearly reproduced in dependency on temperature and environmental conditions for SCC 1.

A standardization of the eluated quantity on a time unit of 1 day results in Fig. 6.1. This diagram shows that after the initial wash-out for Ca is finished (after approx. 42 days) a continuous dissolving process starts. During this process about 0.1 g (-> 100 mg) of Ca per m² concrete surface are dissolved per day. There is no relevant wash-out of Mg. Only during the initial wash-out a small amount of Mg is edulcorated. The total volume of the edulcorated Mg (referring to the total test time) accounts for less than 1 % of the washed-out amount of Ca.

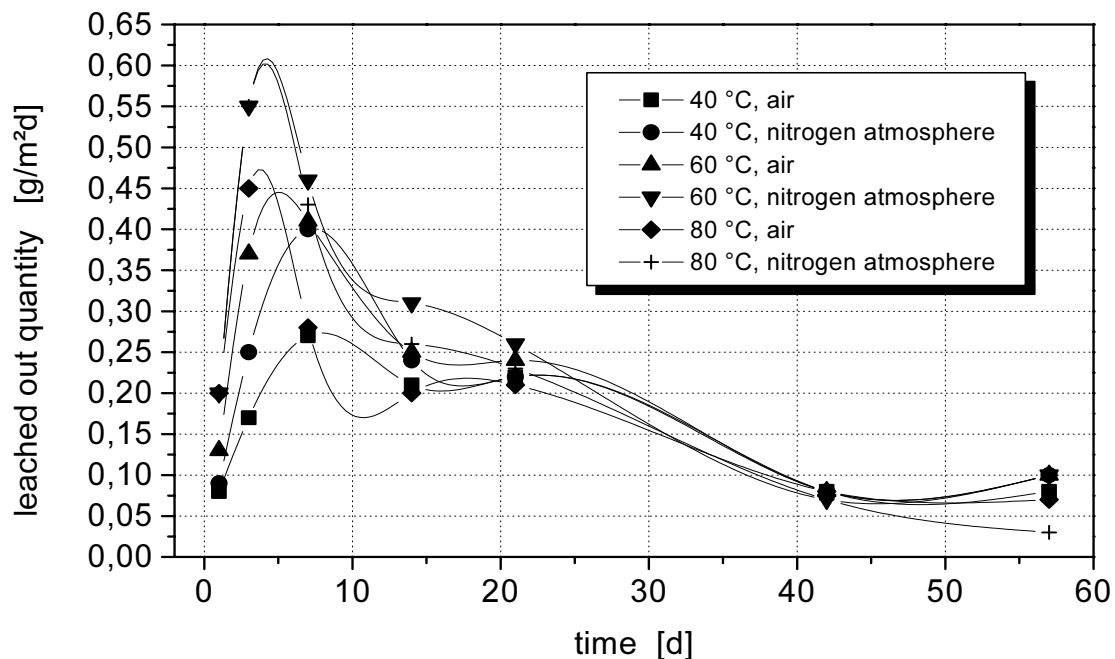


Fig. 6.1: Leaching of Ca from concrete surfaces (standardized on g/m²d) at various leaching temperatures and environmental conditions for SCC 1, according to [7]

Table 6.1: Total volume of eluated / leached out Ca and Mg per m² concrete surface in dependency on temperature and environmental conditions for SCC 1, according to [7]

Test temperature [°C]	Eluated total volume of Ca [g/m ²]		Eluated total volume of Mg [g/m ²]	
	air	nitrogen	air	nitrogen
40	7.31	8.67	0.02	0.02
60	9.06	9.89	0.016	0.016
80	7.7	8.4	0.013	0.013

Influence of temperature on the leaching process / Self-healing

Since for the operation of heat reservoirs the variants with and without nitrogen pads are considered in principal and the precipitation of calcium carbonate goes off differently, the influence of temperature on both variants shall be discussed below. The original reaction out off the concrete / cement is



The solubility, as a rule, increases in accordance with a rising temperature. However, some combinations, as for example Ca(OH)₂, show an anomaly, [3]:

