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**Carbonation of indoor concrete : measurements of depths and degrees of carbonation**

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2013

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# Carbonation of indoor concrete

Measurements of depths and degrees
of carbonation

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# 1  Introduction

The goal of the research program $CO_2$-cycle in cement and concrete was to develop a model to be able to estimate the $CO_2$ uptake in the existing concrete in a country. To accomplish that a lot of information is needed. One area of little knowledge is the carbonation of concrete indoors. Therefore it was decided that concrete houses of different ages should be tested to increase the knowledge about carbonation indoors and to give input to the calculation model.

Carbonation is the result of interaction between carbonate ions and cement hydrates. The speed and degree of carbonation is related to the type of binder and the porosity and the degree of water saturation in the pores.

The rate and mechanism of carbonation in indoor climate is known to be fast due to low humidity in the pores, but there is very little data from real cases. Most of existing data comes from laboratory experiments at low RH.

This report includes a number of investigated older structures were the emphasis has been to find out rate and degree of carbonation in indoor climate. It is assumed that the structures were made with pure Portland cement (CEM I) as mixes with other components were uncommon in Sweden in before 2000.

The structures inspected and analysed were;

1. An office building in Limhamn (Malmoe) built in 1967.
2. An office building in Kungsör (Stockholm) built in 1967 and 1977/78.
3. An office building  in Liljeholmen (Stockholm) built in 1947 and 1929.
4. An office building in Stockholm built in 1942 and 1992.
5. A garage built 1960.
6. Laboratory samples exposed to indoor climate during five and ten years respectively.

Samples were taken mainly by drill cores. The concrete material was analysed on carbonation depth by phenolphthalein and by thin sections in polarising microscope. Analyses were also made in scanning electron microscope (SEM). Relative humidity was measured in some samples. All the measurements are presented in chapter 2-7.

The degree of carbonation was measured by LECO analyses and by a new method where the samples are dissolved in nitric acid (Fridh 2013). Amount of remaining cement was indicated by thin section and analyses by semi quantitative X-ray analysis.

By impregnating the thin section by epoxy containing fluorescents dye the porosity and indirectly the strength can be revealed. By comparing the sample with reference samples it is possible to compare the porosity and to get a w/c-equivalent that can be used to estimate strength. Carbonation rates in samples of different concretes made in laboratory and kept at 20 °C is fairly well known. For a normal indoor concrete with a water/cement ratio of around 0.6 and a compressive strength of between 25 and 35 MPa gives a rate of carbonation of around 6 mm x√years (Lagerblad 2005). This value is used as a comparison reference in the report.

## 1.1 Investigated properties and used measuring techniques

### 1.1.1 Depth of carbonation

The depth of carbonation was investigated with phenolphthalein and thin sections. Phenolphthalein is an indicator liquid that changes colour from purple to white when pH drops below 9 in a water solution. The depth of carbonation is the distance from the surface to the purple front that appears during the measurements.

A thin section is a thin plate of the material that has been ground to such a thickness that it becomes transparent to light. With the help of a polarizing microscope it is possible to distinguish between portlandite (calcium hydroxide crystal) of intact cement paste and calcite of carbonated cement paste. Thus the carbonation and carbonation front can be studied in detail. One question was if the simple measurement with phenolphthalein could give adequately information to the model for calculating the $CO_2$ uptake for a country. If so there will be many more investigations available.

### 1.1.2 Degree of carbonation

The degree of carbonation, i.e. how much of the original cement that is carbonated is important for calculations of the amount of captured carbon dioxide.

A new method is used to determine the degree of carbonation. In this method a sample is dissolved in nitric acid and the increased pressure during the release of

$CO_2$ from the calcite is measured. By analysing the remaining liquid the amount of CaO can be determined and the ratio between the amount of $CO_2$ and CaO is defined as the degree of carbonation, (Fridh, 2013).

Another method used is the "LECO" method. By this method the amount of $CO_2$ and sulphate can be revealed. With knowledge of the composition of the cement the amount of CaO can be estimated from the amount of $SO_4$. This in turn makes it possible to estimate the degree of carbonation.

Both methods assume that there is no carbonate rock in the aggregate.

Another method used is X-ray diffraction. This is basically a qualitative method that reveals the crystalline minerals in the mix as a peak in a diagram. The cement components and portlandite are minerals while the cement gel is almost amorphous. The calcite formed by the carbonation process is also a mineral and gives a distinctive peak. By comparing the intensity of the peaks it is possible to give a semi-quantitative estimate of the amount of different minerals in the mix. By comparing samples from different depth it is possible to track changes in mineral quantity.

It is possible to analyse the carbon content in scanning electron microscope and analyse the composition by energy dispersive analysis. In this case a thin gold cover instead of the normal carbon cover is used.


### 1.1.3 Humidity

In some cases a moisture profile was established. The samples were drilled out without any water and placed in test tubes. During the measurement with a Vaisala sensor the tubes were stored in climate room with +20°C and 55 % RH.

# 2 Office building in Limhamn constructed in 1967

An office building built 1967 was chosen as a representative building from a time when extremely large amount of buildings were built in Sweden.

Nine cores (d= 94 mm) from different parts of the building (3 inner walls, 2 floors with one side connected to the ground, 1 floor with room climate on both sides, 1 external wall (2 cores) and 1 beam) where drilled with access to water. The cores were chosen so that the influence of surface material and construction component of the building on the carbonation could be studied. In three of the building components was also a moisture profile determined.

Some of the cores revealed other materials than mortar and concrete. These were excluded from this report.

## 2.1 Core 1: Interior wall

Wall thickness 180 mm. Concrete with binder of probably CEM I. There was 7-8 mm of mortar on the concrete surface and a painted plastic weave on surface of the mortar. A more porous layer could be found between the outer mortar and the concrete, figure 2.1.
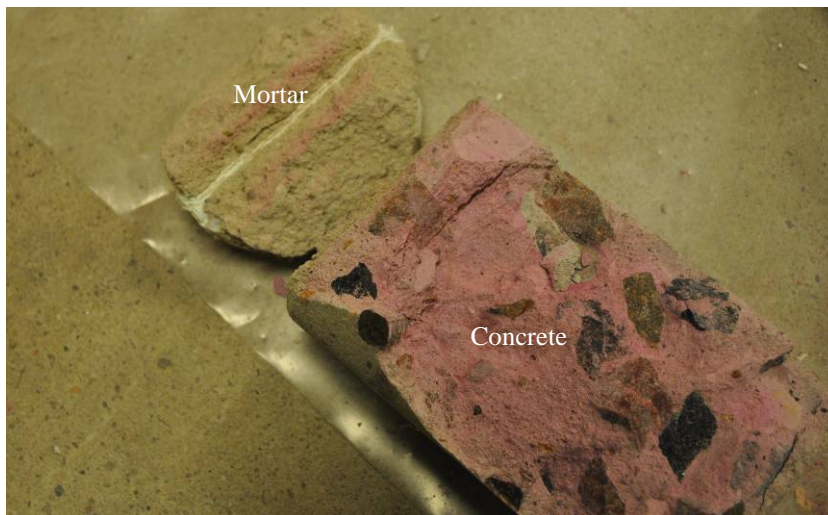


*Figure 2.1.Split Core from inner wall.*

### 2.1.1 Phenolphthalein measurement

The core was split just before applying the phenolphthalein. The pH change had not reached through the mortar to the concrete. A straight line of the phenolphthalein at the bottom of the mortar layer (7-8 mm in) was seen.

### 2.1.2 Degree of carbonation

To investigate the degree of carbonation two slices (11 mm each) were sawed from the concrete. For the slice 0-11 mm was the degree of carbonation found to be 0.22 and for the slice 16-27 mm was the degree of carbonation 0.14.

### 2.1.3 Thin section

The thin section reveals carbonation of most of the mortar, figure 2.2. At the contact to the concrete the mortar is only partially carbonated. The concrete is then carbonated to a depth of between 1 and 5 mm.

