Construction and Building Materials 173 (2018) 775-785

Contents lists available at ScienceDirect

ELSEVIER

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

The application of a new oxidation mortar bar test to mixtures containing different cementing systems



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HIGHLIGHTS

• The applicability of oxidation mortar bar test to cementing systems was investigated.

• Extended exposure of blends with metakaolin to oxidizing solution causes significant expansion of the paste.

• The use of SCMs reduces expansion through reducing the permeation of oxidizing agents into the samples.

• The effects of SCMs on reducing oxygen permeation through concrete under field exposure needs to be investigated.

ARTICLE INFO

Article history: Received 16 June 2017 Received in revised form 27 March 2018 Accepted 3 April 2018 Available online 24 April 2018

Keywords: Iron sulphide-bearing aggregate SEM Expansion Metakaolin Cement paste

ABSTRACT

The effects of different cementing systems on the expansion of mortars containing iron sulphide-bearing aggregate was studied. Using a recently developed oxidation mortar bar test, the results showed that cementing systems containing low-calcium fly ash, metakaolin, slag, high-sulphate resisting Portland cement, or low heat of hydration Portland cement could reduce the expansion by 50–85%. The main suggested mechanisms behind the reduced expansion is the more refined pore structure of samples with SCMs, and the reduced C_3A of low heat of hydration Portland cement. The refined pore structure reduces the permeation of the oxidizing solution into the samples. The similarity of this to penetration of oxygen into concrete under field exposure needs to be determined. Soaking the samples for >3 h in the oxidizing agent can produce excessive expansion – not related to oxidation of iron sulphide phases – in samples with cementing blends containing reactive alumina such as metakaolin.

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1. Introduction

Cases of severe concrete damage due to the oxidation of iron sulphide minerals in some types of concrete aggregate have been identified in different locations around the world. In 1974, Bèrard et al. [1] reported a case of sulphide damage in concrete foundation. The aggregates used in the concrete contained trace amounts of pyrite (FeS₂) and pyrrhotite (Fe_(1-x)S). The same authors indicated that the reaction that caused the expansion and deterioration in concrete was the oxidation of the pyrrhotite. Similarly, a case was found in Penge, South Africa, in 1979 and reported by Oberholster and Kruger [2]. Lugg et al. [3] reported cases of damage in Cornwall and Devon areas in the UK. They attributed the incident

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to the use of mudrock containing iron sulphide phases during the period from 1900 to 1950. Lugg et al. [3] confirmed that pyrite was the most problematic mineral in the mudrock and that the damage was aggravated by the high relative humidity. Schmidt et al. [4] reported that higher concentration of oxygen, a higher level of pH, and smaller size of sulphide mineral particles lead to the faster disintegration of the iron sulphide-bearing aggregates. The mechanism of damage due to oxidation of iron sulphidebearing aggregates has been reported in the literature [5]. In summary, the iron sulphide phases, particularly pyrrhotite and some types of pyrite, are unstable upon exposure to oxygen and humidity. Under this exposure, they oxidize and result in the formation of sulphuric acid and ferrous ions [5,6]. Then, the ferrous ions are oxidized to produce rust products including ferric hydroxide (ferrihydrite or rust) and FeOOH or ferric oxyhydroxide (goethite) [5]. The sulphuric acid reacts with one of the hydrated

Portland cement paste products, i.e. Ca(OH)₂, to form gypsum [7], which can then react with the aluminate phases in cement paste forming ettringite [8]. The formation of ettringite in hardened concrete can lead to expansion and disruption [8]. While a small amount of ettringite formation may not cause damage, the formation of an excessive amount within the cement matrix or at the interface between aggregate and paste is known to cause cracks [8].

Besides ettringite, thaumasite is also known as one of the products of sulphate attack, which can have deleterious effects on concrete structures. Thaumasite is a calcium-silicate-sulphatecarbonate-hydrate, which favorably forms at temperatures below 15 °C (preferably at 0-5 °C) [9], although it was also reported to form at temperatures above 20 °C [10]. The source of sulphate could be ettringite, where thaumasite can use it as a nucleation site [11], or the gypsum formed as a result of the oxidation of iron sulphides phases in aggregate [12]. Also, thaumasite was reported to form without the need of ettringite as a nucleation site [11,12]. Both calcium and silica usually come from calcium silicate hydrates (C-S-H). The source of carbonate might be from the air, which causes the transformation of calcium hydroxide to calcium carbonate, from carbonate fillers used to produce Portland cement, or from the aggregate itself. Thaumasite has received attention [11,13] as it consumes C-S-H resulting in strength loss. Other researchers [14] reported that thaumasite could cause expansion, in addition to decomposition of C-S-H.