- solubility of Ca(OH)₂ in cold water (20 °C) 1.85 g/l
- solubility of Ca(OH)₂ in hot water (80 °C) 0.77 g/l

The velocity of chemical reactions is a function of concentration and temperature. The solution of a salt - in this case it is Ca(OH)₂ - is a physical-chemical reaction, that goes off according to the reaction equation described above. In case of a constant concentration as it can be found in the solid (concrete), the velocity of the dissolving process rises in accordance with a heightening temperature. Regarding the solubility of Ca(OH)₂ in water, two competing parameters influence the solubility in case of a rising temperature:

- increase of velocity of the reaction resp. velocity of the dissolving process caused by an increase of temperature
- reduction of the solubility because of a reduced solubility at higher temperatures

As a result of both influencing factors, it can be expected that the solubility of $\text{Ca}(\text{OH})_2$ from solid $\text{Ca}(\text{OH})_2$ (portlandite in a cement phase) in aqueous systems will be optimized, when, because of the influence of the increased environment temperature, the increase of velocity of the solubility still is distinctly above the reduction of solubility that is caused by temperature. The measured maximum concentrations of Ca-ions in the eluates (leaching solutions), resulting from the dissolving of $\text{Ca}(\text{OH})_2$ at a temperature of approx. 60 °C, can be explained through this mechanism, (c.f. Fig. 6.2).

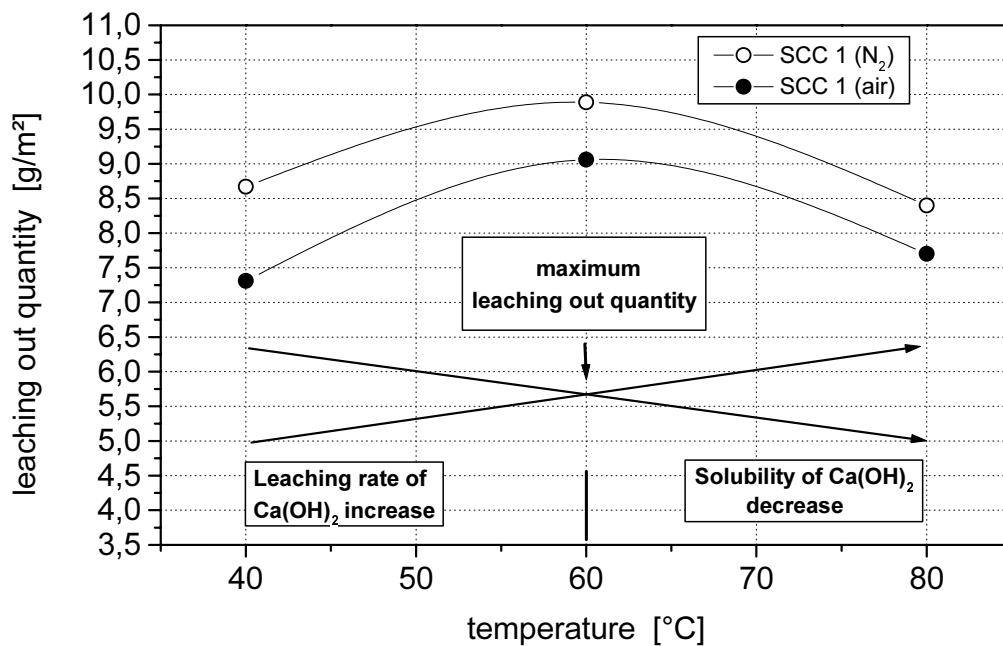


Fig. 6.2: Leaching rate of Ca for SCC 1, in dependency on temperature and environmental conditions (access of air resp. nitrogen atmosphere)

The influence of other Ca-compounds from the cement can be ignored in this context, because either the reaction velocity of the solubility resp. the solubility itself can be regarded as very low or other Ca-compounds such as calcium carbonate are only existing on a secondary scale, caused by the low carbonatisation of the concrete samples. In addition, CaCO_3 is much less soluble than $\text{Ca}(\text{OH})_2$. If no CO_2 enters the solution containing calcium hydroxide, the dissolving process is finished after the equilibrium concentration in the water is attained and no further $\text{Ca}(\text{OH})_2$ is dissolved from the concrete surface.