Remaining cement grains can be observed in the carbonated part of the concrete. This suggests that the concrete was allowed to carbonate before the mortar was applied. The low depth of carbonation of the concrete (1-5 mm) in part explains the low degree of carbonation. Moreover the concrete contains grains of limestone aggregates. This probably explains why the slice that according to thin section is uncarbonated between 16 and 27 show carbonation.
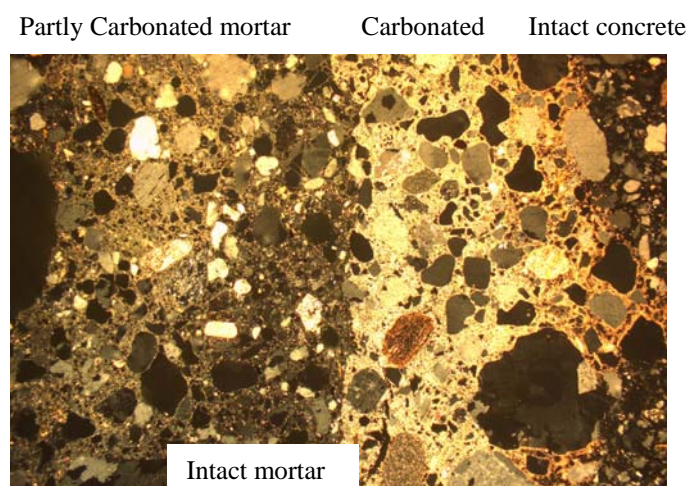


*Figure 2.2. Thin section of interfacial zone between cover mortar and concrete. The inner part of the mortar is uncarbonated while the outer part of the concrete is carbonated. This indicated that the mortar was applied after a while when the concrete was partly carbonated.*

### 2.1.4 Moisture profile

The RH was measured on samples from three depths in the wall, table 2.1. The samples were drilled out without any water present and placed in test tubes. During the measurement the tubes were stored in a climate room with +20°C and 55 % RH.

Table 2.1: RH at different depths in an inner wall.

| Depth (mm) | RH (%) |
|------------|--------|
| 0-20       | 47,5   |
| 50-75      | 48     |
| 90-95      | 48     |

### 2.1.5 Conclusions

There is a plastic weave and a mortar on the top of the concrete. We do not know when these were applied. Presumably the mortar was applied after casting of the concrete as uncarbonated mortar could be found at the contact to the concrete. A concrete without surface cover should have carbonated more than 40 mm (Lagerblad 2005).

The degree of carbonation is low. This is presumably due to remaining cement grains and the fact that most of the concrete was not carbonated. Moreover, pieces of limestone in the aggregate will indicate give a higher degree of carbonation that is actually the fact. The painted plastic weave presumably slowed down the carbonation of the mortar.

## 2.2 Core 2: Interior wall

Wall thickness and the drill core was 190 mm. Concrete was made from Portland cement, probably CEM I. On one side the concrete was covered by a mortar with a thickness of 7-8 mm. The mortar was covered with a painted plastic weave (same as for core 1). On the other side of the wall was the concrete painted and covered by glazed tile plates, figure 2.3.

*Figure 2.2. Core 2: Inner wall.*

### 2.2.1 Phenolphthalein measurement

The core was split just before applying the phenolphthalein. The depth of carbonation of the concrete was 3-4 mm. The concrete covered with paint and glazed tile plates was uncarbonated.

### 2.2.2 Thin section

The thin section was taken from concrete beneath the mortar. The depth of carbonation was between 3 to 4 mm. This carbonation is probably due to carbonation before the concrete was covered. The w/c equivalent is about 0.6.

### 2.2.3 Conclusions

The degree of carbonation of the concrete is very low. This is presumably due to the protection by the mortar and the painted weave which both hindered carbonation. Presumably the mortar was, like in figure 2.1, applied sometime after the concrete was casted. The tiles and paint on the other side of the wall stopped access to $CO_2$ and therefore carbonation.

## 2.3 Core 3: Interior wall

The wall thickness is 100 mm. This wall turned out to be of aerated concrete with mortar on the surface. The mortar was painted on one side and ceramic tiles were the surface material on the other side, figure 2.4.
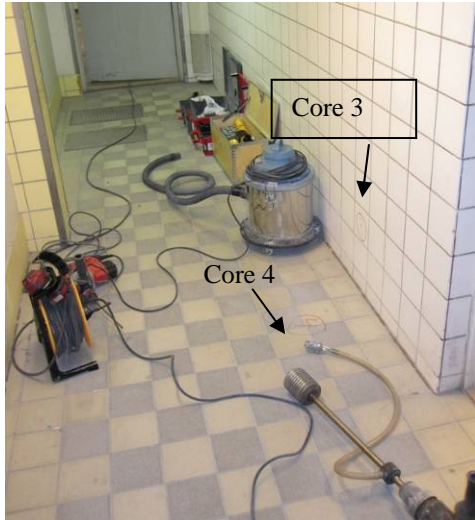
*Figure 2.3. Core 3: Inner wall.*

### 2.3.1 Phenolphthalein measurement

The core was split just before applying the phenolphthalein. Only a thin line was seen 3-4 mm from the painted surface. The material seems completely carbonated from the other surface (with ceramic tiles). There is no documentation regarding if the tiles were applied directly after completion of the wall but it is believed that the tiles was applied a long time after the wall was built.

## 2.4 Core 4: Ground floor

This floor was a construction component including many different materials. The surface material was ceramic tiles on mortar and that was followed by mortars of different kind separated by a capillary breaking layer. The second mortar layer was followed by an insulating material and an additional concrete layer which was placed on macadam, figure 2.5. The total thickness was 250 mm.

*Figure 2.4. Core 4: Ground floor.*

### 2.4.1 Phenolphthalein measurement

The core was split just before applying the phenolphthalein. The upper two layers of mortar are partial carbonated. Spots of dark pink are found in both layers, with larger areas in the lower part. The tiles have probably been applied long after the floor was constructed or that carbon dioxide from the ground wanders through the construction component and carbonates the porous part of the construction.
The lower part of the core was partially carbonated.

### 2.4.2 Thin section

The top of the core showed no indication of carbonation in thin sections.

The bottom of the core showed a patchy carbonation (see figure 2.6) to at least 45 mm (the length of the thin section). The patchy texture is in part due to bad mixing that gives an uneven porosity and thus a patchy appearance. The w/c equivalent of the dense parts is around 0.55 while that of the porous parts is above 0.7. The dense parts are mainly uncarbonated while the porous are carbonated.

20 mm  20 mm          45 mm          45 mm
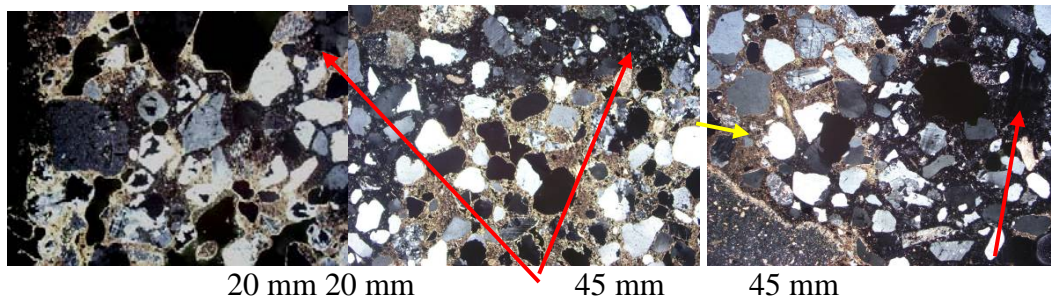
*Figure 2.6. Thin sections in polarized light. The light colour shows carbonation. It shows the patchy carbonation where carbonation follows porous parts. Yellow arrows shows carbonated patched while red arrows shows uncarbonated patches. The depth is from concrete at the bottom of the drillcore.*

### 2.4.3  Moisture profile

The RH was measured on samples from six depths of the floor, table 2.2. The samples were drilled out without any water present and placed in test tubes. During the measurement the tubes were stored in a climate room with +20°C and 55 % RH.

