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# Study of the Deterioration of Concrete Incorporating Sulfide-Bearing Aggregates

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# ABSTRACT

In the Trois-Rivières area (Québec, Canada), several hundred foundations of private houses, apartment and public buildings were built using concrete incorporating an anorthositic gabbro coarse aggregate containing sulfide minerals. A large number of those concrete structures developed serious deterioration within 3 to 5 years after their construction, which triggered, in many cases, their demolition/replacement. To help understanding the deterioration mechanisms and the conditions most prone to the development of premature damage, as well as quantifying the rate of deterioration, concrete blocks, 1 m x 1 m x 0.225 mm in size, were extracted from housing foundations just prior to their demolition. The blocks were disposed on an outdoor exposure site where they were monitored for the development of cracking and expansion over a five-year period. More than three hundred cores were extracted at different depths in eight housing foundations. A variety of tests including in-situ relative humidity measurements, petrographic, physical, mechanical and chemical tests on those cores were conducted for a full condition assessment of the concrete. A good correlation was observed between the monitoring of crack and the development of expansion in concrete foundation walls of different initial damage degree. Deterioration was also found to be largely influenced by the exposure conditions and RH conditions within the concrete. The determination of the total sulfur content of the concrete is a way to estimate the amount of sulfide minerals in the aggregate but significant variability was observed on values measured on different core samples from the same foundation.

Keywords: Pyrrhotite, Sulfide-Bearing Aggregate, Oxidation, Expansion, Cracking, Relative Humidity.

# 1.0 INTRODUCTION

Fine and coarse aggregates typically represent 60 to 75% of the concrete material; it is therefore not surprising that their composition and physicomechanical properties have a major impact on the behavior of concrete. Concrete aggregates may also contain some amounts of iron sulfides, mainly pyrite and pyrrhotite that are common minor constituents in many rock types. These sulfide minerals are unstable in the presence of oxygen and moisture and can oxidize and induce sulfate attack phenomena already recognized as problematic in many environments.

In recent years, the owners of nearly 1,400 singlefamily homes and other commercial buildings in the Trois-Rivières area of Quebec, Canada, have faced significant durability issues with their foundations and concrete slabs and floors (Rodrigues *et al.* 2012). Evidence of pyrrhotite oxidation has been observed and associated to the concrete deterioration. In many cases, cracking and deformation problems appeared on structures less than five years after their construction. More recently, hundreds of homes in the state of Connecticut (USA) have developed significant damage between 10 and 20 years after construction (Willie and Zhong, 2016). The problems have been related to the oxidation of pyrrhotite present within the aggregate from a quarry in the Willington county, Connecticut.

The signs of concrete deterioration observed in the distressed concretes mainly consist of map cracking on the surface of the walls with some crack openings reaching more than 40 mm. Yellowish and brownish discoloration was often observed surrounding these cracks. Pop-outs are observed on the walls showing the presence of oxidized and rusted aggregate particles. Small bumps easily detachable from below showing oxidized and rusted aggregate particles can be seen.

Pyrrhotite is well known as an unstable mineral in the presence of oxygen and humidity, where sulfide minerals oxidize to form acidic, iron and sulfate-rich by-products according to the following equations:

 $Fe_{1-x}+(2-x/2) O_2+H_2O \rightarrow (1-x) Fe^{2+}+SO_4^{2-}+2xH^+.$ 

The sulfuric acid generated through this process reacts with the solid phases of the cement paste to form gypsum (CaSO<sub>4</sub>·H<sub>2</sub>O) and secondary ettringite (Ca<sub>6</sub>Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>(OH)<sub>12</sub>·26H<sub>2</sub>O). The oxidation of ferrous iron produces ferric ions which precipitate to form iron oxyhydroxide. Both reactions generate secondary minerals that can cause expansion.

In this case, a deleterious process involving the oxidation of sulfide minerals and the sulfate attack of the cement paste is thought to have caused the swelling and cracking of the affected concrete elements. In fact, the presence of secondary reaction products including iron oxyhydroxide, gypsum, ettringite, and thaumasite was observed and support this hypothesis (Rodrigues *et al.*, 2012).

