

# Influences of sampling and collecting methodology for obtaining concrete dust in order to determine the chloride content

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## Abstract

Steel bar corrosion induced by chlorides is one of the main mechanisms leading to the degradation of concrete structures, with chloride content having a determinant value for the useful life of such structures. Sampling and the methodology for collecting concrete powder dust can influence the determination of chloride content.

The present study is based on an experimental campaign that sought to assess the influence of sampling and of the methodology of collecting concrete powder in determining the chloride content. For this, slabs of conventional concrete were produced, with and without addition of chloride, to extract cores. From these cores, concrete dust was collected by two methods: cutting and crushing, and dry grinding, with the proportion of aggregate in the test area of each specimen of concrete being evaluated. The influence of the dust collection method in the concrete was also assessed, gathering chloride content values for the two methods used and comparing them to the chloride content obtained by the sum of the chloride content of each constituent of the concrete.

The present study contributed to increase the knowledge of the determination of the chloride content from different methodologies for collecting hardened concrete dust, constituting a starting point for the establishment of future experimental campaigns to quantify the mass of the concrete dust sample for the considered depth.

The results showed that, for depths where the segregation has no effect, the average of at least 5 determinations led to a good estimate of the chloride contents of the concrete.

**Key words:** Concrete, steel corrosion, chloride content, concrete dust collecting methodology.

## 1. Introduction

The use of reinforced concrete as a construction material is due to its capacity of being shaped, and tensile strength provided by the incorporation of steel bars in its interior, associated with a low manufacturing and production cost.

Steel bar corrosion induced by chlorides is one of the mechanisms leading to the degradation of concrete structures, particularly those inserted into a marine environment, those subjected to icing salts and pool structures.

According to Tuutti, the evolution in time of the degradation of reinforced concrete structures, due to corrosion of steel bars, can be described by two periods – initiation and propagation.

The initiation period is characterized by no loss of functionality of the structures. In this stage two main factors can occur: decreased alkalinity of the concrete film that protects the steel from corrosion by reaction with carbon dioxide (CO<sub>2</sub>) in the atmosphere (carbonation), and the presence of a sufficient amount of chlorides, contained in the concrete or from the exterior of the structure. These phenomena may occur simultaneously in concrete or isolated causing the destruction of the passive layer of the steel, which prevents corrosion [1].

The cement type, concrete composition, water/cement ratio and curing affect the pore structure of the concrete, influence the initiation period. Using CEM III or CEM IV, with low water/cement ratio, with proper curing, provides better resistance to chloride ingress, thus leading to greater initiation period [1], [2].

The propagation period occurs between the destruction of the passive film, starting the active corrosion due to electrochemical reactions in the pore solution of concrete that produce steel corrosion, and reaching an unacceptable limit of concrete performance. During this period, the degradation of the concrete due to corrosion is visible [1].

Corrosion due to chloride ingress occurs by steel bar pitting, although general steel bar corrosion can occur if the level of contamination is very high, and therefore, causing the complete destruction of the passive film. The corrosion rate of carbonation is usually between 20 and 50 µm/year as chloride-induced corrosion is between 50 and 100 µm/year for good quality concrete, and 100-500 µm/year for low quality concrete [3].

The chloride concentration profile in concrete from the exposed surface inwards is a valuable tool for assessing the risk of corrosion of reinforcement steel in concrete structures exposed to marine or deicing salt environments. From the knowledge of the chloride profile of a structure at a given age, one can get some qualitative information about the rate of ingress of such ions into the structure, and then predictions about the reinforcement corrosion onset time can be formulated [4].

Several research studies have been conducted on the resistance to penetration of chlorides in mortar to be used in the repair of reinforced concrete structures, revealing the beneficial effect of the addition of polymers as cementitious modifiers due to its high performance in increasing the resistance to penetration of chloride in cementitious mortars [5] [6] [7].

