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# Development of a quick screening staining test for detecting the oxidation potential of iron sulfide-bearing aggregates for use in concrete



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#### A R T I C L E I N F O

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#### 1. Introduction

#### 1.1. General problem

In Trois-Rivières area (Province of Quebec, Canada), the deterioration of the concrete foundations and slabs of hundreds of recent built private houses and commercial buildings has been reported. The first signs of damage, such as map-cracking, pop-outs and yellowish to brownish surface staining (Fig. 1A to C), were, very often, visible 3 to 5 years after construction. In several cases, extreme measures, which include the total replacement of the affected concrete foundation, have been taken, but with important costs [1–3] (Fig. 1D).

The research that is being led by Rodrigues and co-workers [2–7] on the concerned affected structures shows that concrete deterioration is associated to the presence of a hypersthene gabbro coarse aggregate containing pyrrhotite, pyrite, pentlandite and chalcopyrite. The oxidation of iron sulfide minerals, mainly of pyrrhotite, followed by internal sulfate attack leading to thaumasite formation is regarded as responsible for the swelling and cracking of the concrete. The mechanisms of deleterious oxidation reactions and its influencing factors are thoroughly described in the work of the aforementioned authors.

#### ABSTRACT

Oxidation of iron sulfide-bearing aggregates is pointed out as responsible of concrete deterioration of numerous buildings in Trois-Rivières area, Canada. Several researchers have been focusing their efforts to fully understand the mechanisms involved in such reaction and to ultimately establish accurate guidelines for the quality control of aggregates. Though this testing programme is already rather well-established, a quick staining test will allow complementing the chemical approach that serves as its initial step. This paper presents a series of attempts to define the most accurate staining method by testing the efficiency of ASTM C 641 and the Midgley test, but also by optimizing them, namely by using different oxidizing reagents. The use of 6% bleach was able to correctly identify the oxidation potential of the investigated aggregates when used as reagent in the Midgley test. Based on the observations made in staining tests, a promising temperature test was also developed.

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#### 1.2. Assessment of the potential reactivity of iron sulfide bearing aggregates

According to the recent team work of several Canadian researchers on the development of a testing programme that can reliably evaluate the deleterious behaviour of iron sulfide-bearing aggregates, a succession of steps, starting with quick screening tests and then moving on to long-term performance tests, must be followed (Fig. 2). Though the two last phases of this testing programme are relatively wellestablished, a quick screening staining test is deemed necessary to complement the existing methodology, namely the chemical approach made by the measurement of the total sulfur content (% S<sub>T</sub>). Currently, some procedures are available for rapid detection of the oxidation of potentially deleterious iron sulfide-bearing materials. These methods are described hereafter.

#### 1.2.1. Midgley test

The procedure proposed by Midgley [9] describes a rapid qualitative test based on the observation of the oxidation of iron sulfide-bearing aggregate particles immersed in a saturated lime water solution. The formation, within 30 min, of blue-green ferrous sulfides, which turn brown in contact with air, is indicative of the presence of potential deleterious material or of the potential reactivity of the aggregates when used in concrete [10]. It must be noted that no specific size or quantity of the material required for testing is suggested by the author of the method.

MacDonald [11] reported to be unsuccessful when testing the oxidation potential of the gabbro used in the concrete of the damaged housing and building foundations in Trois-Rivières area when using the Midgley test. The same researcher replaced the standard oxidizing agent by 3% NaCl and obtained more satisfactory results.

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Fig. 1. Visual signs of concrete deterioration on the foundations of Trois Rivières private houses and the ultimate measure taken for replacing the affected structures: A) map-cracking filled with caulking material; B) pop-outs; C) discoloration; and D) total replacement of a concrete foundation.

#### 1.2.2. ASTM C 641

The ASTM specifications C 330 [12] and C 331 [13] for lightweight concrete aggregates refer to the standard test method ASTM C 641 [14] for iron staining materials. This test is based on the work carried out by Seaton [15] on cinder aggregates. In its procedure, 200 g of aggregate enclosed in filter paper are exposed to a steam bath during 16 h. The extent and density of staining formed is then appraised by comparison with a provided petrographic stain index (visual classification method). When the material develops heavy or very heavy staining, a chemical analysis method is required in order to determine the amount of Fe<sub>2</sub>O<sub>3</sub> that was deposited on the filter paper. An amount equal or greater to 1.5 mg of Fe<sub>2</sub>O<sub>3</sub> leads to the rejection of the material.

#### 2. Objectives and scope of the work

The existing staining tests have not been regarded as sufficiently accurate for the identification of potentially reactive sulfide-bearing aggregates for concrete applications. The development of a reliable quick chemical test urges from the need to complement the existing methodology, such as the determination of  $S_T$  content, used as a first step for detecting the presence of the potential deleterious material. The passing or failing on these preliminary tests will allow a more satisfactory initial evaluation of iron sulfide-bearing aggregates thus

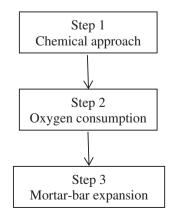


Fig. 2. Simplified scheme of the different steps for the evaluation of the deleterious behaviour of iron sulfide-bearing aggregates (adapted from [8]).

avoiding, in some cases, the need to recur to extensively long performance tests.

The current work presents a series of attempts to define the most adequate chemical test for the detection of potential deleterious iron sulfide-bearing aggregates using different oxidation agents. Furthermore, and from the observations made in the chemical tests, a temperature test and a staining method were developed. The scrutiny of the oxidation temperature of the investigated aggregates is made in order to check if such parameter would allow the quantification of the sulfide oxidation potential and, thus, to single out the possible deleterious materials since the staining method is rather of qualitative nature.

#### 3. Materials and methods

#### 3.1. Aggregates

Seven iron sulfide-bearing aggregates (MSK, B&B, GGP, SB, SW, SPH and SDBR) from diverse origins and sulfide contents were selected in order to assess their sulfide oxidation potential when used for concrete applications. The aggregates' main characteristics are displayed in Table 1. These aggregates were previously investigated by Rodrigues et al. [3,7] in an attempt to develop a new mortar bar expansion test for assessing the potential reactivity of sulfide-bearing aggregates in concrete and also to quantitatively assess their oxidation potential using an oxygen consumption test.

MSK is the iron-sulfide bearing hypersthene gabbro used as aggregate in the concrete foundations of Trois Rivières houses. A similar lithology sampled close to MSK quarry, but particularly enriched in iron sulfide content because samples were hand-picked due to their visually high iron-sulfide mineral content, was collected and investigated as B&B. Also from Quebec, the GGP granitic gneiss and SB hornfel were selected on account of their mineralogies and iron sulfide contents. Aggregate SB is also alkali-silica reactive. Cases of concrete deterioration due to the presence of pyrite and pyrrhotite have been reported regarding aggregates SW and SPH in Switzerland and Spain, respectively [16-21]. As so, the incorporation of such metamorphic rocks in the investigation of the oxidation potential of iron sulfide-bearing aggregates seemed relevant. In addition, both aggregates are also classified as alkali-silica reactive. From the Sudbury area (Canada), the SDBR mine waste material, which is extremely rich in iron sulfides, was also studied. It must be noted that the behaviour of the material SDBR was not investigated in all the tests presented in this paper. Two other

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