In this study, metric concrete blocks (1 m x 1 m x 0.225 mm in size) were extracted from residential foundations and disposed on an outdoor exposure site to help understanding the deterioration mechanisms and the conditions most prone to the development of premature damage. The blocks were monitored for the development of cracking and expansion over a five-year period (Francoeur, 2016).

Core samples taken from these blocks, as well as from other residential foundations prior to their demolition, were subjected to a variety of tests. Also, in-situ temperature and relative humidity were measured at different depths in the wall of foundations of eight houses the day before their demolition so as to correlate the concrete damage to the ambient conditions. As part of the laboratory work, sulfur analysis was performed to attempt to correlate the sulfur content in the sample and the concrete damage. Finally, a global performance-based protocol for the evaluation of the quality control of the aggregate materials is presented.

# 2.0 METHODOLOGY

Figure 1 illustrates the configuration of the concrete blocks on the outdoor exposure site. Metric blocks, with different levels of deterioration, were extracted from residential foundations and installed on this site. Deterioration levels were based on visual inspection of structures and range from level 0 (no visible cracking) to level 3 (high damage with crack opening of 2 to 12 mm). Ratings 1 (low) and 2 (medium) indicate intermediate levels of damage. In total, five series of five blocks (5 blocks per house of deterioration level 0, 1, 2, 3 (2x)) were instrumented on the site to perform annual monitoring of expansion, cracking index and cracking density.

# 2.1 Quantification of the Cracking – Cracking Index Method (CI)

Quantification of concrete cracking is performed using the Cracking Index Method (LCPC 1997). This method consists in measuring, using a crackmeter, a transparent plastic card with lines of different thickness, the opening of all the cracks intercepting one of the six axes of the lines traced on the surface of the metric blocks (Fig. 2). Metal studs were inserted into the concrete blocks to form the 500-mm grid. These studs were also used for expansion measurements.



**Fig. 1.** Configuration of the outdoor exposure site located in Trois-Rivières North



Fig. 2. Metal studs and grid established on the surface of the blocks

All cracks with a width greater than 0.05 mm are measured using the crackmeter perpendicular to their direction following the grid lines. All measurements taken are then reported in a table giving a value of the Cracking Index (CI) in mm/m used to quantify the importance of cracking according to the method proposed by LCPC (1997).

# 2.2 Expansion Measurement

Using the four studs placed at the corners of the grid traced on the metric blocks, the dimensional changes

**Table 1.** Characterization Scale of the Cracking Index

 Method

CI value (mm/m)	Importance of crack		
0 to 0.5	Negligible		
0.5 to 1	Low		
1 to 2	Moderate		
2 to 5	High		
5 to 10	Very High		
> 10	Significant		

are measured through a 500- mm gage-length comparator. These measurements are taken three to four times a year to evaluate the movement of concrete during temperature changes, as well as the overall evolution of expansion.

## 2.3 Cracking Density

A mapping of the cracking was carried out by tracing each visible crack on a plastic film fixed to the surface of the concrete block (Fig. 3). The layers thus produced were digitized in order to determine the surface density of cracking (total length of crack per evaluated surface). Mapping is performed at the same time as the CI measurements, i.e. by drawing the new cracks with a different color at each measurement to monitor the evolution of the cracking.



Fig. 3. Example of crack mapping

# 2.4 Instrumentation of Pre-Demolition Foundations

Eight residential foundations showing varying levels of deterioration from low-damaged (1) to severely damaged (3) were selected (Table 2). For each foundation, cores were extracted at three levels, i.e. above ground, at 30 cm below ground level and at the base of the housing foundation. In-situ temperature and relative humidity measurements were taken at these three levels and at different depths (50-75 mm, 100-125 mm, and 150-175 mm) inside the concrete (from the inside) using Vaisala equipment (HUMICAP® SHM40). To do so, dry drilling (16mm in diameter) is first carried out in the foundation wall using a percussion drill at various depths (75, 125 and 175 mm) in the concrete. The hole is then cleaned with a suitable brush and compressed air. A plastic tube is then inserted into the hole while leaving a concrete section of 25 mm long at the bottom of the

hole. The tube is specially machined to insulate this 25 mm section. The probe (Model HMP40S) is then inserted into the hole and a rubber stopper is placed in the tube to once again isolate the 25 mm section at the bottom of the hole. The probe is left in place for a period of one hour so that equilibrium is reached before readings (Fig. 4).