The scope of this work is to study the influence of sampling and of the methodology for collecting concrete powder dust samples to determine the chloride content, used to create a chloride concentration profile. For this study, concrete samples were cast with and without the addition of

chloride, to extract cores. From these cores, concrete dust was collected by two methods: cutting and crushing, and dry grinding, with the proportion of aggregate in the test area of each specimen of concrete evaluated. The influence of the dust collection method in the concrete was also assessed, gathering chloride content values for the two methods used and comparing them to the chloride content obtained by the sum of the chloride content of each constituent of the concrete.

## 2. Materials

The cement used was CEM I 42.5 R, according to NP EN 197-1.

Two kinds of gravel and two kinds of sand were used to produce the concrete. Their maximum size is 22,4; 10; 2 and 0,5 mm and the fineness module is 8,26; 6,81; 3,56 and 1,96 respectively.

For the concrete with chloride addition, sodium chloride with 99,5% purity was used.

## 3. Concrete mixing and curing

Four slabs of 300 x 300 x 100 mm<sup>3</sup> and six cubes of 150 mm were cast in metal molds. Half of the slabs and the cubes were cast with concrete with chloride added in the mixing water (CL), and the other half were cast with the concrete without chloride (OPC). Both concretes produced have the same mix design, shown in Table 1, with the exception of the sodium chloride added in the mixing water of the concrete with chloride addition. The slump of both concretes was 130 mm.

Table 1 – Concrete mix proportion

Mix proportions (Kg/m <sup>3</sup> )	
Gravel 2	376
Gravel 1	624
0/4 Sand	577
0/2 Sand	194
Cement	374
Water	187
W/C Ratio	0,50

The maximum chloride content by mass of cement indicated in NP EN 206-1 is 0.4% for concrete with steel reinforcement for exposure classes XC, XF and XA and 1% for the concrete without reinforcing steel or other embedded metals, for all classes of exposure [8]. The value of the content of chlorides in the concrete made in the laboratory was set between 0.8 to 1%, as it corresponds to a situation where steel bar corrosion is assumed to take place. The chloride content of the OPC was 0,04% and that of the CL was 0,84%.

After the casting process, the molds were covered with a plastic sheet and the concrete was removed from the molds after 24 hours. The slabs and cubes were then stored in a saturated chamber, allowing a moist curing. The duration of the curing process was 7 days for the slabs and 28 days for the cubes.

## 4. Test methods

According to RILEM, there are three methods for sampling procedures [4]:

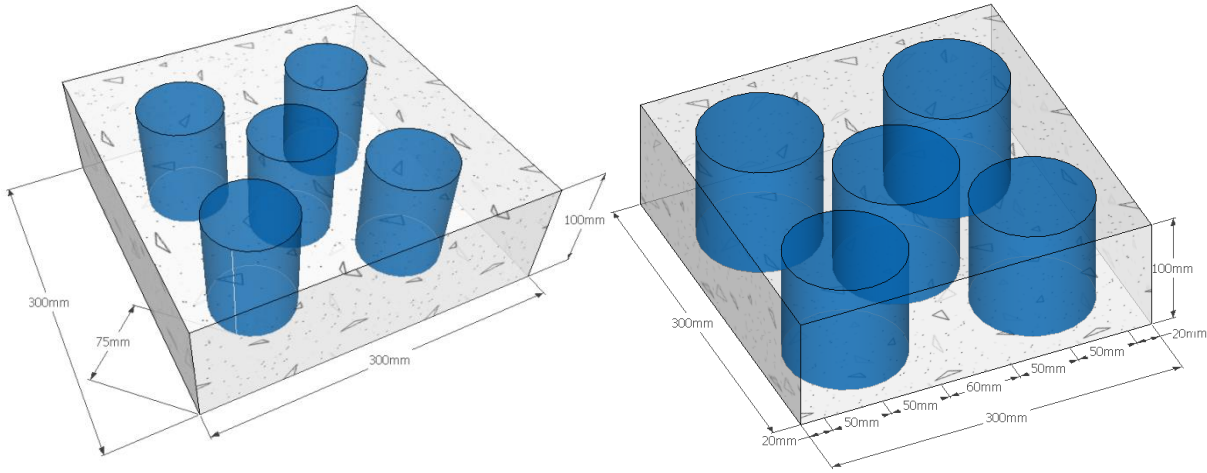
Dry drilling method - sampling by drilling with a rotary hammer and a masonry bit, working directly on the surface of a concrete specimen or real structure. This procedure allows to obtain powdered samples corresponding habitually to depth steps of about 5 mm [4];

Grinding method - sampling by grinding the surface of a concrete specimen or real structure [4];

Cutting and crushing method - sampling by saw cutting concrete slices of adequate thickness, usually not less than 10 mm, from a specimen or core, and posterior crushing of the slices, manually (mortar and pestle) or mechanically (ball or ring mills), to get the powdered sample [4].

