

**PYRRHOTITE IN CONCRETE AGGREGATE. INTRODUCTION TO MECHANISMS, DAMAGE POTENTIAL
AND ONGOING RESEARCH**

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Abstract

Pyrrhotite is an iron sulfide which when present in concrete aggregate may lead to expansive reactions, cracks and finally disintegration of the concrete. This potential risk when using aggregates containing sulfides in concrete production has been known for several decades, and international concrete standards take this into account. Though, recent examples of deterioration of concrete structures in Canada and USA, and the rejection of tunnel masses for application as concrete aggregates for the construction of the Follo line tunnel in Norway, have raised questions regarding the test methods and regulations for sulfides and pyrrhotite in concrete aggregates. A Norwegian R&D project led by NTNU is currently looking into the characterization and quantification of sulfide minerals in aggregates (WP1) and working with the development of test methods for documentation of the damage potential in concrete (WP2). There is a close collaboration between the Norwegian research team and researchers from the university of Laval in Canada which have been working with pyrrhotite in concrete for over a decade. This paper introduces the basic chemistry of the reactions of pyrrhotite in concrete, presents examples of deterioration caused by pyrrhotite and lists the relevant regulations currently in place in Norway and North America. Eventually, the challenges and research questions raised will be discussed and how we are planning to tackle these will be presented.

Keywords: Pyrrhotite, Concrete durability, Iron sulfides, Testing methods

INTRODUCTION

Iron sulfide minerals occur in many igneous and metamorphic rocks, albeit mostly in minor quantities. These minerals may be reactive when water and oxygen are present. Iron sulfides in concrete aggregates should therefore be handled with caution. Pyrrhotite is such an iron sulfide mineral and will under the right circumstances react into voluminous or non-cementing reaction products, causing expansion and disintegration of the concrete, when present in the aggregates.

During the last decade (2010-2020) several thousands of damage cases have been reported in North America, mainly from residential housing. Concrete foundations showed extensive signs of map cracking, pop-outs, discoloration near cracks and white stains around aggregate particles only 3 to 5 years after construction [1]. The deterioration was deemed so severe that many of the affected foundations had to be replaced. Rodrigues and the Canadian research team mainly from university of Laval, Canada, have done extensive work on developing a three-step testing protocol on concrete aggregates containing iron sulfide minerals to handle the problem [2].

In Norway, we were confronted with pyrrhotite in concrete aggregates in connection to the construction of the Follo line (2010-2022), a 20 km long tunnel South-East of Oslo, Norway. The contractor and commissioner had planned to use 10-15% of the tunnel masses to produce the concrete elements for the tunnel lining. Though, during the project, routine testing of the tunnel masses revealed an elevated sulfur content as well as indications of the presence of pyrrhotite. Out of precaution, it was decided to deposit the tunnel masses and instead purchase aggregates from a local quarry for the concrete element production. This incident led to considerable additional costs and deposition of around 1,5 million tons of tunnel masses. Questions were raised regarding the accuracy and relevance of the test methods and regulations for concrete aggregates containing pyrrhotite.

So far, there are no known cases of concrete deterioration in Norway caused by pyrrhotite-bearing aggregates. In the future, however, the use of crushed aggregates for concrete production is expected to increase due to depleting reserves of natural aggregates. The difference in the surfaces of the aggregates, i.e. fresh surfaces of crushed aggregates compared to the weathered surfaces in natural aggregates, can lead to an increased damage potential of pyrrhotite in crushed concrete aggregates [3].

In 2019, as part of a Norwegian preliminary research and development (R&D) project, an international workshop on the topic of pyrrhotite in concrete aggregates was arranged by the Norwegian Public Roads Administration (NPRA) in Oslo [4]. In 2020, a main R&D project was initiated focusing on test methods and regulations for concrete aggregates containing pyrrhotite, as well as the underlying degradation mechanisms. The project is led by NTNU, and project partners are SINTEF, NPRA, Bane NOR and HeidelbergCement Northern Europe. The project is cooperating closely with the Canadian research team. This paper sums up the challenges with pyrrhotite in concrete aggregate, the current regulations, potential test methods, research questions and planned future work in the Norwegian R&D project (2020-2024).