Table 2. Damage level of selected foundations

# housing foundation	Damage level	
#1 and #2	1 (low)	
#3 and #4	2 (medium)	
#5 to #8	3 (significant)	



Fig. 4. Instrumentation for in-situ temperature and relative humidity measurements

# 2.5 Chemical Approach – Total Sulfur Content

The determination of the total sulfur content is used for a rapid detection of the presence of sulfide minerals. The sulfur content can be measured directly on the aggregates particles prior to use in concrete. This method can also be used to diagnose a concrete problem; the measurement is then carried out on a concrete sample. Aggregate total sulfur content is an approximation of the sulfide content calculated from mineral stoichiometry. This technique cannot identify the type of sulfide present in the aggregate and only serves as a screening test.

The total sulfur content of material is determined by an infrared absorption method using an Eltra CS 800 carbon/sulfur analyzer. The sub-sample required for analysis is less than 1 g.

#### Total sulfur content of aggregate material

The total sulfur content of aggregate particle is measured on a powder sub-sample of less than 1 g. For this reason, a sample preparation method is proposed to avoid sampling bias.

#### Total sulfur content of concrete sample

For the diagnosis of concrete, it may be useful to know the sulfide contents of the aggregate particles used in its manufacture. To do this, a core taken from the foundations is crushed and pulverized to obtain a representative powder sample of the concrete. The sulfur content is measured directly on the concrete sample. The sulfur content measured is the total sulfur content of the concrete. Various calculations are performed to estimate the total sulfur content from coarse aggregates based on the following assumptions:

- dry density of concrete: 2150 kg/m<sup>3</sup>,
- cement dosage: 250 kg/m<sup>3</sup>,
- sulfur content of the cement: 3.9 (% w) SO<sub>3</sub>,
- proportion of coarse aggregate: 1100 kg/m<sup>3</sup>
- no sulfur contribution from fine aggregate (value of less than <0.005% (% w) S measured).</li>

In this study, the foundations of three houses were sampled to evaluate the variability of total sulfur analyzes. Indeed, in practice, when assessing the damage of a house, usually only one core is extracted to analyze the total sulfur content. However, it may be interesting to note the variability that can occur between cores taken at different levels or on different walls of the foundation. Thus, 3 samples per wall and three on the concrete floor were taken to carry out this representativeness study. The total sulfur content was determined on a complete core.

# 3.0 RESULTS AND DISCUSSION

## 3.1 Crack Index and Expansion Measurements

Figure 5 compares the expansion measured on concrete blocks of different levels of deterioration. For all blocks, the expansion increases over time. It should be noted, however, that the blocks of the A series (damage level 0) were extracted from a common wall of a semi-detached house, e.g. a foundation wall which has not been previously exposed to external conditions. This explains why the expansion values on blocks of series A are very low.

Figure 6 shows the cracking index development over time for the blocks of series A and B. The blocks of the series A do not show any visual damage during the first two years of outdoor exposure (2011-2013) Subsequently, cracking begins to develop and progression tends to accelerate over time. The blocks of the B series show a significant increase in cracking over time.

The B-series blocks with a damage level of 3 all showed a steady increase in the extent of cracking during the period studied, which resulted in very high CI values from 3 to 8.2 mm / m). It is interesting to note that the increase in CI values, although real in the vast majority of cases, is generally more "irregular" than in the case of expansion measurements. This is largely due to the fact that several operators were involved in the tests. However, a steadily increasing trend of cracking was observed in all the samples examined.



