Construction and Building Materials 341 (2022) 127786

Contents lists available at ScienceDirect

Construction and Building Materials

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

Effect of immersing geopolymer slag-fly ash mortar in sulfuric acid on strength development and stability of mass

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ARTICLE INFO

Keywords:
Fly ash
Slag
Sodium silicate
Sodium hydroxide
Compressive strength
Tensile strength
Geopolymer mortar

ABSTRACT

This article presents the findings of a study aimed at assessing the effect of immersing mortar with alkali-activated ground granulated blast furnace slag and fly ash binders in highly acid solution, on compressive strength development and stability of mass. Effects of slag & fly ash contents on strength development and mass change were evaluated by testing mortars with three groups of binders including 100%slag, 75%slag + 25% fly ash, and 50% slag + 50% fly ash. The precursors were activated using a combination of sodium silicate (SS) and sodium hydroxide (SH) solutions. It was found that immersing samples in 10% sulfuric acid solution, one day after casting, didn't not cause decrease in strength or mass from 7 days to 90 days, and the pattern was consistent for NaOH concentrations from 10 M to 16 M. This was attributed to presence of water in the sulfuric acid solution which provided a closed curing environment. Similar samples cured by direct exposure to air developed lower compressive strength at the age of 28-days than samples immersed in sulfuric acid solution. At lower solution molarity (NaOH concentration = 10 M), mortars with 75% slag and 25% fly ash binder developed the highest compressive strength of 87 MPa after 28 days of immersion in sulfuric acid solution, compared to 49.85 MPa for samples cured in air, further confirming the positive effect of water as curing medium. The study also found a strong correlation between splitting tensile strength and compressive strength of air-cured mortar when the binder is 100% slag. The relationship weakens as fly ash content is increased from 0% to 50%.

1. Introduction

Ordinary Portland cement (OPC) is the dominant binder for concrete infrastructure, but its production has been associated with the release of significant amounts of CO_2 into the atmosphere which exacerbates global warming. In the post-COVID 19 environment, investment in infrastructure projects that fuels economy may increase leading to further demand for concrete binders. Many alkali-activated materials, such as ground granulated blast furnace (GBS), and fly ash produce concrete with competitive fresh properties, mechanical properties, and durability. Therefore, environmental concerns over the production of OPC and interest in producing concrete with improved qualities motivated the pursuit of alkali-activated materials as alternative binders. The use of GBS, fly ash, and silica fume in combination with OPC as binders in optimum quantities is known to enhance most concrete properties of interest to the construction industry [1]. GBS, a byproduct of iron manufacturing industry, and fly ash, a byproduct of coal-fired power stations must be landfilled periodically. Therefore, their reuse as binders for concrete structures saves valuable land space and decreases the environmental impact of these industries. Although properties of concrete/mortar with alkali-activated GBS binder has been studied and reported in the literature, several questions are still lingering with regard to long term strength and durability in aggressive environments, especially when the binder is a blend of GBS and fly ash [2]. Alkali-activated slag- fly ash binders are likely to produce enhanced concrete properties as they tend to compensate the shortcomings of each other while imparting additional favorable properties [2].

In OPC-based concrete, acids are recognized as a cause for strength and mass reduction. When low pH sulfuric acid comes in contact with concrete, it lowers the pH of the naturally alkaline concrete. If the pH of concrete drops below the stability limit of the hydrates, they lose calcium and transform into amorphous hydrogel. The acid attack reaction products include calcium salts of the acid and other hydrogels. Contact of OPC-based concrete with acidic solutions causes decomposition of calcium hydroxide along with development of gypsum, which may lead to softening and scaling. Sewerage systems are examples of environments that may bring highly acidic solutions in contact with concrete and cause substantial degradation of strength and mass in the long term [3,4]. Damage and mass reduction in concrete elements due to sulfuric acid attack were shown to increase with the content of OPC binder [5]. Prediction models by Ren et al [6] indicate that alkali-activated slag/fly ash concrete provides better resistance to sulfuric acid damage, compared to OPC-based concrete.