For this work two methods were chosen: the cutting and crushing method and the grinding method.

Five cores were extracted from the CL concrete slabs for each method, and one control core for the cutting and crushing method was extracted from the center of the OPC concrete slab. The cores, with different diameters: 75 mm for cutting and crushing and 100 mm for dry grinding, were located at approximately the same position in the slabs, to avoid potential factors such as segregation, which could influence sampling. The core extraction location for the cutting and crushing method and for the grinding method are shown in Figure 1.



Cutting and crushing core extraction location (Ø 75mm)      Dry grinding core extraction location (Ø 100mm)  
Figure 1 – Location of the cores extracted from the CL concrete.

The top 2 mm of all the cores was removed, as recommended by several studies: LNEC FE01 recommends at least 1 mm [9]; NT BUILD 443, at least 1 mm [10]; and CEN/TC51 N953 recommends removing a 10 mm layer of the concrete surface [11].

### Cutting and crushing method

For the cutting and crushing method, two slices of each core were cut with 10 mm thickness: one near the concrete surface, at a depth of 2 to 12 mm, and the other at half of the depth, 45 to 55 mm, as shown in Figure 2. These slices were crushed with a jaw crusher, providing a sample of about 100 g of crushed concrete for each slice. The samples were prepared for chemical determination of chloride content, according to LNEC FE01-2013 section 8 [9]. First the size of the sample was reduced through quartering, thus obtaining a smaller sample of about 20 to 35 g. This sample was passed through the 125- $\mu$ m sieve, and the fraction retained was crushed into a fine powder in a ball mill. This fine powder was passed through the sieve, and the small fraction retained was crushed with the mortar and pestle, until the entire sample passed through the 125- $\mu$ m sieve.

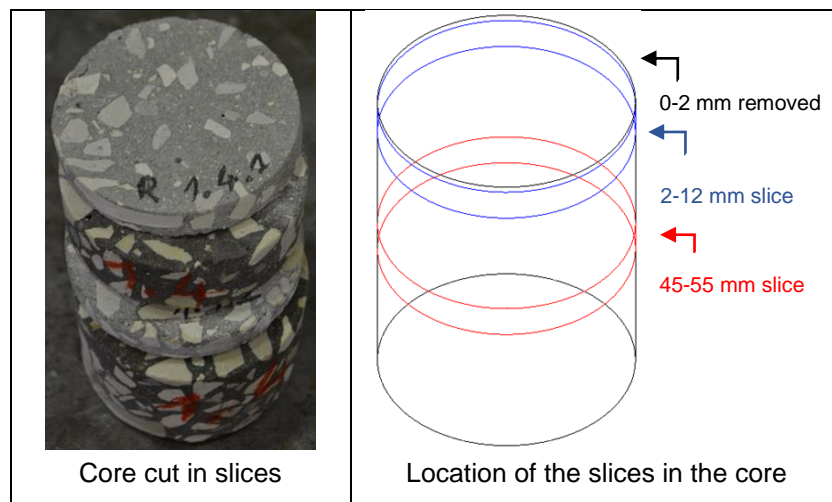


Figure 2 – Cores of the cutting and crushing method

### Grinding method

For the grinding method, the cores were placed in the grinding device, with the commercial name of Profile Grinder Kit – PF1100, of Germann Instruments A/S (GI), to collect the dust from a 10 mm layer, at depth of 2 to 12 mm, as shown in Figure 3. After grinding this layer, the cores were cut, to allow grinding of another 10 mm layer, at the depth of 45 to 55 mm, as shown in Figure 3. This grinding equipment performs a circular grind of 73 mm diameter, thus giving results comparable to the 75 mm diameter of the cutting and crushing method. After the concrete dust was collected from all grinded layers, about 90 g per sample, these samples were passed through the 125  $\mu$ m sieve, according to LNEC FE01-2013 section 8 [9]. Because all the samples from the grinding method consisted in a fine powder, there was no need for quartering. The fraction retained on the sieve was crushed in a ball mill and passed again through the sieve. The small fraction retained was crushed with the mortar and pestle, until the entire sample passed through the 125- $\mu$ m sieve.