CHEMISTRY OF PYRRHOTITE REACTIONS

The general formula for pyrrhotite is $Fe_{1-x}S$ where x ranges from 0 to 0.125 [5]. The value of x affects the crystalline structure and magnetic properties of the mineral [5]. Like other iron sulfides, pyrrhotite is unstable in a system with water and oxygen. Thus, it will initially react into ferrous ions Fe^{2+} , sulphates SO_4^{2-} and protons H^+ . There are two possible subsequent reactions. Firstly, the ferrous ions can oxidize further to give ferric ions Fe^{3+} which in turn could yield ferric hydroxide, commonly known as rust. It should here be noted that the ferrous ions could also oxidize into ferric oxyhydroxide, also known as rust. The second important, and more severe, consequence of the original reaction of pyrrhotite, water and oxygen is the formation of sulfuric acid (H_2SO_4). Reacting with portlandite in the cement paste it will produce gypsum, which in turn yields the formation of ettringite and in the presence of carbonates at lower exposure temperatures, potentially thaumasite. The gypsum, ettringite and thaumasite are crystalline reaction products, which upon formation can result in cracking and disintegration of the concrete.

Gypsum is suspected to be the culprit of both softening of the concrete's surface and its expansion [6]. Thaumasite and ettringite have a similar crystal structure (hexagonal rods), making it hard to distinguish them using for example X-ray diffraction, though they have a very different effect on the concrete. Ettringite can when formed in hardened cement paste, such as for delayed ettringite formation (DEF) or external sulfate attack, lead to expansion of the concrete, resulting in cracking and finally disintegration [7]. Thaumasite formation requires besides the presence of sulfate ions, also carbonate ions and a temperature below 15 degrees Celsius [8, 9]. Thaumasite has low cementing properties and will lead to disintegration of the concrete by degradation of the cement paste. Rodrigues and the Canadian research team's assessment of deteriorated structures in the Trois-Rivières area found that thaumasite plays a key role in the disintegration of the concrete [1].

CASE EXAMPLES OF DETERIORATION INVOLVING PYRRHOTITE

Although it is unclear whether pyrrhotite in concrete aggregates has caused problems in the Nordic countries, there have been several discoveries in other parts of the world, where the damage is severe. The most recent cases are located in North America [1, 10, 11], but it also seems appropriate to look at a big case in the UK [12] and the work done by Schmidt et al. on a Swiss dam [13]. Both a brief description of these cases and relevant work related to them are presented in this section.

Residential housing in Trois-Rivières, Canada [1]

In the Trois-Rivières region of Canada, between the cities of Quebec and Montreal, thousands of residential buildings, and some commercial buildings, had developed obvious structural problems [1]. An important fact to be noted is that in many cases these signs of deterioration came only 3-5 years after construction. The visual inspections documented that map cracking, pop outs and yellow rims on some surface particles were present throughout the different basements (see Figure 1). The largest cracks were found at the corners of the foundations, frequently located close to rain gutters, indicating the importance of humidity in the reaction processes. These larger cracks could have widths of 40 mm, but with 10 mm as a more frequent maximum value. The occurrence of yellowish stains seemed to be linked to the most cracked parts of the concrete. The deterioration was deemed so severe that many of the foundations and bearing walls had to be replaced. Such an intervention is both a major inconvenience to the residents and a financial cost that in many cases was higher than the original construction costs.