Table 2.2: RH at different depths in ground floor.

| Depth (mm) | RH (%) | Comment |
|---|---|---|
| 20-40 | 54,5 | |
| 50-70 | 55 | |
| 70-95 | 74 | Under internal capillary braking layer |
| 120-140 | 74 | |
| 170-185 | 86,5 | Under insulation |
| 185-200 | 86 | |

### 2.4.4  Conclusions

The tiles on the top hinder carbonation. The carbonation at the bottom of the core shows that a bottom plate above a capillary breaking layer can be carbonated. The carbon dioxide presumably comes from organic decomposition.

## 2.5  Core 5: Ground floor, painted surface

Though this core was taken in the same floor as core 4 in an adjacent room weren't the materials the same. The building component consisted of three to the eye different mortar layers which were separated by thin well defined layers. The mortar layers where followed by insulation, concrete layer and macadam, figure 2.7. The total thickness was 250 mm.

*Figure 2.7. Core 5: Ground floor, painted surface.*

### 2.5.1 Phenolphthalein measurement

The core was split just before applying the phenolphthalein. The top layer was carbonated 10 mm. The next 20 mm was partially carbonated. Layer 2 was not affected at all of the carbon dioxide when the third layer was completely carbonated. That layer was followed by a layer of insulation material that was followed by a layer of macadam. Again it seems as it is possible for concrete to be carbonated underground if it is drained. The RH is 86 %.

### 2.5.2 Thin section

The top mortar layer showed complete carbonation in 9 mm and thereafter partial carbonation 3-5 mm. The bottom of the concrete part of the core was found to be completely carbonated in 22 mm with a distinct front. This was followed by a partial carbonation for an additional 3 mm. The w/c equivalent of the concrete was about 0.65. Signs of alkali silika reaction (ASR) were found. ASR is an indication of reactive aggregates and a high humidity.

### 2.5.3 Moisture profile

The RH was measured on samples taken from five depths of the floor, table 2.3. The samples were drilled out without any water present and placed in test tubes. During the measurement the tubes were stored in a climate room with +20°C and 55 % RH.

Table 2.3: RH at different depths in ground floor, painted surface.

| Depth (mm) | RH (%) | Comments |
|---|---|---|
| 20-40 | 53 | |
| 50-85 | 64 | |
| 110-135 | 64 | |
| 170-185 | 85 | Under insulation |
| 185-195 | 86 | |

### 2.5.4 Conclusions

The results show as in core 4 that concrete above a capillary breaking layer can carbonate presumably due to organic decomposition. In this case the carbonation front is clear maybe due to a more effective capillary breaking layers or more homogeneous concrete.

## 2.6 Core 6: Intermediate floor

The construction component was 200 mm thick. The different layers were linoleum carpet, a concrete layer with smaller aggregates, and a layer with even smaller aggregates followed by more regular concrete and paint on the roof side, figure 2.8.



*Figure 2.8.Core 6: Intermediate floor.*

### 2.6.1 Phenolphthalein measurement

The core was split just before applying the phenolphthalein. The layer just beneath the linoleum carpet was carbonated to a depth of 4-5 mm. The concrete layer behind the paint on the roof side was carbonated to a depth of 70 mm, see figure 2.6.1. It is not known when the different layers on the floor side were applied.

### 2.6.2 Degree of carbonation

To investigate the degree of carbonation nine slices (11 mm each) were sawed from the concrete covered by paint on the roof side, figure 2.9.
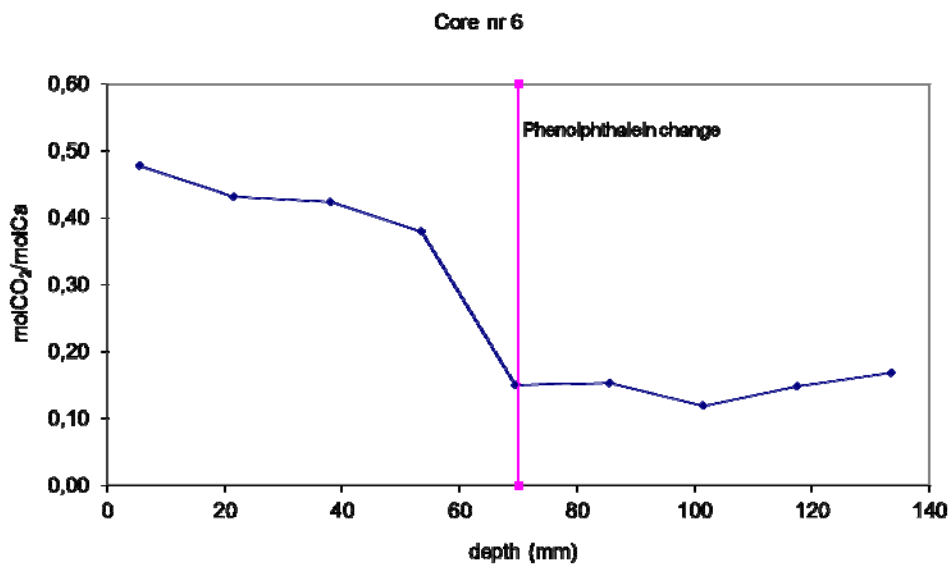


*Figure 2.9. Core 6: Degree of carbonation at different depths. Depth defined as the centre of each slice. From the pained surface and uppvards towards the floor. The concrete contains some carbonate aggregate particles giving an apparent higher degree of carbonation.*

The pH-change coincide with the drop in degree of carbonation just as (Möller, 1994) also found. For the first 60 mm is the degree of carbonation decreasing slowly from 0.49 to 0.40 and thereafter drop to 0.15 in just 11 mm. The degree of carbonation is fairly constant after that. One must however consider that the concrete contain some small carbonate aggregates and that this gives a higher degree of carbonation. Presumably the degree of carbonation in the interior of the concrete is close to 0 %. Thus the degree of carbonation at the surface is closer to 40 than 50 %.

### 2.6.3　Thin section

Two thin sections were made, one from the top (floor) and one from the bottom of the core (roof). The thin section on the upper part of core 6 shows that the carbonation under the linoleum is small.

For the thin section taken from the painted roof side the first 3 mm completely carbonated, followed by 15 mm that was partly carbonated and the remain of the thin section (20-45 mm) was vaguely carbonated, figure 2.10. This measurement agrees with the other measurements on this core but show with a greater detail how the carbonation proceeds. In the thin section it is possible to observe unhydrated cement grains in a carbonated matrix.
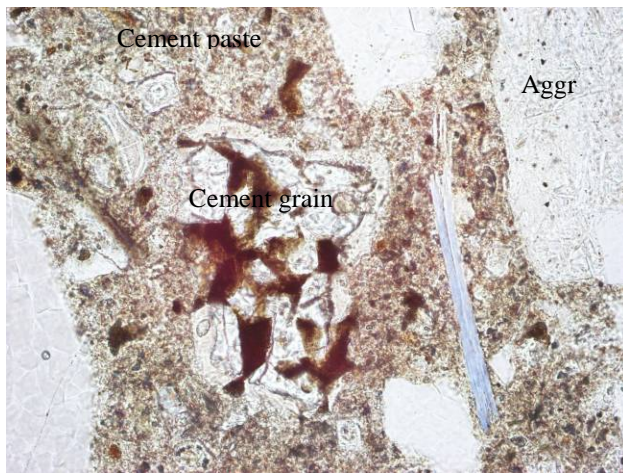


*Figure 2.10. Unhydrated cement grains in a carbonated matrix (the carbonated roof side of the drill core.*

### 2.6.4　Conclusions

The results show that carbonation is complex. The problem is that we do not know when the different layers were applied. Presumably the linoleum carpet was applied late as we know from other samples that this material hinders access to $CO_2$ gas. There are partial carbonation due to slow reactions and remaining cement grains. This will affect the measurements of degree of carbonation. The degree of carbonation is not a straight line (see figure 2.9). This is probably due to partial carbonation and remaining unhydrated cement grains. The linoleum carpet hinders carbonation from the top. One would expect larger carbonation on the roof side that is only painted.