Gypsum caused by sulfuric acid attack formed mostly on the outer

https://doi.org/10.1016/j.conbuildmat.2022.127786
Received 30 December 2021; Received in revised form 3 May 2022; Accepted 5 May 2022
Available online 11 May 2022

surface of mortar when alkali-activated GBS is the sole binder, without penetrating to the inner core of the samples. The Al/Si ratio decreases in the outer layers affected by the attack, compared to inner layers that remain free of gypsum. Therefore, mortar with alkali-activated GBS perfumed well when immersed in sulfuric acid [7]. Resistance of mortar to sulfuric acid damage increased with GBS content when the binder consisted of alkali-activated combination of GBS and fly ash. The enhanced resistance to sulfuric acid may be due to the denser matrix that characterizes geopolymerization products of GBS, which restricts movement of sulfuric acid through mortar. On the other hand, mortar samples with alkali-activated fly ash experienced extensive damage in the form of compromised porosity caused by leaching of gypsum, when immersed in 3% sulfuric acid solution [8]. The vulnerability of mortar with alkali-activated class F fly ash binder was also reported by Bakharev [9], which transpired in the form of substantial mass loss and strength degradation. X-ray diffraction (XRD) studies and scanning electron microscope (SEM) images showed that the acidic medium caused depolymerization of aluminosilicates and formation of zeolites. Experimental study by Gu [10] demonstrated that alkali-activated fly ash-based mortar experienced substantial decrease in strength and mass after 492 days of immersion in 1% sulfuric acid.

In alkali-activated mortar/concrete with GBS-fly ash blended binder, the fundamental geopolymerization product is C-A-S-H gel [11]. When alkali-activated fly ash is one of the precursors, it develops sodium aluminosilicate (N-A-S-H) gel. C-A-S-H matrix is denser with less permeable pore system compared to N-A-S-H [12]. Uppalapati [13] reported that few hours after mixing mortar with a combination of GBS and fly ash binders, only the GBS-based C-A-S-H was found, along with limited amount of N-A-S-H, which was attributed to the slower reactivity of fly ash [13]. A product identified as C-N-A-S-H was also reported in alkali-activated mortar with GBS/fly ash blended binder, which may be a hybrid phase between C-A-S-H and N-A-S-H [14].

When immersed in 10% sulfuric acid solution, deterioration of mortar prepared with alkali-activated GBS/fly ash binder is related to corrosion caused by SO^{-4} penetrating through permeable voids, and the rate of water absorption [15]. Sulfuric acid attack on alkali-activated mortar with GBS/fly ash binder also causes decalcification of C-A-S-H gel which frees calcium to combine with SO^{-4} and create gypsum. C-A-S-H, which is vulnerable to decalcification due to sulfuric acid, is typically the result of geopolymerization of GBS, while N-A-S-H, which doesn't experience decalcification is formed by geopolymerization of fly ash [16]. However, C-A-S-H forms denser matrix, which impedes acid penetration through pore system, contrary to N-A-S-H. Therefore, as GBS content increases in GBS/fly ash blended binder, resistance of mortar to sulfuric acid increases.

Several studies reported enhanced retention of compressive strength in GBS-based mortar after exposure to sulfuric acid compared to fly ash-based mortar. However, fly ash-based mortar exposed to $MgSO_4$ solution for 10 years experienced less cracking and swelling compared to OPC-based mortar. The extensive damage in OPC-based mortar was attributed to development of ettringite and gypsum [8].