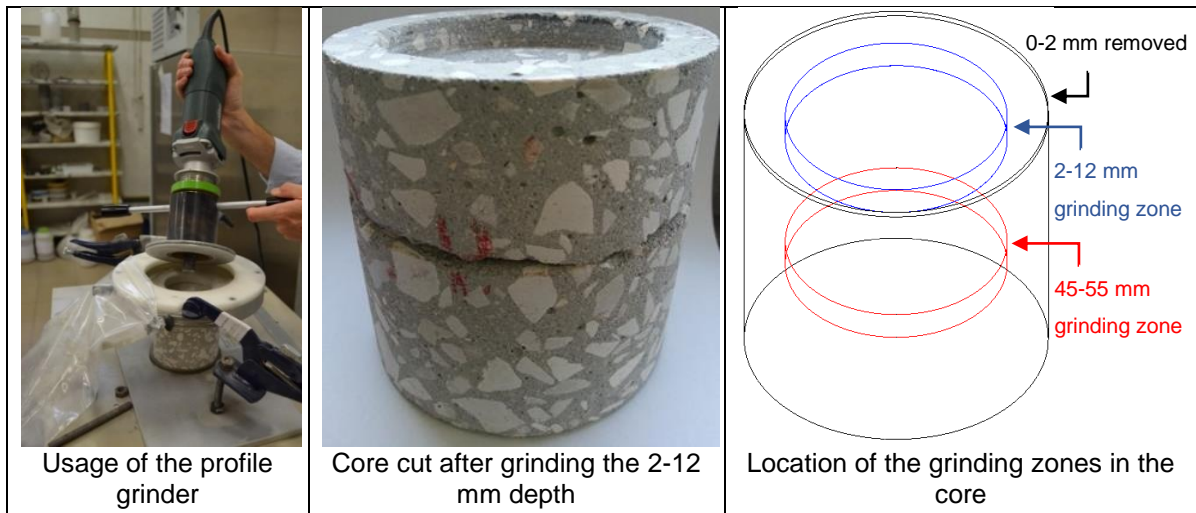


Figure 3 - Cores of grinding method

### Chloride contents determination

The samples from both the cutting and crushing and the grinding methods were placed in a ventilated oven to constant weight ( $105 \pm 5$ ) °C and subsequently cooled in a desiccator to room temperature, according to EN 14629 section 4.2 [12]. A smaller testing sample with ( $5.00 \pm 0.05$ ) g was extracted, from the prepared samples, to determine the total content of chloride. The chemical analysis was performed, according to EN 14629 section 4.3 [12], and the chloride content by mass of sample, determined by potentiometric titration method with a solution of silver nitrate with a concentration of 0.05 mol/l, was calculated using Equation (1):

$$CC = 3,545 * f * \frac{V_3 - V_4}{m} \quad (1)$$

CC – total chloride content, by mass of sample (% of mass of sample);

$V_3$  – volume of the silver nitrate solution used in the titration (ml);

$V_4$  – volume of the silver nitrate solution used in the blank titration (ml);

m – mass of concrete sample (g);

f – molarity of silver nitrate solution.

The six 150 mm cubes were tested to determine their compressive strength, according to NP EN 12390-3 [13]. The compressive strength of the cubes was 49.1 MPa.

Two 150 mm diameter cores were extracted from the OPC slab to determine the dry density of the hardened concrete, according to NP EN 12390-7 [14]. The dry density of the hardened concrete was 2229 kg/m<sup>3</sup>.