## 2.7 Core 7 and 8: External wall

This core was taken from an external wall, figure 2.11, to be able to compare the different measurements from internal concrete with concrete exposed to outdoor climate. The core was 100 mm long and had no surface cover.
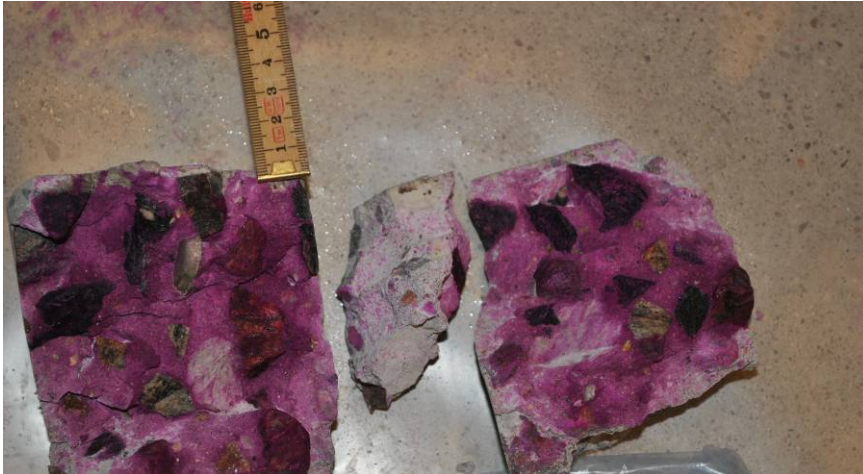


*Figure 2.11.Core 7: External wall*

### 2.7.1 Phenolphthalein measurement

The core was split just before applying the phenolphthalein. Around 2-3 mm of the outer part of the outdoor concrete was indicated to be carbonated. This is an expected result.

### 2.7.2 Degree of carbonation

To investigate the degree of carbonation two slices (11 mm each) were sawed from the concrete. For the 0-11 mm slice the degree of carbonation was 0.30 and for the 16-27 mm slice the degree of carbonation was 0.10. The concrete aggregates contain some minor amounts of limestone particles and thus the real degree of carbonation is lower.

### 2.7.3 Thin section

The concrete was carbonated about 5 mm and the w/c equivalent was 0,55-0,60. The depth of carbonations is around 5 mm. Thus the 0-11 mm slice contains both carbonated and uncarbonated cement paste.

### 2.7.4 Conclusion

A higher degree of carbonation was expected as the wetter outdoor climate is will increase the degree of hydration and thus increases the amount of paste available

for carbonation. The façade was however exposed to rain which will slow down the carbonation process.

The thickness of the tested layers where, however, larger than the carbonated layer (see below) and thus the degree of carbonation of the carbonated layer is underestimated

### 2.7.5   Core 8: Inside of external wall

The inside part of the external wall was also investigated, figure 2.12. The concrete had two layers of different paste/mortar and the outer layer was painted.



*Figure 2.12.Core 8: Inside of external wall.*

### 2.7.6   Phenolphthalein measurement

The core was split just before applying the phenolphthalein. All the paste/mortar was carbonated. 4-5 mm of the concrete was carbonated which could imply a higher carbonation rate but the time for applying surface layer is not known. The concrete could have been carbonated before the surface layers were applied.

## 2.8   Core 9: Concrete beam

A beam subjected to same indoor climate at both sides was tested, figure 2.13. The beam was 250 mm thick and pained on the three exposed sides.

*Figure 2.13 Core 9: Concrete beam.*

### 2.8.1   Phenolphthalein measurement

The core was split just before applying the phenolphthalein. 35 mm of both sides of the beam was carbonated. This could imply a paint open to carbon dioxide or that the beam was carbonated before the paint was applied.

### 2.8.2   The degree of carbonation

To investigate the degree of carbonation four slices (11 mm each) were sawed from one end of the core and ten slices (5 mm each) from the other end was investigated, figure 2.14.
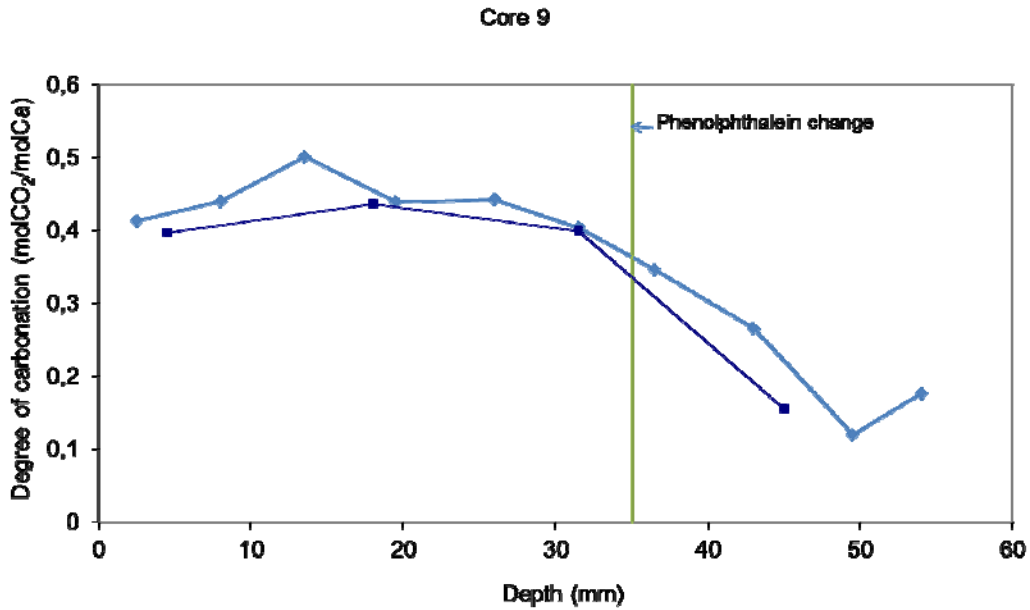
*Figure 2.14. Core 9: Degree of carbonation at different depths. Depth is centre of each slice and the figure shows two different measurements from opposite sides of the core.*

### 2.8.3 Thin section

The concrete was completely carbonated about 30 mm, partly carbonated 15 mm and vaguely carbonated for the rest of the thin section (see figure 2.15) and the w/c equivalent was 0.60-0.65. Un-reacted cement grains are found in the entire sample. The fine aggregate contains some limestone particles.
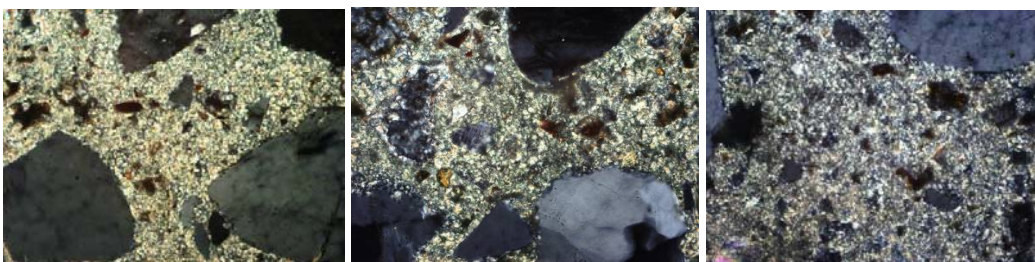


*Figure 2.15. Thin section pictures. Crossed polarizers. From the surface (left side) to a depth of 35 mm (right side). Pictures 0.3 mm. Carbonatisation can be observed by a lighter more yellow colour. From this it is possible to observe that the cement paste at the surface layer is much more carbonated that the interior.*

### 2.8.4 X-ray diffraction

The used method to determine the degree of carbonation only measures the amount of carbonated material. In indoor concrete there remains cement grains and thus it will be counted as noncarbonated. X-ray diffraction is a qualitative method but it can be used semi-quantitative. Thus values in the table do not give any absolute value but they can be compared.

