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Compressive strength and microstructural properties of dry-mixed geopolymer pastes synthesized from GGBS and sewage sludge ash

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HIGHLIGHTS

- Geopolymer pastes of adequate strength can be prepared by using sewage sludge ash (SSA) as a precursor.
- The dissolution of hematite in the SSA was confirmed by XRD, QXRD, FTIR and EDX.
- A C-(N)-A-S-H type gel with Fe substitutions was formed in the optimal mixture.
- A theoretical basis was built up to explain strength gains in geopolymerization of SSA to facilitate its use.
- Drying shrinkage was well below the 0.06% allowable limit.

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ABSTRACT

Mixed solids (precursors) containing equal portions of ground granulated blast-furnace slag (GGBS) and sewage sludge ash (SSA) by weight were activated by mixed alkaline solutions that was comprised of sodium hydroxide and sodium silicate in prescribed proportions to prepare geopolymer pastes in a dry-mixed form. Tests were carried out to provide data on compressive strength and microstructural properties of the geopolymer pastes synthesized from different mass ratios of alkali to the mixed solids, i.e., Na_2O content, and different molar ratios of SiO_2 to Na_2O in the mixed alkaline solution (i.e. different moduli). The results showed that the maximum compressive strength of 32.8 MPa at 28 days could be achieved at the optimal Na_2O content of 4.0% and a modulus of 0.95. Analyses of the microstructures of the geopolymer pastes by XRD, QXRD, FTIR, SEM and EDX revealed that SSA participated in the geopolymerization process and the quartz and hematite crystals in it were largely dissolved in the reaction. The microanalyses also revealed that the main reaction product of the optimal geopolymer mixture was a C-(N)-A-S-H type gel with Fe substitutions. The drying shrinkage of all specimens were less than 0.06% at the age of 14 days. Testing for metal(loid)s showed that SSA could be used with GGBS as precursor materials for geopolymerization with no leaching problems. This work provides a potential option to recycle SSA and builds up a theoretical basis for proportioning design in utilizing GGBS and SSA in dry-mixed geopolymers.

1. Introduction

Portland cement is the most widely used binder material in building and other infrastructure construction. However, cement production utilizes natural resources, consumes high energy and generates huge amounts of carbon dioxide. Since the first research conducted by Joseph Davidovits in 1979 [1], vast number of studies have been conducted on geopolymers. Typical source materials for making geopolymers are those rich in silicon (Si) and aluminum (Al). It was suggested that the production of geopolymers is an environmental-friendly process in which three-dimensional aluminosilicate materials are synthesized [2,3]. The process involves the dissolution of Si and Al species from the precursor materials by an alkaline media as well as possible surface hydration of the particles of the precursors, followed by coagulation and gelation of the dissolved minerals into monomers which undergo further polycondensation to form a gel and subsequently a hardened geopolymer structure [4]. Many industrial by-products such as ground granulated blast-furnace slag (GGBS) [5] and fly ash (FA) [6] have been found suitable for producing geopolymers. To reduce problems of greenhouse gases emission and disposal of industrial by-products, geopolymers are promising materials to replace cement as alternative binder materials in construction works

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because they exhibit equal or even superior mechanical [7,8], durability [9,10] and high temperature [11,12] properties when compared with ordinary Portland cement (OPC) based products.

Sewage sludge ash (SSA) is produced from thermal treatment of dewatered sewage sludge. According to the official website of the Environmental Protection Department in Hong Kong [13], this city currently produces approximately 1200 tonnes of dewater sewage sludge every day and the amount may grow to 2000 tonnes a day in 2030. Through the sewage sludge incineration plant (the largest in the world at this moment), the sludge is incinerated to SSA and the volume of the waste can be reduced by 90%. Despite the benefits with incineration, the ash still needs to be treated properly and the current method is disposal in landfills. However, Hong Kong is facing an acute shortage of landfill space since the existing landfills will reach their capacities in a few years [14] while further extension is almost impossible due to strong objections from the public [15]. To relieve the burden on the landfills, recycling of SSA should be explored.

From a broad review of the literature, different ways of utilization of SSA have been explored. Some studies have been conducted on sintering materials composing SSA, including clay with SSA to produce bricks and tiles [16,17], clay and sewage sludge with SSA to produce lightweight aggregates [18,19], and SSA alone or with additives to manufacture ceramic and glass-ceramic products [20,21]. Another application of SSA which has attracted much research interest is its use in cement-based materials. Under this application, SSA is added as a supplementary cementitious material to partially replace cement in mortar or concrete [22,23]. The main drawbacks of some of the above applications include energy consumption in sintering, greenhouse gases emission, natural materials consumption and the need to use OPC.