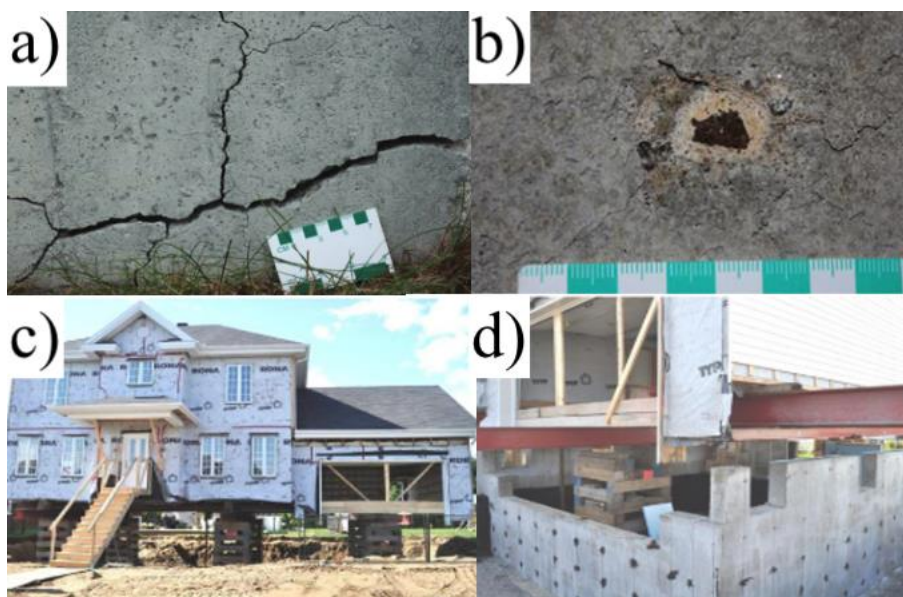


Figure 1 – a) Cracking of foundation. b) Yellowish rim on aggregate pop out. c) and d) The process of replacing a concrete foundation deteriorated by pyrrhotite. [1]

After the visual inspections, the researchers at the university of Laval began investigating numerous concrete cores from the distressed foundations. Examinations of such samples can only really say what minerals and reaction products are present, and not quite how they came about. However, it was evident that several of the particles containing sulfides (mainly pyrite and pyrrhotite) were covered with rust (iron oxyhydroxide). All concrete cores proved that the aggregates contained iron sulfides, not surprisingly, bearing in mind that the rock material was igneous and with varying degree of metamorphism.

Assessments of the aggregate and the reaction products were conducted using a variety of techniques. Examinations with the stereomicroscope showed that the aggregate particles had large cracks, and that these cracks propagated into the cement paste. Similar cracks also occurred in oxidized aggregate particles, which were completely covered in iron oxyhydroxide surrounded by a white rim. Overall, the examination showed that most of the pyrrhotite had been oxidized, while pyrite was predominantly intact.

Residential housing in Eastern Connecticut, US [10]

A report from 2016 by Kay Wille and Rui Zhong, to the Attorney General of the State of Connecticut, gives insight into another area troubled by concrete deterioration caused by iron sulfides [10]. The report was requested in 2015, after many foundations of houses in Eastern Connecticut showed severe deterioration. Wille and Zhong conducted both visual inspections and rigorous laboratory investigations on samples from the structures in question. The visual inspection of 21 different houses showed map cracking, reddish stains and white powder on the concrete surface, crack widths larger than 4 mm and quite significant deformation of concrete foundation walls. These symptoms are comparable to the cases from the Trois-Rivières region. Among their discoveries was the occurrence of ettringite resulting in severe damage to the concrete.

The mundic problem, South-West England [12]

Sims, Santo and Brindle have done extensive work on the mundic problem ravaging parts of South-West England. They composed a guidance note, most recently edited in 2015 [12]. ‘Mundic’ is originally a word from the Cornwall region of England used to describe pyrite. Today, however, mundic should be understood as concrete problems where the petrography of the material is characterized by sulfides and sulfide decomposition products. The guidance note (and its preceding editions in 1994 and 1997) tries to both explain the potential problems related to sulfide-bearing concrete aggregates and suggests a sampling and testing procedure for concrete characterization, primarily for domestic and small commercial buildings.