**Fig. 5.** Expansion of concrete blocks (A: damage level 0; B: damage level 3)



Fig. 6. Crack index of concrete blocks

Expansion rates were calculated from the expansion data. The blocks of the A-series show an average annual expansion rate of 0.028% with values from 0.009 to 0.074%. The B-series blocks have an

average annual expansion rate of 0.074% with values measured from 0.036 to 0.076%.

The average annual rate of increase of the cracking index was also calculated. The blocks of the A-series show an average annual CI rate of 0.181 mm/m with values from 0.072 to 0.294% for A4 and A5 blocks, respectively. The B-series blocks have an average annual CI rate of 1.087 mm/m with values measured from 0.914 (Block B5) to 1.334 mm/m (Block B1).

## 3.2 Cracking Density

Figure 7 shows an example of the determination of the surface density of cracks. Different shades of grey is used to represent each measurement in order to follow the evolution of cracking for the period beginning in 2011 to fall 2014. From these images, it was possible to calculate a crack density, i.e. the total crack length per evaluated area,  $m/m^2$ .

Figure 8 presents the calculated crack density for blocks of series A and B. It was clear from the cracks mapping that the block from the A-series had a lower crack density than those of the B-series. The calculation of the crack density makes it possible to quantitatively assess the evolution of the cracking. The rate of cracking over time can also be estimated from these data. After 150 weeks of testing, the blocks of the B-series have three times higher crack density values than those of the A-series, with values of 30 and 10 m/m<sup>2</sup>, respectively.



Fig. 7. Evolution of cracking over time



Fig. 8. Cracking density calculated based on crack mapping

#### 3.3 Instrumentation of Pre-Demolition Foundations

Table 3 presents examples of relative humidity and temperature measurements monitored in three residential foundations prior to their demolition (houses #2, #3 and #7).

**Table 3.** Temperature and Relative HumidityMeasurements at Different Depths in Three DifferentResidential Foundations

Level	Dopth	Hous	House #2		House #3		House #7	
	(mm)	T° C	RH (%)	T° C	RH (%)	T° C	RH (%)	
Up	50-75	19.6	83	21.8	87	22.9	89	
	100- 125	19.8	80	22.1	88	23.0	90	
	150- 175	20.0	84	21.7	88	23.3	-	
Centre	50-75	17.9	95	20.7	92	22.2	96	
	100- 125	17.8	94	20.2	95	22.3	98	
	150- 175	17.8	97	20.2	95	22.6	98	
Low	50-75	17.9	94	19.9	92	21.2	96	
10 12	100- 125	17.8	93	19.9	90	21.0	95	
	150- 175	17.9	93	19.9	92	21.0	96	

For houses #2 and #3, a slightly lower temperature was measured at the upper level of the foundations. The day of the measurements, the air temperature was between 18 and 19° C and the weather was cloudy. For house #7, the air temperature was 23° C. There was no significant variation in temperature values measured at different depths into the concrete. Relatively lower relative humidity values were

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obtained at the upper level of the foundation, whereas the values measured in the central and lower levels are on average rather similar. The upper part of the foundation is directly exposed to the weather conditions, allowing it to dry in dry weather or, on the contrary, to be wetter in rainy weather. Moreover, there was no significant variation in relative humidity values measured at different depths into the concrete, the values were very stable.

#### 3.4 Chemical Approach – Total Sulfur Content (Stot)

Until now, the chemical approach, involving a measurement of the total sulfur content, is a tool used for the rapid detection of the presence of iron sulfide minerals in concrete aggregates. The S<sub>tot</sub> measurement is used to calculate the sulfide content based on stoichiometry of the sulfide minerals present in the aggregate particles.

Because of the very small amount of material used for testing, a rigorous sample preparation method is required to ensure the representativeness of the samples. Following critical review, a sample preparation method was proposed as illustrated in Fig. 9 for the preparation of aggregate samples.



