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Microstructure and composition of fly ash and ground granulated blast furnace slag cement pastes in 42-month cured samples

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HIGHLIGHTS

- Fly ash offered greater capability in increasing Si/Ca ratio than slag.
- Al/Si ratio in C-A-S-H can be estimated by using SEM-EDS.
- The formation of Mg-Al LDH interbedded with TAH in slag paste was strongly suggested.
- MCL of aluminosilicate anions in GGBFS paste was much greater than in other pastes.
- MCL_{Si} of pure silicate anions in the pastes was approximate.

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ABSTRACT

Microstructure and composition of 42-month cured pastes using Portland cement (OPC), fly-ash cement (FAC), and ground-granulated-blast-furnace-slag cement (GGBFSC) were investigated. The granular structure of C-A-S-H in outer products of GGBFSC and FAC pastes was finer than in OPC paste. Fly ash offered higher capacity in increasing the Si/Ca ratio of C-A-S-H than slag in the pastes. However, their effects on the chain length were opposite. The incorporation of aluminum into silicate anions in GGBFSC paste was greater compared to others. AlO_4^{5-} played an essential role in bridging the shorter silicate anions together, forming a longer chain of aluminosilicate anions in the pastes. Mg-Al layered-double hydroxide (LDH) was abundant in the inner products of slag particles with the interbedding of TAH, leading to a low ratio of Mg/Al ratio on hydrotalcite-like phase.

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1. Introduction

Cement-based materials appear to be the most important materials used in all branches of civil engineering. Extensive research on both cement paste and concrete have provided more knowledge and understanding on the nature of cement hydration products, which determine not only the physical and mechanical properties but also the durability of the structures. Among these studies, the microstructure and composition of calcium silicate hydrates (C-S-H) garner an important attention. C-S-H is the primary product of cement hydration and forms approximately 50%–60% of the paste volume [1]. The characteristic of C-S-H phase is variable and unstable. They are influenced by many factors such as cement composition, water to cement ratio, and curing condition. These factors challenge the characterization of C-S-H [1–6]. In addition, the intermixing of C-S-H with other phases in cement hydration products at nanoscale causes a great obstruction of the characterization of C-S-H composition [1,6]. Therefore, the microstructure and composition of C-S-H are still not well-known despite a number of extensive studies.

Supplementary cementitious materials, which are usually by-products or waste materials, have been used widely in cement and concrete industry recently. Their utilization concerns not only the environmental issue but also involves the performance of concrete, the durability of structure, and the construction technology [7,8]. These supplementary cementitious materials affect the outer products (OP), which are formed in the originally water-filled space,

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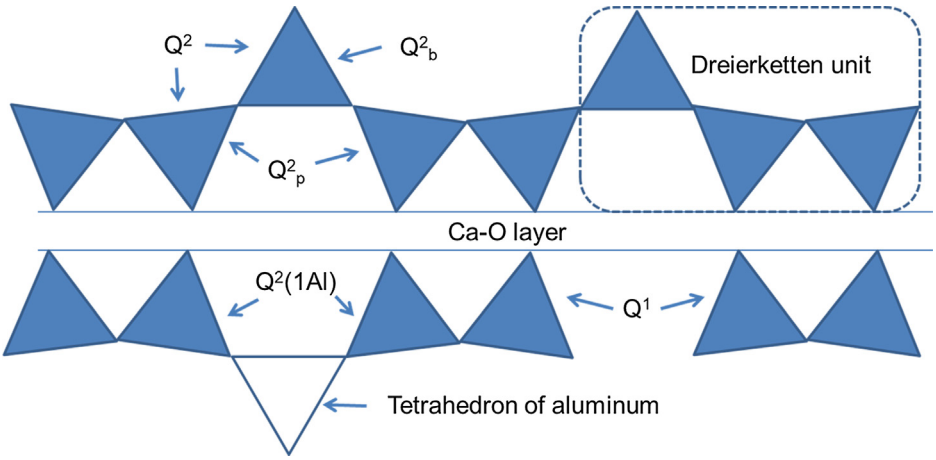

Fig. 1. Schematic illustration of C-A-S-H – Each triangle represents a silicate/aluminate tetrahedral unit [1,6].

as well as the inner products (IP) of cement-based materials, which are produced in the cement-filled space [1]. Furthermore, the composition and properties of supplementary cementitious materials usually vary, even from the same source. These variations impact the hydration process, the microstructure and composition of C-S-H as well, making it far from being well-defined [1,6–8].