As mentioned, mundic is a Cornish word, making it no surprise that the problem is most prevalent in the South-West region of Cornwall and Devon. Mining industry is something this part of England is known for, which for a long time provided an easily accessible (albeit low-quality) source of aggregate for concrete production in terms of debris and refuse. In the 1950s, the British Standards for aggregates were introduced, bringing the use of mine waste rocks and -tailings to a halt. In other words, most of the buildings in question when it comes to the mundic problem are constructed in the first half of the 20th century. The wider implications of the problem became apparent in the mid-1980s. A common denominator for the examples of sulfide-caused concrete deterioration in Cornwall and Devon is that it was hard to visually identify the mundic problem before it was at an advanced stage. Figure 2 shows a typical case of mundic damage from South-West England.



Figure 2 – Typical Mundic damage in residential building in South-West England [12].

The procedure which was first introduced in 1994 aims at classifying the concrete material into 5 classes, through 3 different stages of examination. This classification of concrete requires both identification of aggregate types and assessment of the concrete's condition. The classes are used for describing how sound and suited the concrete structure is for its current use. For more details see [12].

Concrete dam, Switzerland [13]

Schmidt et al. investigated concrete from a 40 year old dam in Switzerland [13]. Being built in the early 1970s, the dam has been closely monitored ever since, which led to the discovery of a steady expansion since the 1980s that is still progressing. In 1998, the upstream expansion at the top was 20 mm and 5 mm in the middle gallery. Corresponding expansion had increased to 45 mm and 10 mm, respectively, in 2010. The concrete displayed obvious deposits of rust and there was also a scent of sulfurous compounds coming from the dam. They also reproduced the concrete and made laboratory samples and exposed them to accelerated conditions simulating the exposure environment of the dam, i.e. storing prisms in water at 60°C for 5 years.

Both pyrrhotite and pyrite were present in the concrete samples from lab and field, but the former was far more frequent. The mineral was randomly dispersed and agglomerated within the aggregates. For the concrete in the dam, 80% of the volume of iron sulfides was pyrrhotite, but only 30% to 40% of this had reacted after 40 years. In the laboratory sample, there was a far lower percentage of pyrrhotite which had reacted, showing the challenge of recreating real conditions in accelerated testing.

The reaction products were predominantly iron hydroxide and goethite. More widespread in the field-exposed concrete sample (but also present for the lab specimen), extensive cracking of aggregate was found, most frequently originating close to iron sulfides in the aggregates and growing into the cement paste. This indicates a direct relation between degradation of concrete and reaction of iron sulfides, and the consequent cracking and expansion of concrete.

As is typical for a sulfate attack, there was widespread occurrence of ettringite in the concrete extracted from the dam. This had formed in the microstructure, mainly situated in the shells left by hydrated cement grains. The vicinity of these deposits showed no serious signs of distress, inferring that ettringite is not the cause of the expansion of the concrete, at least not the main one. The case might be that ettringite is growing where there is available space, without exerting any internal pressure on the surrounding material. There is a caveat to these deductions, namely that the expansion of the concrete is not very big, making it hard to pinpoint one single responsible factor and/or reaction product.

CURRENT REGULATIONS

European and Norwegian regulations

As previously stated, the potential dangers of using sulfide-bearing aggregates in concrete have been subject to discussion for many years. The European concrete aggregate standard NS-EN 12620 states [14]:

- *Total sulfur content (S_T) of aggregates determined in accordance with EN 1744-1:1998 clause 11, shall not exceed 1% by mass in general, and 2% by mass for air-cooled blast furnace slag. If pyrrhotite is present in the aggregate, a maximum content of 0.1% S_T shall apply [15].*

Two observations highlighted in the state-of-the-art report by Danielsen et al. [4] are the fact that no specific classification or detection method for pyrrhotite is mentioned, and that 0.1% S_T by mass is quite significantly more liberal than for example 0.10% by mass. The former means that up to 0.149% total sulfur by mass is accepted. Several Norwegian concrete aggregates have documented a total sulfur content in this narrow range from 0.10-0.15% [16].