Solubility of acid attack reaction products is an important factor which determines the extent of damage in case these product leach out of the cement matrix [17,18]. It was reported that acetic acid is amongst the organic acids that develop products more soluble than sulfuric or other strong acids, when it comes in contact with mortar with alkali-activated precursor [17]. Calcium salts resulting from attack of mortar matrix by organic acid may precipitate on sample surface or internal surfaces of the pore system. However, even if salts precipitate on the exterior surfaces of mortar samples, they may or may not protect the matrix from further deterioration deeper into sample core, depending on the molar volume of calcium salts [18]. In general, alkali-activated mortar with GBS and fly ash binder demonstrated better resilience against damage due to various acids compared mortar with OPC binder, due to their lower calcium content [19]. Calcium in OPC-based concrete becomes available to form calcium salts after acid attack as it is abundant in ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}{\cdot}26H_2O$) and sodium hydroxide ($Ca(OH)_2$). Mortar/concrete with alkali-activated GBS/fly ash blended binders offer promising alternatives to OPC in terms of resistance to damage caused by acid exposure. As GBS geopolymerization products densify the pore system, they also enhance the resistance to sulfuric acid when its content is increased at the expense of fly ash in alkali-activated slag-fly ash binder systems. Fly ash on the other hand is essential to offset some of the drawbacks of GBS, such as the shorter setting time [16]. Fly ash enhanced 28-day strength of concrete significantly when used as replacement for up to 20% of OPC, and enhanced resistance to chloride penetration when used to replace up to 40% of OPC [20–23]. However, in alkali-activated mortar with GBS/fly ash binder, chloride ingress through samples was rated as moderate to high, when fly ash content was $\geq$20% of the total GBS-fly ash binder [24]. Alkali-activated mortar samples exhibited low diffusivity and higher chloride binding capacity when the GBS:fly ash ratio was 80:20 or higher.

Curing conditions affect strength development of mortar/concrete with alkali-activated GBS/fly ash binder. Mortar with alkali-activated GBS/fly ash binder cured under water at ambient temperature increased in compressive strength from the age of 3 days to 28 days for mixes with GBS contents ranging 50% to 90% of the total GBS/fly ash binder content [25]. The molarity of the activator solution also affects the rate of strength development, depending on fly ash content, when alkali-activated samples are cured under water [16]. Mortar prepared using 15 M NaOH solution with 50%GBS + 50%fly ash binder developed the highest 28-day compressive strength, compared to samples with higher GBS content, while mortar samples with GBS as sole binder dropped below the strength at the age of 14 days [25].

Strength development of mortar/concrete with OPC or alkali-activated binders are both influenced to varying degrees by curing temperature and relative humidity (RH) [26]. Strength development of mortar with alkali-activated binder is critically affected by the curing environment where samples are protected from moisture loss (closed curing system), or exposed to air (open curing system). Curing of alkali-activated mortar with blended GBS/fly ash binder in open environment was found to produce more porous void system, while curing in closed environment produced denser pore system and higher strength. The enhanced pore system in closed curing environment was attributed to the retardation or inhibition of leaching of oxyanionic metalloids [27].

This article evaluates the effect of immersing mortar with alkali-activated GBS and fly ash binder in 10% sulfuric acid solution on the development of compressive strength and stability of mass. Geopolymer mortar samples were immersed in sulfuric acid solution 24 h after casting, according to ASTM C267 – Method B [28]. There are two opposing factors influencing alkali-activated mortars immersed in sulfuric acid: 1) the detrimental effect of highly acidic solution on polymeric matrix, 2) the protective and closed curing environment provided by water in the acidic solution, which promotes dense pore system and improved compressive strength. The effect of immersion in sulfuric acid solution on development of compressive strength is compared to the response when samples are cured in open environment by direct exposure to air. The effect of the activator solution alkalinity on compressive strength development of geopolymer mortar immersed in sulfuric acid solution, or cured in air was studied for binders with slag content $\geq$ 50%. The relationship between splitting tensile strength and compressive strength at the age of 28-days of mortar samples cured in air is evaluated and mathematical models were developed.

