



Microstructure, strength, and moisture stability of alkali activated glass powder-based binders



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ABSTRACT

Thermally assisted alkali activation of silica-rich glass powder to produce sustainable binders is investigated. Glass powder activated using NaOH provides higher compressive strengths than NaOH activated fly ash binders at lower heat curing temperatures. Sodium silicate gel is the reaction product when glass powder alone is used as the source material, while a combination of sodium silicate and sodium aluminosilicate (N–A–S–H) gels form in activated glass powder–fly ash blends. The activated glass powder-containing binders are found to disintegrate and lose strength when exposed to moisture or an alkaline solution, with the pure glass powder binders suffering the highest strength loss. Structural changes to the reaction product on exposure to moisture are explained using microstructural and FTIR spectroscopic observations. Doping the systems with Al containing (metakaolin) and Ca containing (slag) source materials, while retaining glass powder as the major component (50% or more), result in the formation of moisture-stable reaction products thereby mitigating the strength loss to a large extent.

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1. Introduction

Alkaline activation of aluminosilicate materials such as fly ash, ground granulated blast furnace slag, and metakaolin has been shown to be capable of producing sustainable binders to replace ordinary Portland cement. The use of these natural or industrial by-product materials results in significant environmental benefits such as the decrease in CO₂ emission and energy consumption attributed to Portland cement production, and the conservation of non-replenishable natural resources [1–4]. In addition, this approach allows for increased utilization of materials like fly ash, the management of which is a concern.

Among the source materials used to produce alkali activated concretes, fly ash and slag are the most commonly studied because of their abundant availability and the presence of soluble silica and alumina contents in these materials that undergo dissolution, polymerization with the alkali, and solidification that provides strength and stability to these matrices [5–10]. Alkali hydroxides or silicates of varying concentrations are generally used as the activating agents. The processing conditions required to produce binders of desirable mechanical and durability properties change with the source material chemistry. For instance, activated fly ash binders require mild to moderate thermal curing in order to facilitate

hardening and the formation of the alkali aluminosilicate binder [4,7,11] whereas activated slag binders harden and develop strength under moist curing [10,12]. Several studies have reported the reaction product formation and property development in such mixtures [13–17], aiding in a better understanding of these binder systems.

While significant progress is being made on increased utilization of industrial waste materials such as fly ash and slag through alkali activation, there are materials from other waste streams that have the potential to be activated to form sustainable binders. Powdered glass is one such material, which is the focus of this paper. Municipal waste streams across the world generate millions of tons of glass every year. In the United States alone, 12 million tons of waste glass is generated annually, with only a quarter of it being recycled [18]. Fine glass powder has been reported to have adequate pozzolanic properties [19,20] and concretes made with partial replacement of cement by glass powder has shown comparable performance to that of fly ash modified concretes [21,22].

Glass powder is rich in silica, and when activated with alkalis, can result in the formation of sodium silicate gel. Only limited preliminary studies have been reported on alkali activation of glass powder. Limited mechanical properties of activated slag-glass powder mixtures have been evaluated, and is reported that increasing the glass powder content decreases the strength under moist curing conditions [23]. There is a need to understand the reaction product formation in binders that contain glass powder as a major component, and their properties so as to enable

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beneficial utilization of this material. The alkali silicate gels, when exposed to water, can leach alkali ions [24] that contribute to the destabilization of the gel structure. This is an important criterion that needs to be studied with respect to the durability of the activated glass powder binders. This paper evaluates the strength development in NaOH activated glass powder ($\sim 72\%$ SiO_2) mixtures, and compares the reaction products formed to those found in activated fly ash mixtures. The hydrolytic stability of the reaction products formed from glass powder activation is investigated and methods to produce water-resistant gels containing a major proportion of glass powder are outlined.

2. Experimental program

The glass powder used in this study is a by-product of industrial and highway safety glass bead manufacturing, with pozzolanic properties as reported earlier [19,21]. The manufacturing process produces the fine powder as a waste, necessitating no grinding or pre-treatment before the glass powder can be used as a source material in activated binder systems. This waste material is either being landfilled or used in limited amounts in fiberglass and paint manufacture. A Class F fly ash conforming to ASTM C 618 [25] is used in this study. The chemical composition of these starting materials are given in Table 1 and their particle size distributions, obtained using a laser particle size analyzer are shown in Fig. 1. Both glass powder and fly ash have very similar particle size distributions. The ground granulated blast furnace slag used in the ternary blends is a Type 100 slag conforming to ASTM C 989 [26], and the metakaolin for the ternary blends is classified as a Class N pozzolan according to ASTM C 618 [25]. Slag and metakaolin were used as materials to partially replace glass powder in order to improve the stability of these binders under exposure to moisture. The slag used has a median particle size (d_{50}) of $8\text{ }\mu\text{m}$ and metakaolin has a d_{50} of $4\text{ }\mu\text{m}$. The chemical compositions of these materials also can be found in Table 1. Laboratory grade NaOH beads were dissolved in water to produce the activator solutions of desired concentration (4, 6, or 8 M). The solutions were prepared and allowed to return to ambient temperature before used in the mixtures in order to negate the effect of high temperatures caused by the dissolution of NaOH in water. Mortars were prepared with glass powder alone, 50% glass powder–50% fly ash (by mass) or fly ash alone as the starting materials. River sand (d_{50} of 0.6 mm) was used as the filler in mortars, which were proportioned using a liquid-to-powder ratio of 0.50, with approximately 40% paste volume fraction. The binder pastes used for the microstructural studies had a liquid-to-powder ratio of 0.40 because the flowability of pastes with a liquid-to-powder ratio of 0.50 was very high.

