Construction and Building Materials 120 (2016) 112–122

Contents lists available at ScienceDirect

Construction and Building Materials

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

Microstructural investigations of palm oil fuel ash and fly ash based binders in lightweight aggregate foamed geopolymer concrete

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HIGHLIGHTS

- POFA and FA are used to fully replace cement to produce geopolymer concrete.
- Micrograph showed OPSFGC of different densities comprises different types of pores.
- XRD analysis showed partial dissolution of crystalline phase on OPSFGC over time.
- FTIR analysis showed presence of amorphous aluminosilicate in OPSGC.
- Addition of foam in OPSGC creates more pores and crack paths in ITZ.

ARTICLE INFO

Article history:
Received 21 November 2015
Received in revised form 4 May 2016
Accepted 9 May 2016
Available online 20 May 2016

Keywords:
Foamed concrete
Geopolymer
Oil palm shell
Palm oil fuel ash
FESEM
EDX
X-ray diffraction
FTIR

ABSTRACT

This article presents the development of lightweight-foamed concrete with geopolymer technology by utilizing industrial wastes such as oil palm shell (OPS) as lightweight coarse aggregate, palm oil fuel ash (POFA) and low calcium fly ash (FA) as binders in the concrete. The main aim of this research is the assessment of morphology and mineralogy on oil palm shell foamed geopolymer concrete (OPSFGC) and POFA-FA geopolymer (PFG) paste, respectively. These evaluations were performed by using field emission scanning electron microscopy (FESEM), energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD) analysis and Fourier transform infrared (FTIR) spectroscopy. Additionally, the compressive strength, water absorption and sorptivity of the geopolymer concrete were correlated with the foam volume. The results of the morphology of the OPSFGC confirm that a more dense and homogeneous geopolymer matrix is formed over the curing period, which results in higher compressive strength. Yet, concrete degradation tends to occur by the propagation of microcracks in the interfacial transition zone (ITZ). Further, the water absorption and sorptivity of OPSFGC show a positive correlation with the foam volume, which reduces concrete strength. Additionally, OPSFGC is able to achieve high early strength of up to 87% of its 28-day compressive strength owing to the oven curing.

1. Introduction

Modern concretes are produced with raw materials such as fly ash (FA), ground granulated blast furnace (GGBS), silica fume (SF) etc. However, ordinary Portland cement (OPC) is still an indispensable material in the modern concrete production [1]. The production of OPC is deemed as one of the main contributors towards the global warming caused by the emission of the carbon dioxide (CO_2). Davidovits [2] reported that the production of 1 ton of cement generates 0.55 tons of CO_2 and involves the combustion of carbon-fuel to yield an additional 0.40 tons of CO_2. Thus, the worldwide production of about 1 billion metric tons of cement produces approximately equal mass equivalent of CO_2. Other than cement, the production of concrete involves the use of sand and aggregate, the quarrying operations of which are energy intensive and release high level of waste [3]. Malaysia the second largest palm oil producer, contributes large quantities of waste such as empty fruit bunches (EFB), palm oil clinkers, oil palm shells (OPS) and palm oil fuel ash (POFA) and these unutilized wastes cause land and air pollution. In the context of sustainability, it is imperative to put some of these waste materials to use.

Geopolymer was first introduced by Davidovits [4] to define cement-less concrete that can be produced by the reaction between alkaline solutions and source materials that are rich in silica and alumina, aided by heat curing and drying [5]. The use of

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http://dx.doi.org/10.1016/j.conbuildmat.2016.05.076
0950-0618/

geopolymer technology not only substantially reduces the CO_2 emissions by the cement industries, but also utilizes fly ash (FA), the industrial waste material [5]. Utilizing agricultural and industrial wastes in geopolymer concrete could lower its compressive strength loss compared to OPC concrete [6]. Geopolymer concrete also demonstrated lower sorptivity when compared with the OPC concrete [7].

The alkali activation of aluminosilicate is acknowledged for the formation of amorphous aluminosilicate gel, which leads to excellent mechanical and chemical properties [2,8]. Therefore, it is essential to study the microstructural characteristics of the geopolymer concrete, especially with the addition of new cementitious materials or aggregates. In addition, this microstructure investigation is vital to comprehend the performance of OPSGC with the introduction of foam. The use of local industrial waste material i.e. POFA in geopolymer concrete is relatively new. Therefore, the microstructural investigation is essential to define the characteristic of the concrete. Field emission scanning electron microscopy/energy dispersive X-ray analysis (FESEM/EDX), X-ray diffraction analysis (XRD) and Fourier transform infrared spectrometry (FTIR) techniques have been used in previous research to characterize FA, POFA, microwave incinerated rice husk ash (MIRHA) and waste concrete powder (WC) [3,6,9]. The occurrence of geopolymerization can be distinguished from the pattern formation of XRD and FTIR [3,10]. There are few types of curing methods for geopolymer concrete i.e. via elevated temperature and ambient. These methods can be analysed using FESEM/EDX to determine the characteristic of geopolymer matrix connectivity [9].

The development of lightweight aggregate concrete (LWAC) through the use of OPS as lightweight aggregate (LWA) with a density of less than 2000 kg/m^3 fulfilled the requirement of LWAC as stipulated in BS EN 206-1 [11]. The use of crushed OPS in concrete manufacturing could increase strength of concrete compared to the concrete with uncrushed OPS [12]. Though the addition of foam in the OPSC reduced the density to 1500–1600 kg/m^3, the foamed OPSC with structural and insulating characteristics was found to have exhibited 37–39% lower thermal conductivity compared to the conventional brick [13]. In their previous work, Liu et al. [14,15] developed a structural grade geopolymer concrete and foamed geopolymer concrete, which contributed higher thermal insulation than the conventional brick by 67%.