## 5. Results and discussion

With the photographic coverage of each side of the slices, before and after crushing, and the grinding areas, before and after the grinding process, and using the software Autocad 2014 to draw polylines around the aggregates, the aggregates area for each face, Agr (mm<sup>2</sup>) was determined. The average aggregate area Agr<sup>average</sup> (mm<sup>2</sup>) was calculated from the Agr of the top and bottom surfaces, for the slices of the cutting and crushing method, and from the Agr of the grinding areas, before and after the grinding process. Figure 4 shows an example of the methodology followed to obtain Agr and Agr<sup>average</sup>, for CL concrete and OPC concrete, using the cutting and crushing method. Figure 5 shows an example of the methodology followed to obtain Agr and Agr<sup>average</sup>, for CL concrete, using the grinding method. The values of the aggregate area of the concrete (Agr) were inferior at the depth of 2 mm compared to the 12 mm depth, and average aggregate area values (Agr<sup>average</sup>), at the depth of 2-12 mm were inferior compared to the 45-55 mm depth. This is an expected effect due to segregation occurring near the molding surface.

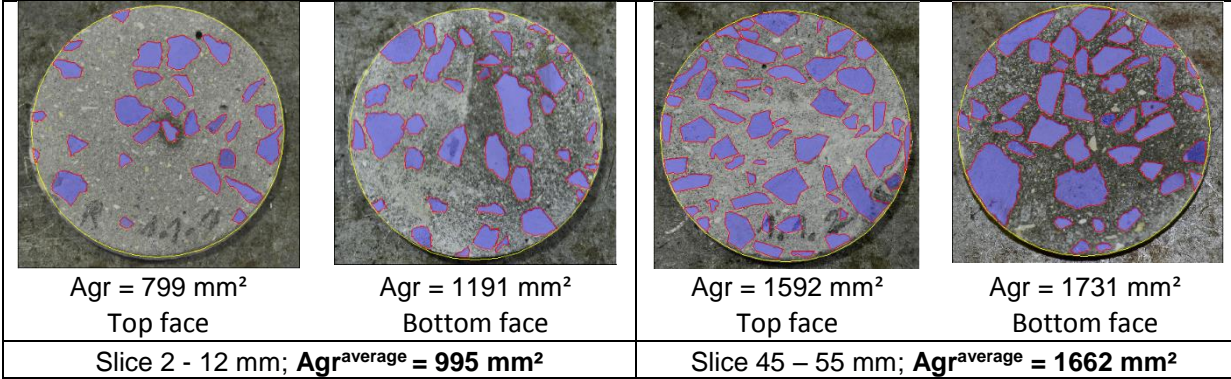


Figure 4 – Example of the methodology used to obtain Agr and Agr<sup>average</sup> for the cutting and crushing method

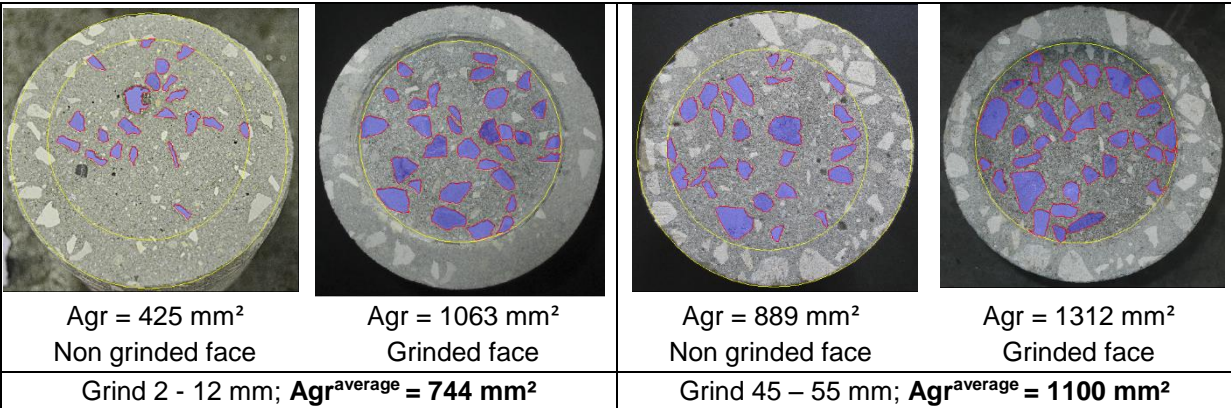


Figure 5 – Example of the methodology used to obtain Agr and Agr<sup>average</sup> for the grinding method