Table 6.2: Solubility of the various CaCO_3 - modifications in warm and cold water, according to [3]

CaCO ₃ - modifications	Solubility [g/l]	
	cold water [20 °C]	hot water [80 °C]
Calcite	0.014	0.018
Aragonite	0.015	0.019

If calcium carbonate has already formed, reactions of the solutions, that contain calcium hydroxide resp. calcium carbonate, with the CO_2 of the atmosphere occur. If air- CO_2 is now (like in the open system) filled into the aqueous solution that contains $\text{Ca}(\text{OH})_2$, the following reactions (6.2 to 6.12) have to be considered.

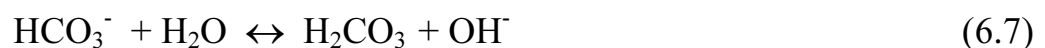
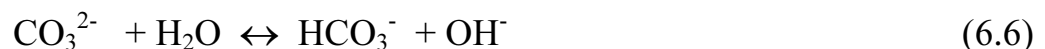
Reactions with water and carbon dioxide resp. carbonic acid



with the following other grades of dissociation:



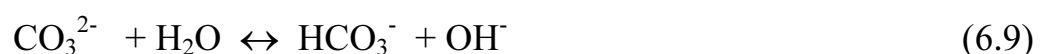
If carbonate originally is in a solution, the following reaction appears:

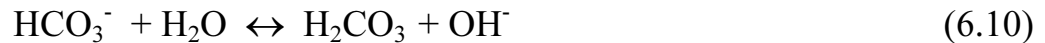


The reaction mechanisms described above also go off in solutions of limestone in water. In water, that is largely free of CO_2 (e. g. in a closed system), CaCO_3 is dissolved on very small scale:

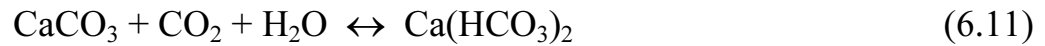


Successive reactions:





In waters, that contain carbonic acid (unsealed system), calcium carbonate reacts as follows:



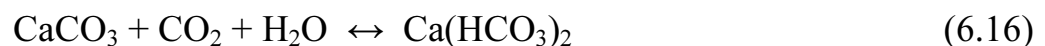
and the successive reactions (6.3) and (6.7). In case of an access of CO_2 from the atmosphere to the solutions containing Ca^{2+} and OH^- -ions, the following reaction sequences will become apparent:



Competitive reaction of (6.13)



In case of a further impact of CO_2 on the existing solution the following reaction arises:



This means that in a first step calcium carbonate (not easily dissoluble) is precipitated from the hydrous system. As a result, the solution loses most of its Ca^{2+} and further $\text{Ca}(\text{OH})_2$ is dissolved out of the concrete. In consequence of a further access of CO_2 the CaCO_3 , that was just formed, is dissolved a new according to the reaction 6.16 and, if so, a residuum of CaCO_3 can vanish; that way, calcareous water develops. If this calcareous water is heated, the reaction 6.16 in particular runs from the right to the left and CO_2 leaks from the aqueous solution, because the main quantum of CO_2 is not chemically but physically dissolved in the water and limestone either precipitates as calcite or as aragonite.

7. SUMMARY

To sum up, it can be emphasized that the reduced solubility of $\text{Ca}(\text{OH})_2$ especially plays the decisive part at an elution temperature of 80 °C. At temperatures of approx. 60 °C an acceleration of the dissolving process can be

assumed; Because of the influence of temperature, an involvement on a minor scale of the superficially existing CaCO_3 in the leaching processes can also be assumed. All in all, the solubility of $\text{Ca}(\text{OH})_2$ in the pore water of the concrete compounds will reduce at lower temperatures and a reduced leaching would be the direct consequence of this temperature effect. The very small quantities of soluble Mg also support the hypothesis that the hydroxides of both terrestrial alkali elements are reactants of the leaching processes in the main. This would also explain, why more Ca is dissolved out of the concrete surface in case of an exclusion of air (N_2 -atmosphere) than in case of air access, [7].

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