The aim of this research study is to determine characteristics of the PFG paste using XRD and FTIR tests. Also, this study focusses on the effect of foam in geopolymer concrete; FESEM/EDX tests were conducted to investigate the mechanical and transport properties.

2. Materials and methods

2.1. Materials

The binders used in the development of geopolymer products are Class-F FA and POFA, which are sourced from the local power station and palm oil mill, respectively. The chemical compositions of both binders that conform to the ASTM C618 [16], are listed in Table 1. The raw POFA obtained from the palm oil mill was sieved through 300 μm to remove coarser foreign particles after oven-dried at 105 ± 5 °C for 24 h. Next, the sieved POFA was ground with the Los Angeles abrasion machine. Both binders have a mean particle size of about 45 μm. The ratio of binders to alkaline solution of 0.55 was used for all geopolymer specimens.

Table 1
Chemical composition of Class-F FA and POFA.

Chemical composition (%)	Class-F FA	POFA	ASTM C618
Silica (SiO_2)	57.6	63.4	–
Ferric oxide (Fe_2O_3)	5.8	4.2	–
Calcium oxide (CaO)	0.2	4.3	–
Magnesium oxide (MgO)	0.9	3.7	–
Potassium oxide (K_2O)	0.9	6.3	–
Sulphuric anhydride (SO_3)	0.2	0.9	⩽4.0
Alumina (Al_2O_3)	28.9	5.5	–
Loss of ignition (LOI)	3.6	6.0	⩽10.0
$SiO_2 + Al_2O_3 + Fe_2O_3$	92.3	73.1	⩾70.0

Mining sand with specific gravity of 2.67 was used as fine aggregate. Different gradations of the fine aggregates were used for different types of specimens, which are shown in Fig. 1. Finer mining sand was used in OPSFGC whereas the coarser sand was used in OPSNFGC. Meanwhile, OPS was used as lightweight coarse aggregate, of which the physical properties are presented in Table 2 and Fig. 1.

The binders were activated by the alkaline solution, which is a combination of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3). The alkaline solution was prepared at least 1 day prior to its use to allow the exothermically heated solution to cool down to ambient temperature. The ratio of Na_2SiO_3 to NaOH was maintained at 2.5 with a NaOH concentration of 14M for all mixtures. Ratio of binder to alkaline activator of 0.55 was used for all geopolymer specimens. Potable water was used for all mixtures. A polycarboxylic ether-based superplasticizer was used for geopolymer concrete at the dosage of 1.5% by mass of binder. The foaming agent, Sika AER-50/50 was used to develop the OPSFGC by adding preformed foam into the mixture using foam generator. The air pressure of the foam generator was maintained at 75 psi.

2.2. Specimen preparation

Two types of mixtures, the geopolymer concrete (OPSNFGC and OPSFGC) and geopolymer paste (PFG paste) were prepared. The mixture proportion of all mixtures is given in Table 3. The OPS was washed and kept in a saturated surface dry (SSD) condition before use. After that, the OPS and fine aggregates were first mixed together in rotary mixer for about 2 min. Then, the binders were added and mixed for another 3 min. A further mixing of about 5 min was done after the addition of alkaline solution, water and SP. The fresh concrete was then poured into steel moulds and covered with plastic layer, and then cured at temperature of 65 °C for 48 h. The specimens were then de-moulded for further testing and analysis.

In order to produce OPSFGC, the pre-formed foam was added after the mixing of liquids in the rotary mixer. The OPSFGC specimens were cast with two different target densities, namely 1300 and 1500 kg/m^3. The PFG pastes were prepared by excluding the coarse and fine aggregates, SP and foam.

The specimens of OPSFGC and OPSNFGC were prepared in 100-mm cube moulds to test their compressive strengths at the age of 3-, 7-, 14-, and 28-day. Similar size of specimens was also used for the 28-day sorptivity test. The water absorption test was carried out with the disc specimen of size 100 ϕ × 50 mm. On the other hand, the PFG paste specimens were cast in 50-mm cube moulds for the compressive strength test at 3-, 7-, 14- and 28-day.

2.3. Experimental methods

Both types of the geopolymer specimens were tested for the compressive strength in accordance with BS EN 12390-3 [17]. The average results were obtained from the triplicate specimens at the age of 3-, 7-, 14- and 28-days. The water absorption test was performed according to ASTM C642 [18]. The specimen was oven-dried at 105 ± 5 °C for 48 h to remove any moisture content. After that, the weight of the specimen was measured soon after immersed in water for 30 min to determine the initial water absorption. The water absorption was calculated using Eq. (1):

$$Water\ absorption\,(\%) = \frac{W_s - W_d}{W_d} \times 100 \quad (1)$$

where W_s is the SSD mass of the test specimen in air after immersion (g); W_d is the OD mass of the test specimen in air (g).

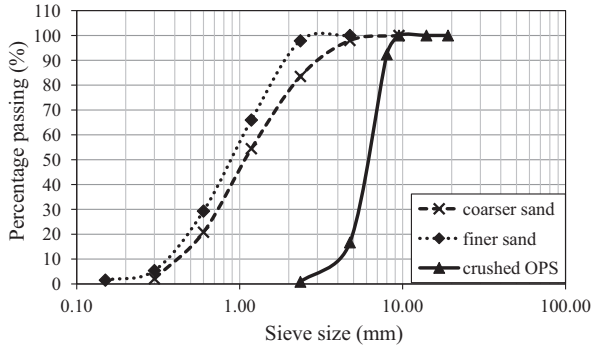

Fig. 1. Grading of OPS and mining sands.