2. Methodology and experimental program

The primary objective of this study is to assess the effect of immersing mortar samples with alkali-activated GBS + fly ash binder in sulfuric acid solutions for up to 90 days. The effect of sulfuric acid on mass and compressive strength of mortars is evaluated by calculating the change in weight and compressive strength at the ages of 7 days, 28

days, and 90 days after casting. Solutions were prepared using three types of binders including 50%GBS + 50% fly ash (G50F50), 75%GBS + 25% fly ash (G75F25), and 100%GBS (G100). The choice of 50% fly ash as upper limit was based on the vulnerability of fly ash-based mortar to carbonation and acid attack, as discussed in the introduction of this article. The precursors were activated using combinations of sodium hydroxide and sodium silicate solutions. The effect of activator solution alkalinity on sulfuric acid resistance of geopolymer mortar samples was assessed by varying the molarity of NaOH solution from a minimum of 10 M to maximum of 16 M, in increments of 2 M. In addition, identical mortar samples with the same three binder combinations and NaOH concentrations were air-cured (exposed), 24 h after casting. The compressive strength and splitting tensile strength tests were conducted after 3, 7, and 28 days of air-curing.

Fly ash used as precursor complies with ASTM C618 class N ($SiO_2 + Al_2O_3 + Fe_2O_3$) > 70%) as well as class F, with pozzolanic properties. The properties of ground granulated blast furnace slag and fly ash used in this study were determined using X-ray fluorescence spectrometry (XRF) and the results are shown in Table 1.

Precalculated amounts of NaOH flakes were mixed with potable water to prepare sodium hydroxide activator solutions with the desired four concentrations of 10 M, 12 M, 14 M, and 16 M. The solution was covered for two hours to cool down to room temperature. Precalculated quantities of sodium silicate solution (SiO_2 = 28.8 wt%, Na_2O = 9.8 wt %, and H_2O = 61.4 wt%.) were mixed with the NaOH solution to create activator solutions with three ratios of sodium silicate/sodium hydroxide (Na_2SiO_3/NaOH) including 1.5, 2, and 2.5. After the mixture of sodium silicate and sodium hydroxide solutions has cooled down to room temperature, a polycarboxylate superplasticizer in the amount of 2.5% of the total binder content was added to the activator solutions. The superplasticizer dosage was selected by trial and adjustment until acceptable flow without bleeding was achieved in all mixes [16]. The total number of activator mixes for each of the binders is 4 NaOH concentrations × 3 activator ratios = 12. Since there are three binders, the total of mixes is 3 × 12 = 36, which are detailed in Table 2. A liquid/binder ratio of 0.55 was selected through trial and adjustment to achieve acceptable flow without bleeding for all of the 36 mixes, along with a fixed total binder content of 436 g per mix [16]. The sand/binder ratio was fixed at 2.75 as per ASTM C109.

Preparation of mortar samples for each of the 36 mixes shown in Table 2 followed the same process. The dry material including sand and binder were preblended, then the activator + superplasticizer solution was poured into the dry mix. The dry blend and liquid were mixed for 5 min and were then poured into 50 mm cubic steel molds and vibrated to remove air bubbles. Studies have shown that adding liquid activator solution to dry blend of sand and binder results in mixes with better workability and mortar with enhanced mechanical properties [29].

All mortar samples were kept in molds for one day after casting, and then removed for conditioning and testing. Two identical sample sets were prepared for each of the 36 mixes. One set is immersed in 10% sulfuric acid solution until the test day, while a second set was air-cured in the laboratory at 22 ± 2 °C and 70% RH. The immersion of samples in sulfuric acid after 24 h in casting was consistent with ASTM C267-20 Method B [28]. This test offers valuable information on resistance of geopolymer concrete to sulfuric acid if exposure occurs shortly after casting. The results will determine whether water in acid solution will provide a protective curing environment that will produce a dense pore system which will mitigate the damaging effect of sulfuric acid. Change in weight and compressive strength was calculated after 7, 28, and 90-days of casting. It was discussed in the introduction section that protective (enclosed) curing of geopolymer mortars may enhance the pore system and compressive strength compared to open curing [27].

The set of samples that was cured in air (exposed) was tested after 1, 7, and 28-days of casting to evaluate the strength development. The correlation between splitting tensile strength and compressive strength for air-cured samples was studied, considering the effect of the relative contents of GBS and fly ash in the alkali-activated binder.