Considering the testing areas, A<sub>test</sub> (mm<sup>2</sup>), for the cutting and crushing method, with 75 mm diameter, A<sub>test</sub>=4418 mm<sup>2</sup>, and for the grinding method, with 73 mm diameter, A<sub>test</sub>=4185 mm<sup>2</sup>, and Agr<sup>average</sup>, the percentage of mortar area by testing area was calculated. The mortar area is shown in Table 2, for the CL concrete, and in Table 3 for the OPC concrete.



The individual results of the chloride contents by mass of cement and by mass of concrete for the CL concrete are shown in Table 2, as well as the average values for each depth (n=5) and the average value for each method (n=10). The chloride contents by mass of cement was calculated by multiplying the results of the chloride contents (% mass of concrete) by the quotient of the density of the dry hardened concrete (2229 kg/m<sup>3</sup>) and the cement dosage (374 kg/m<sup>3</sup>).

Table 2 – Chloride content and mortar area by testing area of the CL concrete

	Cutting and crushing method				Grinding method			
	Sample	Chloride content (% concrete mass)	Chloride content (% cement mass)	Mortar area by testing area (%)	Sample	Chloride content (% concrete mass)	Chloride content (% cement mass)	Mortar area by testing area (%)
Depth 2-12 mm	1.1.1	0,19	1,15	77	2.1.1	0,20	1,17	82
	1.2.1	0,20	1,21	78	2.2.1	0,22	1,32	83
	1.3.1	0,19	1,15	84	2.3.1	0,21	1,25	86
	1.4.1	0,18	1,06	74	2.4.1	0,20	1,21	81
	1.5.1	0,17	1,03	71	2.5.1	0,20	1,21	83
	<b>Average (n=5)</b>	<b>0,19</b>	<b>1,12</b>	<b>77</b>		<b>0,21</b>	<b>1,23</b>	<b>83</b>
Depth 45-55 mm	1.1.2	0,12	0,73	62	2.1.2	0,15	0,90	74
	1.2.2	0,13	0,79	62	2.2.2	0,13	0,76	68
	1.3.2	0,14	0,83	65	2.3.2	0,12	0,69	63
	1.4.2	0,13	0,75	60	2.4.2	0,13	0,75	65
	1.5.2	0,12	0,73	64	2.5.2	0,12	0,74	64
	<b>Average (n=5)</b>	<b>0,13</b>	<b>0,77</b>	<b>63</b>		<b>0,13</b>	<b>0,77</b>	<b>67</b>
<b>Average (n=10)</b>	<b>0,16</b>	<b>0,94</b>	<b>70</b>		<b>0,17</b>	<b>1,00</b>	<b>75</b>	

The individual results of the chloride contents by mass of cement and by mass of concrete for the OPC concrete are shown in Table 3.

Table 3 - Chloride content and mortar area by testing area of the OPC concrete using the cutting and crushing method

	Sample	Chloride content (% concrete mass)	Chloride content (% cement mass)	Mortar area by testing area (%)
Depth 2 - 12 mm	3.1.1	0,01	0,05	71
Depth 45 - 55 mm	3.1.2	0,01	0,04	58
<b>Average (n=2)</b>		<b>0,01</b>	<b>0,05</b>	<b>65</b>

Figure 6 illustrates the variation of the individual results of the chloride contents at 2-12 mm and 45-55 mm depths for both methods used. It should be noted that the individual results included in Figure 6 are ordered as Table 2, starting with the five values from 2-12 mm followed by the values from 45-55 mm.