EN 1744-1998, the standard for testing of chemical properties of aggregates, gives both a reference method (Clause 11.1) and an alternative method (Clause 11.2) for documenting the total sulfur content [15]. The reference method starts off with exposing the aggregate to hydrogen peroxide and hydrochloric acid, making sulfur compounds react into sulfates. These anions (SO_4^{2-}) are then precipitated as barium sulfate ($BaSO_4$). The barium sulfate is weighed, making it possible to give a ratio of total sulfur content as a percentage by mass of the aggregate. The alternative method takes advantage of a different quality of sulfur. The aggregate sample is placed in a pure oxygen atmosphere and is subsequently ignited, turning sulfur composites into sulfur oxides. Infrared detectors analyzing the gas can then quantify the content of sulfur in the aggregate as a percentage of the total mass. Although the principle of making all sulfur in a sample react is the same for the two methods, there is nothing that conclusively gives reason to believe that these two procedures will lead to identical results in every case. As part of WP1, an ongoing PhD is looking into, among other things, how different the results from the two methods are, which method is the most accurate and how we can improve the accuracy and precision.

There is a Norwegian national annex to the European concrete aggregate standard NS-EN 12620, which goes further than the main standard when it comes to sulfur contents and pyrrhotite [14]:

- *Maximum sulfur content (S_T) in aggregate equals 1.0% by mass*
- *For S_T values > 0.1% by mass, the aggregate must be tested for any pyrrhotite. The testing must be conducted using differential thermal analysis (DTA).*

The DTA method, used commercially by SINTEF, is based on comparison between a reference material (for example aluminum oxide Al_2O_3) and the material subject to examination [17]. By exposing these samples to identical heating and cooling programs under a specific atmosphere, a recording of temperature differences between the two of them can be used to analyze crystallization, melting, sublimation and other transformations through determining enthalpy changes. In the DTA analysis, the sulfides are irreversibly oxidized in an exothermal reaction. This method is among the test methods that is critically assessed as part of the ongoing PhD study.

After 2004, when it became mandatory for concrete aggregate producers to document the total sulfur content, an extensive amount of commercial testing has been conducted. As part of the preliminary R&D project in 2019, Haugen and Lindgård [16] compiled and analyzed all available test results at SINTEF from 2004 to 2018. Of a total of 235 samples tested, 19 samples (equivalent to 8%) with $S_T > 0.1\%$ contained pyrrhotite, thereby disregarding them for use in concrete according to NS-EN 12620.

Canadian and American regulations

In the US, there is yet no mention of sulfur contents in aggregates in the main concrete aggregate standard, ASTM C33-18. However, the ASTM C294-12 makes a remark concerning the expansion and reaction potential of marcasite, pyrite and pyrrhotite.

The question of pyrrhotite occurrence in aggregate and reactivity has been discussed in Canada far more extensively than in the Nordic countries. For the past ten years, there has been a lot of research leading to numerous papers from the university of Laval, resulting in among others the three-step testing protocol for aggregates shown in Figure 3. The first phase consists of a total sulfur content test on the aggregate, while the second phase looks at the oxygen consumption and oxidation potential of the aggregate. Eventually, there is a third phase investigating the expansion of mortar bars through an accelerated laboratory procedure with bleach as the reaction (oxidizing) agent. The international cooperation is showing its importance through this testing protocol, since the research and knowledge build up in Canada makes it easier for other countries like Norway to continue investigating the problem aiming to find suitable and reliable test procedures and corresponding national or regional regulations, of which the industry as a whole will benefit. There has been a similar cooperation between Canada and Norway for the development of the Norwegian AAR (Alkali Aggregate Reactions) regulations and test methods since the 1990's.