2.1. Experimental procedure – compressive and tensile strengths

The compressive strength of 50 mm cubic mortar samples immersed in sulfuric acid or cured in air was evaluated based on the procedure described in ASTM C109. Mortar samples were prepared so that each of the three binder compositions, G100, G75F25, and G50F50 was activated by 12 alkaline activator solutions representing 4 NaOH concentrations and 3 Na_2SiO_3/NaOH ratios for each NaOH concentration. The test dates for samples immersed in sulfuric acid were 7, 28, and 90-days, while air-cured samples were tested at the ages of 1, 7, and 28-days.

Sample preparation for the splitting tensile strength was based on ASTM C192 and the mechanical test was conducted based on the procedure described in ASTM C496. Splitting failure modes of all samples were typical to concrete specimens as shown in Fig. 1. Each tensile strength data point reported is the average of two tests conducted on standard 100 mm × 200 mm cylindrical samples.

2.2. Experimental procedure – change in weight of samples immersed in sulfuric acid

Resistance of mortar with alkali-activated GBS/fly ash precursor to degradation due to sulfuric acid attack was conducted according to ASTM C267-20 [28]. After 20 mm cubic mortar samples were cast as described in the previous section, they were kept in molds for 24 h, then removed and weighed. Mortar samples were then immersed in 10% sulfuric acid (H_2SO_4) solution until the test day (Fig. 2). On the 7th, 28th, and 90th day, samples were removed from immersion tanks, dried externally, and weighed. Eq. (1) was used to determine the difference in weight after immersion in sulfuric acid for the prescribed number of days. The samples taken for testing varied by binder composition, NaOH molarity, and activator ratio.

$$weightchange(\%) = [(W_{imm} - C_{initial})/C_{initial}] \times 100 \quad (1)$$

where:

$C_{initial}$ = weight of specimen 24 h after casting.
W_{imm} = weight of surface-dried specimens after immersion in sulfuric acid solution.

3. Results and discussion

3.1. Effect of immersion in sulfuric acid on weight of mortar samples

The effect of immersing mortar with alkali-activated GBS-fly ash binder in sulfuric acid may vary depending on the relative amounts of GBS and fly ash in the binder, as well the concentration of sodium hydroxide solution. In the present study, the molarity of H_2SO_4 solution was approximately 1 M, making the solution highly acidic. Fig. 3 shows cubic samples inspected for sulfuric acid damage. Limited or no visual

Table 1
Chemical composition of GBS and Fly Ash Precursors.

	CaO	SiO_2	Al_2O_3	SO_3	Fe_2O_3	TiO_2	K_2O	MnO	SrO	ZrO_2	CuO	Cr_2O_3	Y_2O_3
GBS (%)	59.44	25.68	8.12	2.75	1.499	1.048	0.609	0.562	0.125	0.065	0.031	0.026	0.016
Fly Ash (%)	4.294	58.158	24.351	0.068	8.517	2.565	1.534	0.094	0.062	0.085	0.037	0.047	0.015

Table 2
Composition of the 36 mixes including GBS and fly ash percentages, NaOH molarity, and ratio of sodium silicate/sodium hydroxide (SS/SH).

	G100 Mixes					G75F25 Mixes					G50F50 Mixes			
No	GBS (%)	Fly Ash (%)	NaOH molarity (M)	SS/SH	No	GBS (%)	Fly Ash (%)	NaOH molarity (M)	SS/SH	No	GBS (%)	Fly Ash (%)	NaOH molarity (M)	SS/SH
1	100	0	10	1.5	13	75	25	10	1.5	25	50	50	10	1.5
2	100	0	10	2	14	75	25	10	2	26	50	50	10	2
3	100	0	10	2.5	15	75	25	10	2.5	27	50	50	10	2.5
4	100	0	12	1.5	16	75	25	12	1.5	28	50	50	12	1.5
5	100	0	12	2	17	75	25	12	2	29	50	50	12	2
6	100	0	12	2.5	18	75	25	12	2.5	30	50	50	12	2.5
7	100	0	14	1.5	19	75	25	14	1.5	31	50	50	14	1.5
8	100	0	14	2	20	75	25	14	2	32	50	50	14	2
9	100	0	14	2.5	21	75	25	14	2.5	33	50	50	14	2.5
10	100	0	16	1.5	22	75	25	16	1.5	34	50	50	16	1.5
11	100	0	16	2	23	75	25	16	2	35	50	50	16	2
12	100	0	16	2.5	24	75	25	16	2.5	36	50	50	16	2.5