At the atomic level, the structure of C-S-H is assumed to be similar to tobermorite. Its structure comprises of calcium oxide layer sandwiched between silicate chains on both sides. The silicate chain consists of repeating dreierketten units. However, the structure of C-S-H in the real cement paste can be incorporated by other ions either in calcium oxide layer, silicate chain or in the interlayer of C-S-H [1,6]. Aluminum was detected in the position of silicon in the silicate chain, especially in the paste using supplementary cementitious materials which contain high content of aluminum such as metakaolin, fly ash, or GGBFS, as illustrated in Fig. 1 [1,6]. Therefore, many researchers have preferred to use C-A-S-H as a more appropriate term to address the primary phase in cement-based materials rather than C-S-H [9–13].

It should be noted that the long hydration process of cement is one of the primary factors limiting the understanding the nature of C-A-S-H while the timeframe of research is usually limited. Reports are predominantly on synthetic C-A-S-H [9–12] or immature cement pastes [13–24] rather than on the really mature cement paste [25]. Although these investigations certainly contribute to critical knowledge on the understanding of the nature of C-A-S-H, it is necessary to employ research on the real mature paste.

The main purpose of this research is to characterize the composition and microstructure of mature pastes on various types of cement. Three types of cement were used for this study, namely OPC, FAC, and GGBFSC. These cement pastes were cast and cured for 42 months before characterizing by means of TG-DTA, XRD, SEM-EDS, and solid-state ^{27}Al and ^{29}Si magic angle spinning nuclear magnetic resonance. Another purpose of this work is to verify the feasibility of SEM-EDS in determining Al/Si ratio of C-A-S-H, which is usually affected by aluminate hydrate phases.

2. Materials and methods

2.1. Raw materials and sample preparation

The chemical compositions of cement, fly ash, and ground granulated blast furnace slag (GGBFS) are provided in Table 1 while the physical properties are presented in Table 2. FAC consisted of 80% OPC and 20% fly ash. GGBFSC is composed of 60% OPC and 40% GGBFS. Three types of cement including OPC, FAC, and GGBFSC were mixed with deionized water at the water-to-binder ratio of 0.4. The pastes were demolded and cured in saturated calcium hydroxide solution at room temperature (~23 °C). The samples were cured for 42 months. After curing, the samples were immersed into 2-isopropanol for 3 days in which the 2-isopropanol was replaced every day in order to stop the hydration process. After that, they were dried in desiccators for 10 days before taking the analysis. A duplicating set of paste samples with the addition of sodium chloride was also mixed and cured for 3 months to use for another aim of study [26].

2.2. Experimental

X-ray diffraction (XRD) measurements were carried out on paste powders by using Rigaku MiniFlex X-ray diffractometer using Cu Kα radiation in scan range of 5°–65° at the step width of 0.02 and the scan speed of 2°/min. TG-DTA were performed from 25 °C to 800 °C on paste powders at the constant rate of 10 °C/min under a constant flow of nitrogen.

Table 2
Physical properties of raw materials.

	Specific gravity (g/cm^3)	Blaine fineness (cm^2/g)	Activity index (%)		
			7d	28d	90d
OPC	3.16	3340			
Fly ash	2.16	3610	–	87	99
GGBFS	2.91	4030	73	97	113

Table 1
Chemical composition of raw materials.

	SiO_2	Al_2O_3	FeO	CaO	MgO	SO_3	Na_2O	K_2O	TiO_2	MnO	Total	IL
OPC	21.9	5.2	3.1	64.4	1.4	1.7	0.2	0.4	0.3	0.3	98.9	2.26
Fly ash	66.42	18.88	3.63	0.90	0.54	0.15	0.04	1.23	0.82	0.03	92.64	1.12
GGBFS	34.3	15.2	0.5	42.8	5.9	0.1	0.2	0.3	0.5	0.5	100.5	0.3

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