



Deterioration of residential concrete foundations: The role of pyrrhotite-bearing aggregate

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ABSTRACT

Premature deterioration of concrete foundations has been reported for a large number of homes in Eastern Connecticut of the United States. The characteristic symptoms are map cracking, whitish deposits at the vicinity of the cracking surface and wide crack opening. It is hypothesized that the volume expansion associated with the oxidation of pyrrhotite-bearing aggregate and the following internal sulfate attack (ISA) due to the delayed secondary mineral formation are the primary causes of premature deterioration. Extensive tests were carried out to validate the hypothesis using a combination of tools such as scanning electron microscopy (SEM), X-ray diffraction (XRD), X-ray fluorescence (XRF) and energy dispersive X-ray (EDX).

The results showed that pyrrhotite and its oxidation products, such as ferrihydrite, goethite and sulfur, were present in or at the surface of the aggregates of the deteriorated concrete, suggesting the occurrence of pyrrhotite oxidation. Expansive secondary mineral formations (SMF) such as ettringite and thaumasite were also identified. The abundance of these SMFs, their spatial distribution in the open spaces together with their close association with matrix cracking suggest the cause of premature deterioration.

1. Introduction

Recently in Eastern Connecticut of the United States, concerned property owners and authorities reported severe premature deterioration of concrete foundations that required immediate remedial action. Common deterioration symptoms included map crackings, whitish deposits at the vicinity of the cracking surface and wide crack openings (Fig. 1). An exemplary mixture design of the concrete is provided in Table 1.

More than 70 core samples were extracted from zones with different degrees of deterioration from seven selected houses in the affected area. In all cases pyrrhotite, an iron sulfide (Fe_{1-x}S ($x = 0$ to 0.125)), was detected in the severely damaged area by X-ray diffraction (XRD) technology. This suggested that a deleterious process had been triggered by the presence of pyrrhotite. While very little literature exists on the deterioration of concrete induced by pyrrhotite oxidation, to the best knowledge of the authors, the first case was reported in the Oslo region of Norway in 1959 [1]. In the Trois-Rivières area (Québec, Canada), premature deterioration of concrete foundations were reported only two to five years after their construction in the late 1990's and 2000's [2,3]. Lee et al. investigated highway concrete deterioration in Iowa (USA) due to the delayed formation of ettringite (DEF) in 2005

[4]. Oliveira et al. reported that the downstream face and galleries damage of the Graus Dam (Spain) was caused by the pyrrhotite bands incorporated in the aggregate in 2014 [5].

Oxidation of pyrrhotite in the presence of water and oxygen leads to a series of chemical reactions. The formation of these products is associated with an increase in volume in comparison to the initial reactants [6]. The expansive oxidation reactions which have been referred to as “primary expansion” are summarized in Table 2 [7]. It is worth noting that the volume changes listed in Table 2 represent the maximum expansion for a complete oxidation reaction and thus overestimate the actual volume expansion. In Ref. [8] it was reported that between 30% and 40% oxidation (by volume of the total iron sulfides) occurs for real concrete structures constructed using pyrrhotite-bearing aggregates. From the point of view of thermodynamic stability, ferrihydrite ($\text{Fe}(\text{OH})_3$) (Table 2, Reaction (I)) is the predominant sulfide oxidation product under alkaline conditions which are representative for concrete [9]. Evidence from field and laboratory investigations also indicated that the oxidation of pyrrhotite may not be complete and elemental sulfur (S^0) can be generated according to the acid-consuming reaction (Eqs. (1) and (2)) [10–12].

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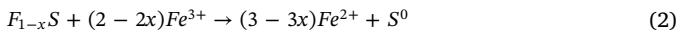
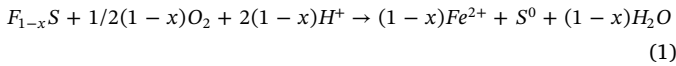
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Fig. 1. Common symptoms of deteriorated concrete foundation.

Table 1
Mixture design of concrete.