Fig. 1. Failure of 100 mm × 200 mm alkali-activated mortar samples during splitting tensile strength test.

signs of deterioration were present on the outer layers of the mortar samples, for all binder combinations evaluated in this study (G100, G75F25, and G50F50), after 90 days of immersion under the acidic solution. No evidence of scaling or softness of samples was noted either. Bakharev et al [30] tested mortar with alkali-activated slag as sole binder in acetic acid solution, and didn't observe signs of damage after 60 days. However, Valencia-Saavedra et al [31] noted superficial damage on mortar samples subject to 1 M sulfuric acid, but the binder consisted of 80% fly ash and 20% GBS. The same study also noted that despite the geopolymer mortar experiencing some damage under 1 M sulfuric acid, the damage was far more severe in OPC-based mortar samples [31]. Bakharev [9] noted severe damage on mortar samples prepared using OPC or OPC + fly ash, to a depth of 4 mm after one

Fig. 2. 20 mm cubic samples in 10% sulfuric acid solution.

month of immersion in acetic acid. The damage experienced by OPC-based mortars was attributed to the high calcium content. In OPC-based or fly-ash based mortar samples, the presence of gypsum deposits was evident within the tested samples after immersion in sulfuric acid [9]. In fly-ash based alkali-activated mortars, damage was attributed to dealumination and depolymerization. Allahverdi and Skavara [32] described the damage in mortar samples after the ejection of aluminum from the aluminosilicate framework as the result of shrinkage cracking leading to diffusion of sulfate anions into the cracks. The sulfate anions will then interact with the counter-diffusing calcium ions leading to formation and deposition of gypsum crystals.

Fig. 4 shows the change in sample weight after immersion of G100, G75F25, and G50F50 mortar samples in 10% sulfuric acid solution. Each data point in the figure is the average of the 4 NaOH concentrations which are 10 M, 12 M, 14 M, and 16 M. In general, change in sample weight at any particular activator ratio (1.5, 2.0, or 2.5) increases with age (7, 28, or 90 days), and this pattern is consistent for each of the three binder compositions (G100, G75F25, and G50F50). Therefore, for the binder combinations covered in this study, where maximum fly ash content is 50% or less, sulfate attack does not cause weight reduction. Instead, immersing the samples under sulfuric acid solution caused increase in weight, likely due to formation of polymerization products. Water in the sulfuric acid solution has probably acted as protective closed curing system that prevented mixing water from evaporating, decreased leaching, and supported the development of polymerization products [27].

Increase in sample weight with increase in activator ratio applies to

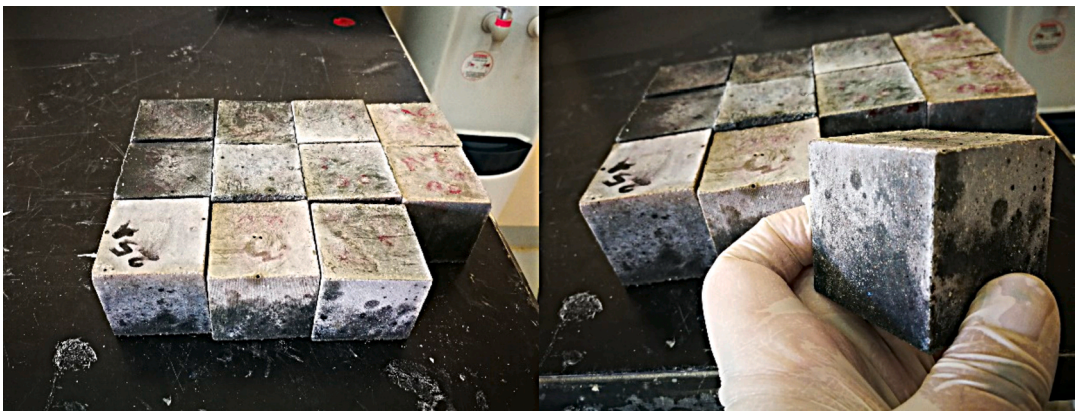

Fig. 3. 20 mm cubic samples visually inspected individually after removal from acidic solution.