Table 2.4. Semi quantitative X-ray analysis of mortar from different depth. The numbers only indicate relative peak measurements.

|                 | 5mm  | 10mm | 15mm | 20mm | 50mm |
|-----------------|------|------|------|------|------|
| Alite           | 11   | 0    | 0    | 0    | 0    |
| Alite+Belite    | 513  | 250  | 92   | 98   | 72   |
| Portlandite, CH | 0    | 291  | 1037 | 1794 | 2115 |
| Calcite         | 827  | 529  | 761  | 456  | 121  |
| Quartz          | 3707 | 4125 | 4309 | 2234 | 1335 |
| Feldspar        | 1010 | 1310 | 1289 | 865  | 828  |

### 2.8.5 Conclusions core 9

This concrete is without surface material and the same rate of carbonation is found in laboratorial concrete with the same strength. The thin sections indicate a lower degree of carbonation with depth. The measured degree of carbonation shows a higher degree of carbonation at the surface and then a falling degree of carbonation from 30 mm and deeper. This can be confirmed by the X-ray. At the surface the amount of unreacted cement is higher probably due to fast drying. This can be observed as a lower degree of carbonation in figure 2.14. Deeper down the amount of remaining cement diminish. Instead it is possible to observe remaining uncarbonated portlandite. The amount of uncarbonated portlandite increases with depth. This indicates that the cement paste contains portlandite crystals protected by layers of calcite that decreases the degree of carbonation. Even at 50 mm depth that presumably is uncarbonated concrete, small amounts of calcite can be found. This is probably small amounts of limestone (calcite) in the aggregate. In part this will give a baseline to the measurement of degree of carbonation as this is based on releases of $CO_2$ gas.

The combined data shows clearly that in indoor climate the degree of carbonation is fairly low do to the sluggish carbonation of remaining cement grains due to the low humidity in the concrete, which in turn is due to dry indoor climate.

# 3 Office building in Kungsör constructed 1967 and 1977/1978

An office in Kungsör, (140 km from Stockholm) has been investigated. One part of the building was build in 1967 and another part in 1977/1978. The basement is made from caste concrete while the house itself is made from precast elements.

The building has been inspected and a preliminary carbonation depth was established with the help of drill cuttings and phenolphthalein. Three drill cores (Ø 20 mm) have been taken from the old building and two from the new building.

In general the carbonation depth indicated by the drill cuttings and phenolphthalein was between 3 and 5 mm. This, however, is a false value as shown by the thin sections. The concrete contains remaining cement grains and the drilling was wet. Thus the remaining cement grains reacted, raised the pH and thus indicated uncarbonated concrete.

## 3.1 Drill cores from the 1967 building

Two drill cores ware taken, one from a wall and one from the floor. They are both from casted concrete.

### 3.1.1 Core from wall in cellar

There is a thin layer of paint on the concrete. The thin section gives a depth of carbonation of around 24 mm. The carbonated cement paste is seemingly more inhomogeneous than the uncarbonated. This is due to inhomogeneous carbonation. The porosity of the uncarbonated concrete is similar to a reference concrete with a w/c ratio of 0.6. The concrete is rich in paste and the carbonated concrete contains numerous unhydrated cement grains.

### 3.1.2 Core from floor in cellar

There is a thick layer of paint on the concrete. The concrete is rather porous and badly compacted. The carbonated cement paste has a more homogeneous paste than the uncarbonated. This can be seen in the thin sections when analysed in UV-light, figure 3.1. The lighter colour shows a larger amount of absorbed fluoresces dye and shows that the carbonated paste is more porous. The depth of carbonation

is 20 mm and the carbonated paste contains large amounts of remaining unhydrated cement grains.
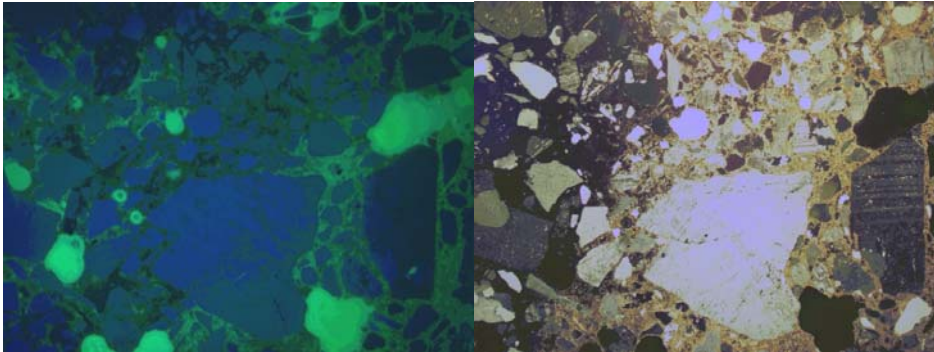


*Figure 3.1. Thin section pictures. On the left side with UV-lighth and on the right side with crossed polarisers. Carbonated paste on the right side. Picture is 5 mm.*

### 3.1.3   Conclusion

The depth of carbonation on both the samples is less than what one would expect from a 42 years old indoor concrete. According to the table in (Lagerblad 2005) one would expect a depth between 35 and 40 mm for a concrete with a w/c of around 0.6. The real depth of 20 and 24 mm indicates that the colour hinders the carbonation somewhat.

## 3.2   Drill cores from the 1987/88 building

The cores were taken from one cast floor and one pre-casted hollow beam element in the cellar.

### 3.2.1   Core from wall

**Thin section**

The depth of carbonation varies between 10 and 14 mm. The carbonated paste is more inhomogeneous than uncarbonated. The carbonated cement paste contains remaining cement grains, figure 3.2.
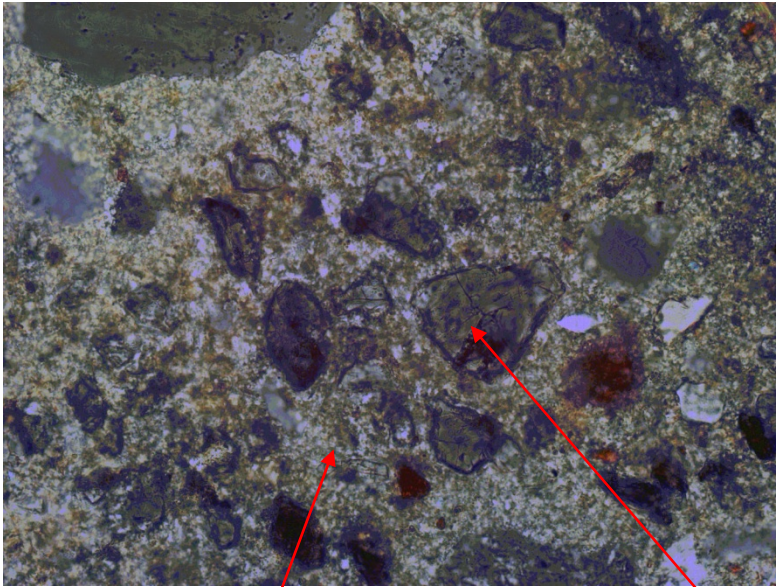
*Figure 3.2. Carbonated cement paste with remaining unhydrated cement grains.*

### 3.2.2 Hollow beam

**Thin section**
The depth of carbonation is around 10 mm but with a diffuse front.

**X-ray diffraction**
The X-ray diffraction explains the diffuse carbonation front. Remaining cement grains can be found in the whole section. Likewise portlandite grains covered by calcite shells can be observed. Table 3.1 shows X-ray diffraction data. X-ray diffraction is a qualitative method but it can be used semi-quantitative. Thus values in the table do not give any absolute value but they can be compared.
The amount of portlandite is less at the surface indicating that the carbonation of portlandite is a slow progressing process.