Fig. 9. Procedures for samples preparation for  $S_{\mbox{total}}$  measurements

According to the procedure described in Fig. 9, a representative 4 kg of the aggregate material with particle size ranging between 5 and 20 mm is first obtained. The sub-sample is split into 2 parts of 2 kg each. A 2 kg sub-sample is crushed to produce particles smaller than 5 mm. Then, a 500 g sample is obtained by splitting. The 500 g is pulverized to produce particles smaller than 300  $\mu$ m. The sample is split to obtain a sub-sample of 50 g that is pulverized to obtain particles smaller than 80  $\mu$ m. Finally, this last sub-sample is split to obtain samples for chemical analysis. Successive sieving, crushing and splitting operations were needed to produce representative subsamples of the original material.

As previously mentioned, total aggregate sulfur content can be estimated from total sulfur content measured on concrete samples as a diagnostic method. The calculation is based on different assumptions about the concrete formulation. The sulfur content is then attributed to the amount of coarse aggregates of the concrete.

The total sulfur content of coarse aggregate calculated for the eight instrumented houses is presented in Table 4.

**Table 4.** Total sulfur content of the coarse aggregatefor the 8 instrumented houses (3 measurements perhouse)

House	Damage level	Mean (x% S)	S. D. (σ % S)	CV (RSD) %
1	1	0.93	0.069	7.4
2	1	1.05	0.157	15.0
3	2	1.12	0.110	9.8
4	2	1.02	0.081	7.9
5	3	1.29	0.176	13.6
6	3	1.10	0.108	9.8
7	3	0.87	0.027	3.1
8	3	1.47	0.281	19.1

S.D. = standard deviation

CV = coeff. of variation (or relative standard deviation).

The values presented in Table 4 show that there is a considerable variability between values with coefficients of variation that vary between 3 and 20% within the same foundation. It is therefore important to determine more precisely this variability of the total sulfur content because the sulfur content measured is an important parameter in the characterization of aggregates.

To do this, 15 samples distributed at different locations in the foundations of two houses were analysed and compared. Three samples were drilled into each wall of the foundation and the last three were samples from the floor. For each wall, one sample was taken from the upper part of the wall, another from the central part and one from the base. The goal is to evaluate the possible variations of the sulfur content of the different concrete samples. Results are presented in Fig. 10. This figure shows the distribution of the total sulfur content of the coarse aggregate measured in two foundations with the mean value represented by the square and the bar representing the standard deviation.

House A shows an average of 1.1% with a standard deviation of 0.10 (coefficient of variation of 9.1%), while house B presents more dispersion with a mean of 1.1 and a standard deviation of 0.18 (coefficient of variation of 16.4%).

The data presented in Fig. 10 clearly show the heterogeneity of the distribution of sulfur content in aggregates. House A is showing a maximum variation of 0.4% while house B shows a difference of 0.7% while the mean values measured are only of the order of 1.1%. These values reflect the heterogeneity existing at the level of the aggregate particles. This heterogeneity of the sulfide minerals content is also observable at the quarry level.



**Fig. 10.** Variability of the total sulfur content in the aggregate measured from 15 concrete cores extracted from the same foundation

#### 3.5 Global Protocol Proposed for the Evaluation of the Quality Control of the Aggregate Materials

Several cases of concrete deterioration involving sulfide-bearing aggregates were reported over the years. However, no specific guidelines are available to enable making a precise decision on the potential deleterious character of some sulfide-bearing aggregates. A three-step evaluation protocol was proposed by Rodrigues *et al.* (2016) for the evaluation of sulfide-bearing aggregates prior to their use in concrete, as illustrated in Fig. 11.

In the first step, the aggregate total sulfur content (S<sub>Total</sub>, % by mass) is measured. The values obtained and existing standards lead to proposing: (1) a STotal > 1% as the value for rejecting the aggregate, (2)  $0.10\% \ge S_{Total} < 1\%$  as the interval value for proceeding to the second step, and (3) the  $S_{Total}$  < 0.10% as the limit value of acceptance of the aggregate material related to sulfide oxidation problem. It is important that the total sulfur content be evaluated on a representative sample of the aggregate material under study. As mentioned previously, it is not uncommon for the determination of the total sulfur content of already deteriorated foundation to be measured on a single piece of only one core. Figure 10 shows that the total sulfur content can show significant heterogeneity and that sampling should take this into account.