The potential of using GGBS to produce geopolymer concrete is well known but knowledge on geopolymerization of SSA or its effects on other geopolymer materials remain limited. SSA is a potential precursor material of geopolymer by composition. It is rich in SiO_2, Al_2O_3 and CaO [24,25], which are generally regarded as the most active compounds for geopolymerization. From the limited literature available, one study reported on the production of geopolymer concrete at ambient temperature using FA and SSA as precursor materials and sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) as alkaline activators and it revealed that the mechanical strength decreased with a higher incorporation percentage of SSA [26]. Another study on the geopolymerization of FA and SSA with NaOH and Na_2SiO_3 under 80 °C steam reported that SSA could improve the mechanical strength of FA-based geopolymer pastes with the best result attained at a ratio of 1 part SSA to 3 parts FA [27]. But it was shown that SSA made little contribution to geopolymerization when blended with metakaolin, particularly at ambient temperature [28]. On the other hand, more encouraging results were obtained in two studies which showed improved geopolymerization of GGBS with SSA added by using NaOH as the alkaline activator. The best result was achieved at a ratio of 1 part SSA to 4 parts GGBS [29] or 7 parts SSA mixed with 2 parts quick lime and 1 part GGBS [30]. This result supported the use of SSA for partial replacement of GGBS in producing geopolymers. Drying shrinkage of SSA based mortar was also evaluated in the study [30]. It was reported that the drying shrinkage of mortars increased with the addition of NaOH and quick lime but such an effect could be counterbalanced by the use of appropriate amounts of GGBS.

All the above studies were carried out on preparing the geopolymers on a wet-mix basis (i.e. the prepared mixes were of sufficient fluidity to achieve full compaction when cast into moulds). This present study however aimed at examining geopolymerization of dry-mixed geopolymer mixtures [31,32], (the mixtures had little fluidity and compacting was achieved by external compressive force) comprising equal weight of GGBS and SSA using a binary alkaline activator, namely, NaOH and Na_2SiO_3. The dry-mixed casting method allowed instant demoulding of the specimens which enables highly efficient production of precast geopolymer concrete blocks.

In this study, focus was on assessing the effects of weight percentage of alkali to the mixed solids, i.e., Na_2O content, and molar ratio of SiO_2 to Na_2O in the mixed alkaline activator, i.e. modulus, on the compressive strength of the geopolymer material, and the microstructure characteristics of the geopolymer. The compressive strength of the hardened geopolymer pastes were tested and their microstructure and composition were analyzed by qualitative and semi-quantitative X-ray diffraction (XRD and QXRD), Fourier transform infrared spectroscopy (FTIR), as well as scanning electron microscopy and energy dispersive X-ray spectroscopy (SEM and EDX). Since long term shrinkage is a major durability concern and drying shrinkage generally accounts for most of the overall long-term shrinkage [33], this study also provided preliminary results of drying shrinkage tests for reference in further durability studies and supporting the practical value of recycling GGBS and SSA as dry-mixed geopolymers. As sewage sludge is a residue from wastewater treatment in which metal(loid)s accumulate and such metal(loid)s may remain in the ash after thermal treatment of sewage sludge, the leaching of metal(loid)s is also a concern in recycling SSA [34–36]. Therefore, the leachability of metal(loid)s from the raw mixed precursors and the geopolymer products were tested.

2. Materials and experimental procedures

2.1. Materials

The GGBS was obtained commercially and the SSA was directly collected from the sewage sludge incinerator in Hong Kong. GGBS and SSA were analyzed for oxide compositions, particle size distributions, mineralogy and morphology using X-ray fluorescence (Rigaku Supermini200), laser size diffraction (Malvern Instrument's Spraytec), XRD and SEM respectively. The physical properties and oxide compositions of GGBS, SSA and the mixed solids are shown in Table 1. CaO, SiO_2 and Al_2O_3 were the most abundant components presented in GGBS. The major components in SSA were SiO_2, Fe_2O_3, Al_2O_3 and CaO. The high content of Fe_2O_3 in the SSA was due to the use of ferric chloride in the primary treatment of sewage in Hong Kong. Fig. 1 shows the particle size distributions of GGBS, SSA and the mixed solids. From the results, it can be seen that the GGBS was finer than the SSA. The particle size distribution of the GGBS ranged from 0.869 μm to 33 μm with a median diameter of 4.38 μm and the particle size of the SSA ranged from 2.92 μm to 194.2 μm with a median diameter of 51.24 μm. The mixed solids had a continuous non-homogeneous grading with a median diameter of 26.81 μm.

Mixtures of NaOH and water glass i.e. Na_2SiO_3 were used as activators. NaOH with a purity of 98% in pellet form and Na_2SiO_3 comprising 28.3% SiO_2, 8.6% Na_2O and 58.4% water by weight were sourced commercially. The NaOH solution was prepared by dissolving NaOH pellets in water. The solution was stirred for at least 10 min to ensure all the pellets were dissolved. After cooling, the NaOH solution was mixed thoroughly with Na_2SiO_3 in prescribed proportions to prepare the alkaline activators.

2.2. Mix proportioning

The mix proportions of the geopolymer specimens are shown in Table 2. The pastes were made by mixing the solid precursors with the alkaline solutions. The mixed solids were prepared by mixing