Table 3.1. Semi quantitative X-ray analysis. Quartz and feldspar are from aggregate grains in filler. Thin section do not reveal limestone in the aggregate.

|  | Outer surface | Surface at hollow | Inside surface | Inside hollow | Central part |
|---|---|---|---|---|---|
| Alite & belite | 49 | 85 | 139 | 76 | 77 |
| Portlandite, CH | 131 | 710 | 690 | 740 | 784 |
| Calcite | 1320 | 565 | 352 | 234 | 213 |
| Quartz | 2923 | 6664 | 5119 | 3131 | 4061 |
| Feldspar | 1515 | 1560 | 2768 | 929 | 1199 |

**LECO**

A LECO analyses gives the amount of carbon dioxide and sulphate in a sample. The amount of sulphate can be used to calculate the amount of cement. It needs cement with known composition.

Outer 5 mm;                $SO_3 = 0.31$    $CO_2 = 5.24$

5-10 mm;                   $SO_3 = 0.44$    $CO_2 = 2.32$

When calculated on the basis of the old house building cement (CEM I) Slite Std that presumably was used it gives a degree of carbonation of 122 % for the outer part and 39 % on the inner part. The result from the outer part is unreasonably high while that of the inner is reasonable. The high degree of carbonation of the outer part could be due to a carbonate aggregate grain, adsorbed $CO_2$ or an error in the contents of sulphate. In any case it shows that the values must be evaluated carefully. Considering that there remains cement grains and Portlandite a degree of carbonation of 39 % is reasonable.

### 3.2.3   Conclusions

The concrete of the walls in the old building is 42 years old. The w/c ration is around 0.6 and the concrete according to the drawings should be a K30. It can be assumed that the concrete cube strength is between 30 and 40 MPa. This gives a rate of carbonation of around 3.7 mm x √years. The expected value lies around 5 mm x √years (Lagerblad 2005). This indicates that the colour delays the carbonation.

The concrete at the floor gives a rate of carbonation of 3.1 mm x √years. This is somewhat lower than on the walls although the concrete is more porous. This may be due to thicker colour and a higher relative humidity from the ground.

In the new building the rate of carbonation is 2.6mm x √years. This is a somewhat lower rate, which may be due to a somewhat denser concrete.

The examination shows that carbonation in a dry indoor climate is complex. There remains uncarbonated cement grains and portlandite. This is probably due to a lack of water to hydrate. The amount of portlandite declines with depth but it is not totally transformed to calcite. This is probably due to remaining portlandite in the core of new calcite compounds.

# 4   Buildings at Liljeholmen in Stockholm constructed in 1947 and 1929

One of the investigated buildings, constructed in 1947, is used as an office. The other building is used as a warehouse and was built in 1929. In 1929 vibrated concrete was not used and thus floors are tamped. The walls are more homogeneous and were probably made of loose concrete that did not need vibration which was used at that time.

## 4.1   Office building from 1947

### 4.1.1   Interior wall

The concrete is covered by painted weave on a mortar. Thin sections show that the 10 mm thick mortar is partly carbonated to5 mm. The underlying concrete is fully carbonated 10 mm and partly carbonated 22 mm. Presumably it is an old wall that later was cover by mortar, weave and paint.

### 4.1.2   Floor

The top of the floor is covered by plastic linoleum carpet. The drill core is through the floor. Below the concrete slap there is a capillarity breaking layer made of coarse aggregate.

Thin section shows that the w/c equivalent in is 0.55-0.60, figure 4.1. No carbonation below the linoleum carpet. From below, above the capillarity breaking layer, the concrete is carbonated for 5 mm and partly carbonated another 5 mm. It is also possible to observe carbonation at the rim of air bubbles.
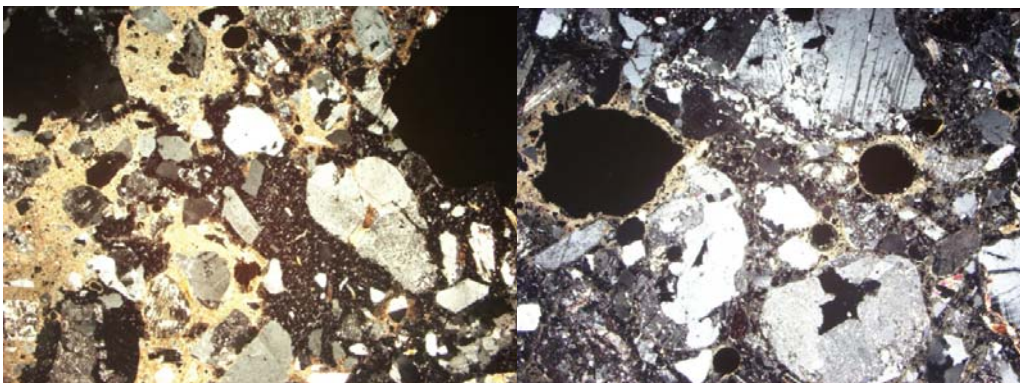


*Figure 4.1. Thin section photos. Crossed polarisers. To the left, patchy carbonation front. To the right carbonation at air bubbles.*

### 4.1.3 Conclusions

Tiles and linoleum carpets hinders carbonation. Paints and weave slows down carbonation. Concrete above a capillarity breaking layer do carbonate. This is presumably due to release of carbon dioxide from organic decomposition.

## 4.2 Building from 1929

The building is today used as a warehouse.

### 4.2.1 Floor

The floor is partly covered with fat and oil. The drill core did not reach the bottom. It seems to be a tamped concrete. The concrete is very inhomogeneous with a mixture of dense and porous parts. Analyzed in thin sections the concrete is totally carbonated around 5 mm. Below this it is carbonated in bands and zones. Lumps of cement are uncarbonated. The cement was coarse grained. The sample shows that oil and fat hinders carbonation. In an inhomogeneous concrete the carbon dioxide ingress flows porous zones.

### 4.2.2 Wall

The wall was not painted. The drill core was taken through the 150 mm thick wall. The concrete has a w/c equivalent around 0.6. The cement paste contains remaining unhydrated large cement grains. Two thin sections from both sides were made. Both showed total carbonation to a depth of 45 mm.

### 4.2.3 Floor

No surface material. The core did not reach the bottom. Fairly clean surface. Badly compacted concrete with lumps of cement. Generally carbonated to a depth of at least 45 mm. Patchy carbonation where the partly hydrated lumps of cement remain uncarbonated.

### 4.2.4 Wall

Drill core only covers one side. No surface cover. The porosity is equivalent to a new concrete with a w/c of 0.60-0.65. Large cement grains remains. The depth of carbonation is more than 45 mm.

### 4.2.5 Conclusions

Fat and oil hinders carbonation. With poorly compacted concrete the carbonation becomes patchy and carbonation follows steaks and zones with higher porosity.

The concrete in the walls without surface cover has a rate of carbonation of around 6 mm x√years. This is close to that of a laboratory concrete.

# 5 Samples from a building in Stockholm

The main building was constructed in 1942. The samples were taken from a part that was constructed in 1992 during a renovation. Samples were taken from a stairwell and beneath wooden parquet in a new room.

## 5.1 Drillcore 1

Wall in stair case. It is covered with paint. Phenolphthalein gives a depth of carbonation of 3-6 mm.

### 5.1.1 Thin section

The depth of carbonation is 5-8 mm, figure 5.1. Unhydrated cement grains remains. The concrete contain natural mainly granitic aggregate. The w/c equivalent is around 0.60-0.65. Rather homogeneous.