**Fig. 11.** Protocol proposed by Rodrigues *et al.* (2016) for the quality control of sulfide-bearing aggregates

The aggregates that are directed to step 2 are then exposed to an oxygen consumption test proposed by Rodrigues *et al.* (2016b). The oxygen consumption test is based on the fact that during the oxidation of the sulfide minerals, oxygen is consumed during the reaction. The test consists in measuring the oxygen content in the headspace of an airtight container in contact with the aggregate stored under conditions favorable to oxidation. Taking into account the limited number of aggregates tested by the authors, the tentative acceptance limit was lower than 5 % of consumed oxygen, while aggregates inducing values equal or higher than 5 % should be tested in the step 3 of the proposed protocol.

Step 3 consists of a two-part mortar bar test. A first part is intended to reproduce the deterioration resulting from the oxidation reaction of the iron sulfides in the aggregates. In Part I, mortar bars are subjected to a 90-day storage period at 80°C and 80% RH with two 3-hour soaking periods per week in a 6% bleach solution which is a solution with a strong oxidizing potential. The aggregate can be immediately rejected if the expansion value is higher or equal to 0.10% at 90 days (Rodrigues et al. 2015). If the values are lower than 0.10%, the samples should be transferred to 4°C and 100% RH for another 90 day-period. This second part of the

expansion test is intended to reproduce the internal sulfate attack due to ettringite and thaumasite formation (Rodrigues *et al.* (2012)). If, at the end of those 90 days, the samples expansion remains stable, the aggregates could be accepted. However, if the expansion continues, the aggregate should be rejected. The proposed protocol and preliminary test limits will need to be further validated through the testing of a larger number of aggregates.

Many types of rocks, and consequently the aggregate materials from which they are obtained, contain small amounts of iron sulfides. It therefore seems unrealistic to require concrete aggregates to contain none of these mineral phases. Analysis of numerous studies shows that pyrrhotite, even in very small quantities, is potentially harmful because it can lead to premature deterioration of structures by oxidation and sulfate attack processes. Unfortunately, there is not enough data to clearly determine how much pyrrhotite is likely to be harmful or harmless. In fact, the establishment of a "universal" pyrrhotite limit appears more or less realistic since several factors influence the reactivity of this mineral.

There is currently no test method to clearly assess the potential deleterious character of sulfide-bearing aggregate materials. A research program was conducted during the 2010-2014 period to highlight specific process that led to the premature deterioration of many concrete structures in the Trois-Rivières area (Quebec, Canada). Among the achievements of this program, a performance test protocol capable of detecting problematic aggregates has been developed. The various tests forming this protocol include the chemical analysis for total sulfur, an oxygen consumption test and a mortar expansion test. Although this is an unprecedented development on the subject, these tests have not been evaluated extensively for their robustness. In addition, the limit values proposed in the protocol are only preliminary since they are based on a limited number of aggregates. A complementary program is currently being developed with the objective of establishing final specifications for the use of aggregates incorporating iron sulfides in concrete.

# 4.0 CONCLUSIONS

In this study, various methods were used to evaluate the level and progression of the damage in concrete blocks made with aggregates incorporating iron sulfides exposed in outdoor conditions in the Trois-Rivières area (Québec, Canada). Volumetric changes were regularly monitored along with measurements of crack openings to obtain a value of the cracking index and a mapping of cracks on the surface of the blocks to calculate a crack density value. The progression of damage evaluated through expansion and cracking measurements is very apparent inside the same series of blocks extracted from the same foundation, as well as from one house to another. The rate of expansion is well correlated with the development of cracking for all monitored blocks.

The determination of the total sulfur content of the concrete is a way to estimate the amount of sulfide minerals in the aggregate. However, values of sulfur content of coarse aggregates calculated from the chemical analysis of several concrete cores extracted from the same foundation showed significant variability. It is therefore very important that measurements are made on a sufficient number of specimens in order to consider the significant heterogeneity of the presence of sulfides in the aggregates.

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