The source materials were mixed together in dry form in a laboratory mortar mixer for 2 min. The activator solution was then

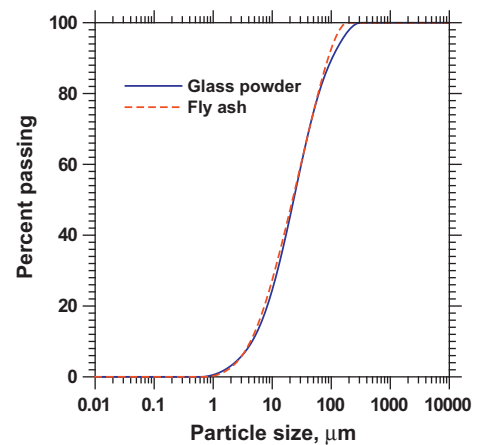


Fig. 1. Particle size distributions of glass powder and fly ash.

gradually added to the dry material and mixed together until a uniform mixture was obtained. After mixing, the specimens were cast in 50 mm cube molds and vibrated on a table vibrator until a homogeneous mixture was achieved. The specimens were covered with plastic sheets and allowed to set for 24 h in ambient conditions before being removed from the molds, and then were placed in the curing oven at the desired curing temperatures for the chosen durations. Heat curing was carried out at $50\text{ }^{\circ}\text{C}$ and $75\text{ }^{\circ}\text{C}$ for 24, 48, or 72 h.

The compressive strengths of the mortar cubes were tested after the respective curing regime (thermal curing, or moist curing after heat curing to determine hydrolytic stability) and after they returned to ambient conditions. To understand the morphology and the composition of the reaction products formed in NaOH activated glass powder pastes, morphological analysis was performed on Au sputter-coated samples using Scanning Electron Microscopy (SEM) coupled with Energy dispersive X-ray analysis (EDX). The samples for Fourier Transform Infrared (FTIR) spectroscopy were prepared by mixing approximately 1 mg of sample with 300 mg of KBr. The spectra of the reaction products at desired ages of curing were obtained using an ATI Mattson FTIR spectroscope in the wavenumber range of $4000\text{--}400\text{ cm}^{-1}$ at a resolution of 1 cm^{-1} .

3. Results, analysis, and discussion

3.1. Compressive strengths of NaOH activated glass powder mortars

The compressive strengths of mortars made using glass powder or fly ash alone or a combination of 50% of each of these materials by mass as the source materials are reported and discussed in this section. Two different NaOH concentrations, 4 M and 8 M, and two different curing temperatures, $50\text{ }^{\circ}\text{C}$ and $75\text{ }^{\circ}\text{C}$ were adopted for this part of the study. Since the specimens cured for 24 h at $50\text{ }^{\circ}\text{C}$ did not develop considerable strength, the curing durations adopted at this temperature were 48 and 72 h. Similarly, when the curing temperature was $75\text{ }^{\circ}\text{C}$, a duration of 72 h resulted in significant specimen cracking especially for the glass powder mortars, and therefore the curing durations at this temperature were restricted to 24 and 48 h.

Fig. 2(a) and (b) depict the changes in the compressive strengths of NaOH activated mortars as a function of the glass powder content for different NaOH concentrations, curing temperatures, and curing durations. It is noted that, in general, addition of glass powder increases the compressive strength of the activated mortars. The only exception is for the 8 M NaOH activated mixtures subjected to $75\text{ }^{\circ}\text{C}$ for 48 h, where the 50% fly ash–glass

Table 1
Chemical composition and properties of the starting materials.

Composition (% by mass)	Glass powder	Fly ash	Metakaolin	Slag
SiO_2	72.5	50.2	48.9	36.0
Al_2O_3	0.40	28.8	44.6	10.5
Fe_2O_3	0.20	5.72	0.47	0.67
CaO	9.70	5.86	–	39.8
MgO	3.30	1.74	–	7.93
Na_2O	13.7	0.96	–	0.27
K_2O	0.10	–	0.24	0.16
TiO_2	–	–	1.44	–
SO_3	–	0.51	–	2.11
LOI	–	2.80	–	3.0
Fineness, % passing 45 μm sieve	74	75	100	95

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