	Cement	Water	Water reducer	Sand	Coarse aggregate (3/4")	Coarse aggregate (1/2")
kg/m ³	285	168	0.356	878	682	341



In addition to the primary expansion due to pyrrhotite oxidation, the released sulfate will react with the alumina-bearing phases of cement forming additional expansive minerals. This is referred to as secondary expansion with the reactions listed in Table 2 [9]. The damage caused by the secondary expansion is referred to as internal sulfate attack (ISA) [3].

The first secondary reaction is the formation of gypsum (Table 2, Reaction (IV), $C\bar{S}H_2$) while the second and third reactions are of this newly formed gypsum with tricalcium aluminate (C_3A) to generate monosulfoaluminate (Table 2, Reaction (V), $C_4A\bar{S}H_{12}$) and another form of ettringite (Table 2, Reaction (VI), $C_6A\bar{S}_3H_{32}$) called delayed ettringite formation (DEF) [13]. This type of ettringite is formed long after the concrete has hardened and is to be differentiated from the ettringite that formed in the first days of cement hydration by the reaction between gypsum and C_3A of cement. Furthermore, thaumasite (another expansive mineral) can be formed if sources of carbonate are available [3]. The formation of ettringite and thaumasite long after the concrete has hardened is referred to as secondary mineral formation (SMF) in this research.

2. Research significance

Though the SMF is believed by many researchers to cause deterioration of concrete, the mechanisms involved remain controversial [3,4,14–21]. Furthermore, there is no uniform standard regulating the content of pyrrhotite allowed in aggregates for concrete. Spanish

Regulations of Structural Concrete (EHE) [22,23], in force between 1999 and July 2008, prohibits the use of aggregates containing oxidizable sulfur compounds (Article No. 28). It is clarified in the comments section that “oxidizable sulfurs (e.g. pyrrhotite, marcasite, and some forms of pyrite), even in small quantities are very damaging to concrete since, as through oxidation and hydration, they form sulfuric acid and iron oxide/hydroxide minerals”. Similar requirements are stated in ASTM C294-12 [24] (Section No. 13): “marcasite and certain forms of pyrite and pyrrhotite are reactive in mortar and concrete, producing a brown stain accompanied by an increase in volume that has been reported as a source of pop outs in concrete”. Recent European standard EN 12620:2008 [25] (Section 6.3.2) makes the following statement: “should the presence of oxidizable iron sulfides in the form of pyrrhotite be detected, the sulfur content provided by them, expressed in S, will be lower than 0.1%”.

This research aims at: (I) assessing the mineralogical, chemical and mechanical properties of deteriorated concrete and understanding the mechanisms and role of pyrrhotite in the deterioration of concrete; and (II) evaluating if the cause of the deterioration of concrete is related to the primary expansion from the oxidation of the pyrrhotite-bearing aggregate and the secondary expansion, or ISA, due to the SMF.

3. Materials and methods

More than 70 core samples were extracted from zones with different degrees of deterioration from seven selected houses in the affected area. For each core the diameter was 3 inches, while the length was 6 to 7 inches for those drilled out of the walls and 3 to 4 inches for the ones drilled out of slabs. The lengths of the cores drilled out of slabs were restrained by the slab thickness. An experienced third-party contractor was hired to carry out the coring process.

Table 2
Primary and secondary expansion reactions after [7].

	No.	Reaction	$\Delta V_{solids}(\text{cm}^3/\text{mole of sulfide})$
Primary	(I)	$8F_{1-x}S + \frac{31}{2}O_2 + 8H_2O \rightarrow 7(FeSO_4 \cdot H_2O) + SO_4^{2-} + 2H^+$	187.08
	(II)	$8F_{1-x}S + 21O_2 + 11H_2O \rightarrow 7Fe(OH)_3 + 8SO_4^{2-} + H^+$	6.04
	(III)	$8F_{1-x}S + \frac{67}{4}O_2 + \frac{25}{2}H_2O \rightarrow 7FeOOH + 8SO_4^{2-} + 16H^+$	0.64
	(IV)	$CH + \bar{S} + 2H^+ \rightarrow C\bar{S}H_2$	41.36
Secondary	(V)	$CH + C_3A + \bar{S} + 11H \rightarrow C_4A\bar{S}H_{12}$	182.89
	(VI)	$3CH + C_3A + 3\bar{S} + 29H \rightarrow C_6A\bar{S}_3H_{32}$	172.19

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