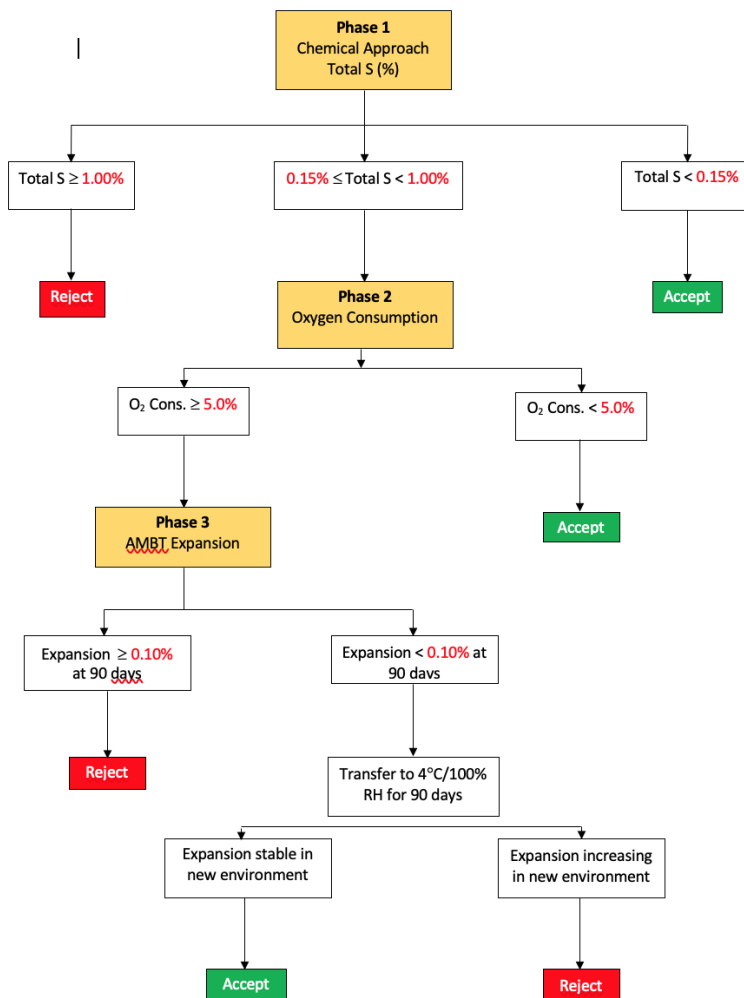


Figure 3 – The three-step testing protocol. Flow chart is based on [2] and [18].

The Canadian standards operate with both informative and normative annexes, where an informative appendix can be formulated as a normative clause after some time, for example in the next edition of the standard. Annex P in the revised 2019 version of the Canadian concrete standard CSA A23 [18] is such an informative addition and states (related to test results from the three-step testing protocol):

- *The aggregate is deemed fit if the total sulfur content (S_T) is $< 0.15\%$ by mass (Step 1). For $S_T > 1.00\%$ by mass the aggregate is deemed unfit. If no pyrrhotite is present, aggregate in the range $0.15-1.00\%$ is accepted.*
- *However, if pyrrhotite is present for aggregates in this range, an oxygen consumption test must be conducted (Step 2). For $< 5.0\%$ O_2 consumed during the test, the aggregate is accepted, while O_2 consumption $\geq 5.0\%$ leads to more testing according to an accelerated mortar bar expansion test (Step 3).*
- *For mortar bar expansion $\geq 0.10\%$ after 90 days of exposure and presence of pyrrhotite the aggregate is conclusively rejected, while an expansion $< 0.10\%$ deems the aggregate fit for the purpose provided the expansion is not exceeding 0.10% during the subsequent 90 days cold storage ($4^\circ C$). (A possible expansion during the first 90 days, but no expansion during the subsequent cold storage might be caused by AAR).*

The limits and regulations do not really differ from the European standard EN 12620, although the testing in Europe is not formally organized into a three-step testing protocol as in Canada.

CHALLENGES AND RESEARCH QUESTIONS – NORWEGIAN R&D PROJECT

With regards to pyrrhotite, researchers, contractors, aggregate and concrete producers and engineers face different challenges. The primary, and ultimate, goal is to be able to use local aggregates in a safe, effective, and environmentally friendly way. Utilizing local aggregates eliminates the financial and carbon emission related costs of transport. In the case of tunnel masses one can also avoid depositing part of the masses. Builders commonly set targets for contractors when it comes to CO_2 emissions, and these are more easily achieved if local aggregates can be applied.