Intact front carbonated paste surface
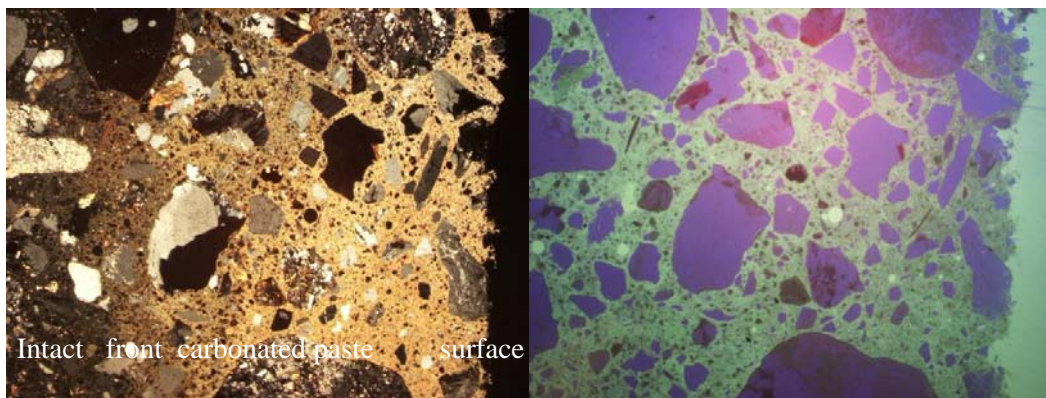
*Figure 5.1. The picture to the left is with crossed polarizers and show the carbonation front. The picture to the right show the same area is uv-ligth and indicates a homogeneous paste with no porosity difference over the carbonation front. The pictures are 4.8 mm.*

## 5.2 Drillcore 2

Floor in staircase constructed in 1992. Covered with paint. The phenolphthalein changes colour at a depth of between 10 and 19 mm.

### 5.2.1 Thin section

The depth of carbonation is between 9 and 20 mm. The w/c equivalent is around 0.6. The concrete is homogeneous.

## 5.3   Drillcore 3

Concrete covered by wooden parquet. There is also a plastic sheet between the concrete and the parquet. The room was constructed in 1992 and the parquet was installed the same year. The phenolphthalein changed colour at a depth of 1-2 mm.

The thin section showed a depth of carbonation of 1-2 mm, figure 5.3. The carbonation is somewhat deeper at cracks and in the interfacial zone between paste and stones.
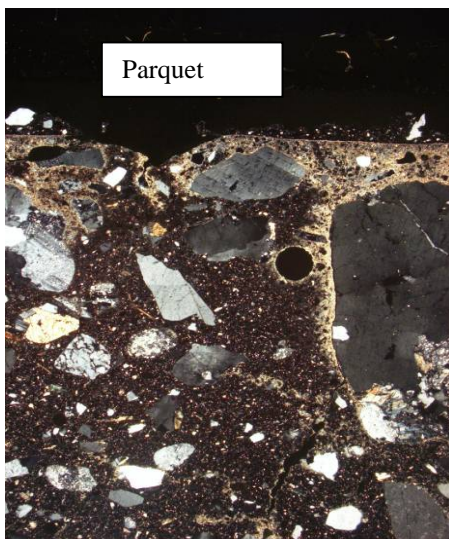


*Figure 5.3. Concrete under parquet. Crossed polarisers. The lighter colour is carbonation.*

## 5.4   Conclusions

The three concretes are from 1992. In the staircase the depth of carbonation is larger at the floor than the wall. The type of paint is different with the thickest at the floor. The depth of carbonation is higher at the floor. This shows that there is a large variation as regard to the ability to block carbon dioxide between different paints. The plastic sheet and the parquet seem to block carbon dioxide almost completely.

# 6  Two samples from a garage built 1960.

The house was built in the late 1960ies. The drill cores come from a bearing column and from the roof in a warm garage. They are covered by a thin paint.

## 6.1   Drill core 1

The compressive strength of the concrete in the column is 48 MPa. Thin sections show a depth of carbonation of 23 mm for the column. The concrete is homogeneous. If the concrete is 40 years this will give a rate of carbonation of 3.6 mm x √years for the column. .

## 6.2   Drill core 2.

The compressive strength of concrete in the roof is 43.5 MPa. Thin section gives a depth of carbonation of 32 mm for the roof. If the concrete is 40 years this will give a rate of carbonation of 5 mm x √years for the column. The rate of carbonation of both drill cores is somewhat faster than expected (Lagerblad 2005).

# 7 Laboratory samples

The laboratory samples are made at CBI in Stockholm. After casting the samples were stored in water for one week and then put in a climate chamber at 20 °C with an RH of 50 %.

## 7.1 Results

### 7.1.1 Sample 1

5 years old. 433 kg CEM I. W/C = 0.48. The cube strength at 28 days was 53 MPa. The depth of carbonation /thin section) was 7 mm. This gives a speed of carbonation of 3.1mm x√years.

### 7.1.2 Sample 2

10 years old. 360 kg CEM I. W/C = 0.47. The cube strength at 28 days was 68 MPa. The depth of carbonation (thin section) was between 8 and 10 mm. This gives a speed of carbonation of 2.8 mm x√years.

### 7.1.3 Sample 3

5 years old, 433 kg CEM I. W/C = 0.81. The cube strength at 28 days was 17 MPa. The depth of carbonation (thin section) was between 10 and 11 mm. This gives a speed of carbonation of 4.7 mm x√years.

The laboratory samples give expected values (Lagerblad 2005). The rate of carbonation in sample 3 should be somewhat larger. The rate of carbonation goes down with increasing strength/porosity.

## 7.2 SEM data from laboratory samples

The SEM (scanning electron microscope) analyses were made with highly polished samples covered with a thin layer of gold, table 7.1. As chemical calibration for carbon pure calcite crystals was used. The data shows that the degree of carbonation varies. A porous gel is more carbonated than a dense gel. When a crystal with the shape of portlandite was analysed its shows that the outer part is more carbonated than the interior. This shows that carbonation is a sluggish reaction.

Table 7.1 Analysis of ordinary carbonated concrete. Backscatter analysis in SEM.
Normalised to 100 % oxides. Calc = Composition of pure crystal.

| Sample | Calc. | 2 | 2 | 2 | 2 | 1 | 1 | | |
|---|---|---|---|---|---|---|---|---|---|
| Analysis spot | CC | Filler CC | Dense gel | Porous gel | Ordinary paste | CH inner | CH outer | Dense gel | Porous gel |
| $CO_2$ | 44 | 42.3 | 41.1 | 54.8 | 47.9 | 25.9 | 40.8 | 47.9 | 71.9 |
| $SiO_2$ | | 1.3 | 23.6 | 15.8 | 17.2 | 3.3 | 5.1 | 16.6 | 10.4 |
| CaO | 56 | 56.4 | 35.4 | 29.4 | 35.0 | 70.8 | 54.0 | 35.5 | 17.6 |
| $CaO/CO_2$ | 1.3 | 1.3 | 0.9 | 0.5 | 0.7 | 2.7 | 1.3 | 0.7 | 0.2 |
| $CaO/SiO_2$ | | | 1.5 | 1.9 | 2.0 | | | 2.1 | 1.7 |

## 7.3   Discussion

The rate of carbonation agrees well with other laboratory data (Lagerblad 2005) and gives the rate of carbonation for concrete without a surface material. The RH measurement of the house in Limhamn shows that the RH inside the concrete is close to 50 % RH and thus the concrete should carbonate at this speed if not covered by something.

The SEM data confirms the results from thin section and X-ray diffraction. At dry indoor climate carbonation is a sluggish process. The carbonation of C-S-H is sluggish and porous parts of the paste carbonate faster than dense ones. Moreover, the interior of the portlandite crystals remain uncarbonated for a considerable time.