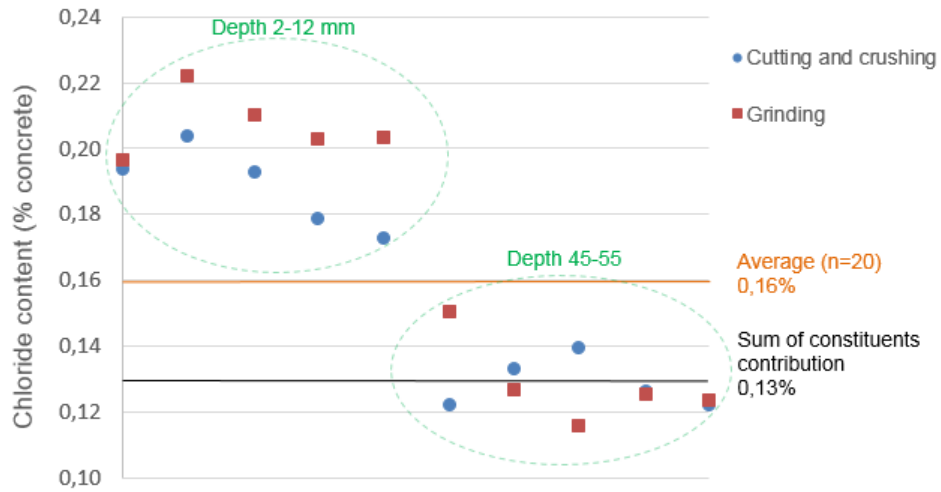


Figure 6 – Influence of the concrete dust collecting method

Figure 7 illustrates the variation of individual results from the chloride contents as a function of the area of mortar in the test area for the two methods of collecting from hardened concrete.

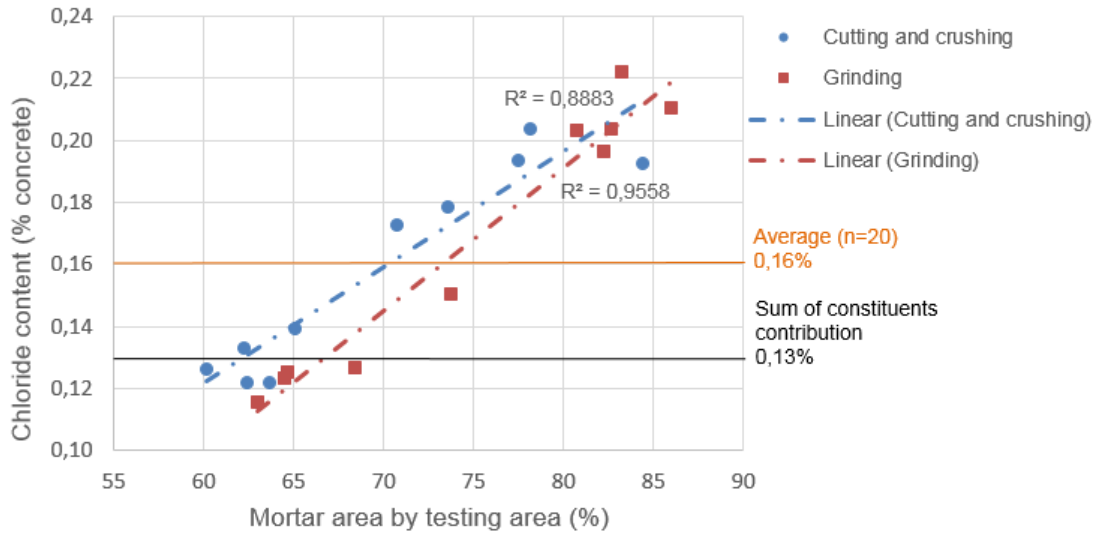


Figure 7 – influence of the cement paste area on the chloride content

Figure 8 illustrates the variation of the result of chloride content (mean value of n individual results) as a function of the number of individual results considered (n) for the two methods.