More than 1000 residential houses and commercial buildings were affected by oxidation of iron sulphide-bearing aggregates in the Trois-Rivières area, Québec, Canada. The deterioration was in the form of pop-outs and map cracking and took place within the first 3–5 years after construction [15,16]. The aggregate used in this concrete was an igneous rock containing pyrite, and pyrrhotite. Following this case, research was carried out and an oxidation mortar bar test was suggested as part of a protocol to evaluate the potential deleterious character of iron sulphide-bearing aggregates [17,18]. This paper adopts the newly-developed mortar bar test

[17] to investigate the effects of different cementing blends on the damage due to oxidation of iron sulphide-bearing aggregates. The significance of this research is to find out whether or not a change in cementing materials can reduce the rate of damage due to the oxidation of iron sulphide minerals. Moreover, the research examines the possibility of the conditions of the test to promote reactions other than oxidation of iron sulphide phases in aggregates, and its subsequent sulphate attack, in samples with Supplementary Cementitious Materials (SCMs).

2. Materials and experimental program

2.1. Cementing materials

A general use Portland cement (GU-PC), low heat of hydration Portland cement (LH-PC), and high-sulphate resisting Portland cement (HSF-PC) were used in this study. The (HSF-PC) is a blended cement containing about 8% silica fume. Also, three types of SCMs were used, namely low-calcium fly ash (FA), slag (S) and metakaolin (MK). The SCMs were used at the partial replacement of GU-PC at 25% for FA (GU-FA), 30% for S (GU-S), and 10% for MK (GU-MK). The chemical composition of the cementing materials, along with the calculated phase contents of Portland cements are listed in Table 1.

The X-ray Diffraction (XRD) patterns and phase identification of the fly ash, metakaolin, and slag samples are shown in Fig. 1.

2.2. Aggregates

Three different coarse aggregates with different sulphur contents were used in this study, as follows:

(1) MAS: a sulphide-bearing aggregate from Québec, Canada, containing pyrrhotite, pyrite, chalcopyrite, and pentlandite. The average sulphide phases comprise about 1% of the aggregate with a total sulphur content of 0.73–1.28% [17].

Table 1

Chemical co	mposition	of the P	ortland	cement	and SCMs	(mass%)	determined	using	X-ray	Fluorescence	(XRF)	۱.
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Material Type	GU-PC ¹	LH-PC ²	HSF-PC ³	FA ⁴	S ⁵	MK ⁶
LOI (1000 °C),%	2.71	2.10	6.89	_	-	-
LOI (750 °C),%	-	-	-	2.78	2.90	1.82
SiO ₂ ,%	19.3	20.2	23.4	47.4	37.6	63.1
Al ₂ O ₃ ,%	5.22	4.00	4.22	23.9	8.23	30.7
Fe ₂ O ₃ ,%	2.14	3.60	2.66	17.4	0.50	1.22
CaO,%	62.0	63.8	54.9	3.67	38.1	0.36
MgO,%	2.37	2.60	2.28	1.00	10.8	0.50
SO ₃ ,%	3.91	2.90	3.48	0.40	2.72	0.05
K ₂ O,%	1.14	_	0.86	1.84	0.52	1.77
Na ₂ O,%	0.23	0.54	0.25	0.65	0.32	0.16
TiO ₂ ,%	0.27	-	0.23	1.23	0.53	0.68
SrO,%	0.09	_	0.22	0.13	-	0.04
P ₂ O ₅ ,%	0.12	_	0.21	0.29	0.02	0.03
Cl,%	0.04	_	0.01	-	0.61	-
ZnO,%	0.01	_	0.06	0.02	-	0.01
Cr ₂ O ₃ ,%	0.01	_	0.01	0.03	-	0.01
Mn ₂ O ₃ ,%	0.06	-	0.07	0.04	-	0.01
Phase compositions of G	U-PC and LH-PC calculated	d using Bogue's equations				
C ₃ S (%)	67	61				
C ₂ S (%)	5	11				
C ₂ A (%)	10	4				

¹ GU-PC: General Use Portland cement.

² LH-PC: Low heat of hydration (or low C₃A) Portland cement.

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³ HSF-PC: High Sulphate Resisting Portland cement, GU-PC blended with 8% silica fume.

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⁴ FA: Fly ash (low Calcium).

⁵ S: Slag.

C₄AF (%)

⁶ MK: Metakaolin.

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