# 8 Discussion

The degree of carbonation and carbonation depth of indoor concrete is fairly complex. In the investigated buildings and in the concretes the RH is around 50 %. At this RH the concrete will be rather dry and the hydration will presumably stop or be very slow. Thus the cement paste contains a large amount of unhydrated cement grains. The degree of hydration presumably depends on the curing and conditions during the construction stage. In some cases the amount of unhydrated cement grains is larger at the surface than in the interior of the concrete. This is presumably a result of faster drying at the surface than in the interior.

The results clearly show that unhydrated cement grains carbonate very slowly. This is presumably due to that cement grains have to hydrate before they carbonate. As the RH in indoor climate is low the speed of hydration is slow and thus the carbonation.

In some cases the unhydrated cement grains gave false depth of carbonation when measured with phenolphthalein. If there was some available water these cement grains hydrated and indicated lower depth of carbonation. This shows that one has to be very careful with using cuttings.

Thin sections and SEM give the most accurate information about carbonation depth. The carbonation is kinetically controlled and the carbonation front is diffuse. At the inner front one can observe a diffuse patchy carbonation. The cement paste is, however, only partly carbonated. Dense C-S-H and portlandite contain cores of uncarbonated material. Successively the cement paste becomes more and more carbonated.

*Degree of carbonation and depth of carbonation*
The investigation showed that a depth of carbonation measured through an indicator fluid such as phenolphthalein together with the degree of carbonation gives a good input to a $CO_2$ uptake model. The investigation show that the depth of carbonation measured with phenolphthalein also coherences with the drop in the degree of carbonation. If the pH-change displaced as a rather distinct front, was equal to the beginning, middle or end of the decrease in degree of carbonation can these measurements not say exclusively when evidence of all three possibilities appeared.

Due to the large amount of remaining cement grains and only partially carbonated cement hydrates the degree of carbonation is fairly low. In general it seems to be less than 0.5 for the normal indoor concrete at the outer part and decreasing inwards. This is what can be expected considering the large amount of remaining cement grains and portlandite in seemingly carbonated concrete. It seems therefore appropriate to calculate with a degree of carbonation of 0.3-0.5 for an indoor concrete.

A very interesting result was that concrete under ground showed significant carbonation. This is probably due to organic decomposition in the ground. The RH is in the measured concrete plates is around 85 %. This shows that the concrete above the macadam is fairly dry and that the capillary pores allows passage of $CO_2$. If the concrete was wet the carbonation would be much less. The rate of carbonation can be fairly high as the partial pressure of $CO_2$ can be higher than in normal air.

Another important thing is that most concrete surfaces are covered with some materials. The results showed that surface layers had a significant influence on the carbonation depth which was expected. Dense layers resulted in very small carbonation depths and no surface layers resulted in significantly larger carbonation depths. The concrete floors in residence or office buildings are in most cases covered by different carpets, ceramic tiles or by parquet. These materials hinder most of the carbonation. The walls are normally painted of covered by wall paper (with glue).

In Table 8.1 are carbonation depths of a collection of samples shown. The samples are those without plaster or thick cover. In brackets on the K-value (mm x √years) of the concrete and of expected values from a concrete without surface cover (see chapter 1). The results show that this concrete and laboratory concrete has k-values close to that expected while the paint hinders carbonation somewhat. Fat and oil do also block some carbonation. As can be seen in the CBI-house the blocking is not related to the thickness but to the type of colour. This is also argued by Johansson (1992). The data do not give exact values as we do not know either the type of colour or concrete quality. Two ways of handling different surface covers are presented in Lagerblad (2005) and Nilsson (2011).

Table 8.1. Compilation of carbonation data. K-values are calculated on inner carbonation front. Weakly carbonated are marked by + xx mm. K-values in bracket are expected rate of carbonation of 6mm x √years (Lagerblad 2005)

| Construction detail | Surface cover | Concrete details | Age | Depth of carbonation | k-value (expected) |
|---|---|---|---|---|---|
| Limhamn | | | | | |
| 2.4. Under floor. | Naked. On macadam | Inhomogeneous. RH 86 % | 42 | 45 mm | 6,9 |
| 2.5 Floor. | painted | Homogeneous RH 55% | 42 | 9+4 mm | 2,0 (6) |
| 2.5 Under floor | Naked. On macadam | Homogeneous. w/c eqv. 0,65 RH 86% | 42 | 22+3mm | 3,9 |
| 2.6 Under floor. | Painted | Inhomogeneous w/c-eqv 0,5 | 42 | 5 + 15 mm | 4,5 |
| 2.9 Floor beam | Painted | Homogeneous w/c-eqv. (0,65) | 42 | 30 + 15 mm | 6,9 (6) |
| Kungsör | | | | | |
| 3.1 Wall in cellar. | Painted | Homogeneous w/c-eqv. 0,6 | 42 | 24 mm | 3,7 (4) |
| 3.1 Floor in cellar | Painted | Inhomogeneous. | 42 | 20 mm | 3,1 (4) |
| Vall in cellar | Naked | Homogeneous w/c-eqv. 0,55 | 22 | 12 mm | 2,6 (2,5) |
| Garage | | | | | |
| 6Underground garage | Naked | Homogeneous. Strength 48 MPa | 40 | 23 mm | 3,6 (3,5) |
| 6Underground garage | Naked | Homogeneous Strength 43,5 MPa | 40 | 32 mm | 5,5 (3,5) |
| Liljeholmen | | | | | |
| 2.2 Under Floor | Naked. On macadam | Homogeneous | 62 | 5+5 | 1,3 |
| 4.2.1 Floor topp | Fat & Oil | Tamped concrete | 80 | 5+ 20 mm | 2,8 (6) |
| 4.2.2 Wall | Naked | Homogeneous w/c-eqv. 0.6 | 80 | >50 mm | > 5,6 (6) |
| 4.2.4 Wall | Naked | Homogeneous, w/c-eqv. 0,65 | 80 | >50 | >5,6 (6) |
| Stockholm | | | | | |
| 5.1 Wall | Painted | Homogeneousw/c-eqv. 0.6 | 18 | 7+2 | 2,1 (4) |
| 5.22 Floor topp | Painted | Homogeneous w/c-eqv. 0.6 | 18 | 9-20 mm | 3,5 (4) |
| Laboratory | | 50 % RH | | | |
| 1 | Naked | Homogeneous w/c 0.48 Strength 53 MPa | 1,5 | 7 mm | 3,1 (3,5) |
| 2 | Naked | Homogeneous. w/c. 0.47 Strength 68 MPa | 10 | 8-10 mm | 2,8 (3) |
| 3 | Naked | Homogeneous. w/c 0.81 Strength 17 MPa | 3,5 | 10-11 mm | 4,7 (9) |

# 9 Conclusions

Concrete carbonates rather fast in dry indoor climate. The speed of carbonation, however, is dependent on the type of cover. Linoleum carpet, parquet and ceramic tiles hinder carbonation effectively. Paint is slowing down carbonation. The effect depends on the type of colour.

The indoor concrete in general seems to contain large amounts of unhydrated cement grains. Moreover, the carbonation process is sluggish and the portlandite closer to the carbonation front is only partially carbonated. On an average it seems like less than half of the available CaO is carbonated. A degree of carbonation of around 0.3-0.5 seems appropriate.

# Referenser

*Lagerblad B,(2005) 'Carbon dioxide uptake during concrete life cycle– State of the art', CBI 2:2005*

*Nilsson L.O (2011) 'A new model for $CO_2$-absorption of concrete structures' reportTVBM-3158, Division of Building Materials, Lund University, 2011*

*Möller J (1994) 'Measurement of carbonation in cement based materials' P-93:11, Department of Building Materials, Chalmers University of Technology, 1994*

*Fridh K (2013) 'A method for measuring the degree of carbonation in concrete', TVBM-7215, 2013*

*Johansson (1992) 'Surface protection for concrete- a literature study' (In Swedish), CBI 1992:1*