Even though the use of local aggregates is seemingly positive, there is no guarantee that it is of appropriate quality. We therefore need to develop a testing protocol for pyrrhotite in aggregates, similar to what we have for AAR. This testing protocol needs to represent the conditions in field, will have to be robust to yield consistent results for different laboratories, as well as preferably being cheap and quick for the aggregate producers and contractors. Whether we will be able to develop potential mitigate measures for iron sulfides and corresponding performance test methods as we have developed for AAR is unsure.

The contractor for the Follo line tunnel wanted to use local aggregates from the excavation, but these masses were halted by the concrete aggregate regulations. Albeit the only possible decision at the time, whether this decision was technically correct is hard to know, and it is one of the questions the ongoing R&D project led by NTNU would like to shed some light on. The Mundic problem, mentioned earlier, shows how local aggregates being used heedlessly could lead to deterioration caused by iron sulfides like pyrrhotite. Even though the industry has developed since this specific case, there is still potential for more accessible and effective quality assuring systems. For example, the cases from Trois-Rivières and Connecticut provide evidence of what could still happen today when there is not enough knowledge about potential concrete deterioration mechanisms. On the other hand, the work done by Schmidt et al. [13] on the Swiss dam shows how challenging it is to produce accurate correlation between accelerated laboratory tests and field observations.

Although there is an increasing focus on the problems of pyrrhotite in concrete aggregates, there are still many unanswered research questions. Within the Norwegian research project, work package 1 (WP1) focuses on the characterization and quantification of low contents of sulfide minerals in concrete aggregates. This is done by Nicolas Oberhardt within a 4-year public sector PhD-project through cooperation between NTNU, NPRA and the Norwegian Research Council (NRC). Prevailing standard methods described in [15] are investigated and compared to advanced petrographic techniques to determine their detection limits, capability, and applicability. New concepts including the low-temperature thermodynamic magneto-chemical characteristics [3] of pyrrhotite are researched to comprise better analytical precision [4]. Some geologically related questions that also need to be investigated are; 1) how much pyrrhotite reacts? ; 2) are there any other minerals affecting its reactivity?

; 3) what are dangerous amounts of pyrrhotite? ; 4) can one be certain that the risk of deterioration of the concrete rises with increasing pyrrhotite content?

Based on the research in Canada and in close cooperation with WP1, WP2 is aiming on developing suitable and reliable test procedures for aggregates, mortar and/or concrete and establishing corresponding acceptance criteria. The latter provides that the test procedures are able to mirror the field conditions, and that a link between laboratory and field results is established. The Canadian three-step testing protocol will be used as a starting point, by validating it for Norwegian aggregates as well as investigating the reaction mechanisms by analyzing laboratory exposed samples as well as field samples. The MSc thesis of Hallvard Lindstad in spring 2022 focuses on assessing the last method in the Canadian testing protocol, more specifically analyzing the reaction mechanisms in relation to the accelerated mortar bar expansion test. The examinations will be conducted with micro-X-ray fluorescence (μ XRF) and scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS).

Understanding the reaction mechanisms will enable us to develop potential mitigating measures, for example whether any additives to the concrete, like supplementary cementing materials (SCMs), would be able to prohibit pyrrhotite from reacting (as is the case for alkali-silica reactions).

The overall aim of the Norwegian R&D project is to provide a reliable and efficient test protocol that enables safe use of local aggregates. Extensive research and comparison of laboratory results and field observations are paramount to ensure the validity of the test methods and reach correct acceptance criteria. Research to solve this main challenge needs to go on for many years. The hope is, however, to establish national (or European) regulations converging the industry towards sustainable use of local, concrete aggregates, including aggregates produced from excavated tunnel masses.

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