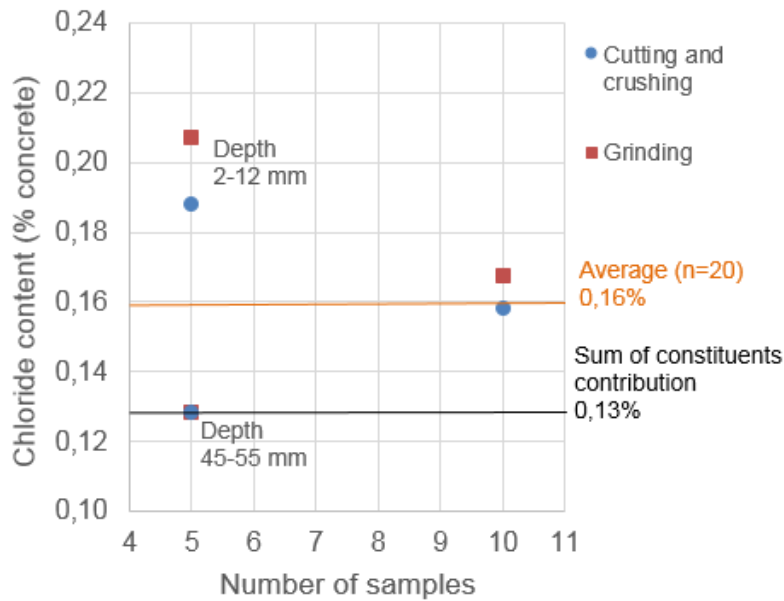


Figure 8 – Influence of the number of samples on the chloride content

From the analysis of Figure 6 to Figure 8, the following aspects can be emphasized:

1. The chloride contents obtained in the two depths differ clearly, the higher values having been obtained in the depth 2-12 mm, closer to the surface (Figure 6). This is due to the segregation of the paste close to the finishing surface, accumulating and retaining a greater amount of paste and/or mortar (Figure 7);
2. For the 2-12 mm depth (Figure 8), the difference in chloride contents between the sum of the chloride constituents contribution (0.13%) and those obtained by the cutting and crushing method (0.19%) was 0.06%, and for the grinding method (0.21%) was 0.08%. For the 45-55 mm depth the difference was null, allowing the following conclusions: i) both methodologies (sampling + collecting method) did not interfere on the chloride contents at depths  $\geq 45$  mm, allowing the correct estimation of the chloride contents; ii) the cement paste segregation inherent to the finishing face loses its effect at depths  $\geq 45$  mm;
3. The results obtained by the cutting and crushing method are inferior to those obtained by the grinding method in the 2-12 mm depth (Figure 6, Figure 8). This may result, in part, from the fact that the cutting of the slices was carried out with water, which can contribute to leach the chlorides in the concrete. However, in the 45-55 mm depth, the results obtained by both methods were practically the same, which suggests that leaching tends to decrease with the reduction of the chloride contents;
4. The percentage of the area of mortar in test area is directly proportional to the chloride content in the concrete, which is expected because the aggregates used in the concrete have only a very small amount of chlorides (0.01 % or less);
5. The analysis of Figure 7 and Table 2 reveals that the samples used in the grinding method have a higher percentage of mortar area in the test area. This occurrence also contributes to explain the difference in the results obtained by the two methods;

6. The results at the 45-55 mm depth confirm that the consideration of the average of all the individual values for each methodology (n=5) led to same value as obtained by the sum of the chloride constituents contribution. This leads to the conclude that, for depths where the segregation effects do not occur, the average of at least 5 determinations will lead to a good estimate of the chloride contents;
7. At the 2-12 mm depth the chloride contents expressed in percentage of mass of cement, estimated from the density of dry hardened concrete and the cement dosage, led to values higher than the sum of the constituents contribution (0.84%). However, at the 45-55 mm depth, a lower value of 0.77% was obtained, allowing the conclusion that chloride migration to the surface exists due to the exudation of the mixing water, associated to the wall effect, as there is an aggregate settlement with mortar concentrating in the surface, due to flattening when casting.

## 6. Conclusion

The present study contributed to increase the knowledge of the determination of the chloride content from different methodologies for collecting hardened concrete dust, constituting a starting point for the establishment of future experimental campaigns to quantify the mass of the concrete dust sample for the considered depth.

The results showed that, for depths where the segregation has no effect, the average of at least 5 determinations led to a good estimate of the chloride contents of the concrete.

This work can be continued by an extensive laboratory campaign to establish a correlation between the maximum aggregate size and the mass of the concrete dust sample to consider for each depth.

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