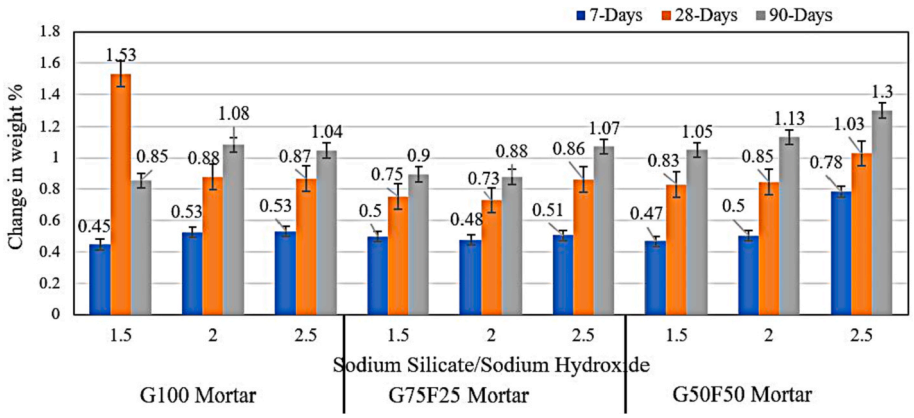

Fig. 4. Increase in sample weight after immersion in 10% sulfuric acid solution for 7, 28, or 90-days. Each data point is the average of the 4 NaOH concentrations included in the study.

all alkaline activator NaOH concentrations from 10 M to 16 M [16]. Similarly, sample weight increased with curing age from 7-days to 90 days, and the pattern is generally consistent for mortar samples prepared using G100, G75F25, and G50F50. The weight gain in G100 mortar at sodium silicate/sodium hydroxide ratio of 1.5 was significantly higher than the rest of the data in Fig. 4, which is likely due to solution instability or human error. The increase in weight consistent with the findings of Degirmenci [33] whose study lasted 180 days, but the concentration of NaOH was fixed at 10 M. The present study covers NaOH concentrations of 10 M, 12 M, 14 M, and 16 M and lasted for 90 days.

Valencia-Saavedra [31] documented for 80%fly + 20% GBS binder, that initial loss in mortar weight occurs due to exposure of samples to 10% sulfuric acid, followed by temporary regain of lost weight, then continuous loss of weight. Therefore, as indicated in the introduction section of this article, high content of fly ash (greater than 50%) in GBS-fly ash blended binder, or fly ash only, contributes to decrease in weight when alkali-activated mortars are subjected to high concentration sulfuric acid. When fly ash content is high, acid attack breaks Si-O-Al bonds of the aluminosilicate structure, and increases the number of Si-OH and Al-OH groups in the alkali-activated system. This produces a greater number of silicic acid ions and dimers that migrate to the solution, leading to mass loss [9]. However, in the present study, the continuous immersion in sulfuric acid solution provided a closed and protective environment, which is the water in the solution, that overcame any sulfuric acid damage by densifying the pore system. The effect of closed curing on pore system and compressive strength development was reported in the literature [16,27].

3.2. Compressive strength of mortar with alkali-activated GBS/fly ash after immersion in 10% sulfuric acid.

Samples were demolded 24 h after casting, then placed in 10% sulfuric acid solution (pH = 1.0) until the test day [28]. Visual inspection, determination of change in mass and compressive strength were done after 7, 28, and 90- days of immersion in the solution. Mortar samples tested varied by binder composition, NaOH molarity, and alkaline activator ratio. Since samples were immersed 24 h after casting, they under two competing influences: 1) the deleterious effect of highly acidic solution, and 2) the favorable closed and protective curing conditions (i.e., the presence of water in sulfuric acid solution). Curing in closed environment reportedly enhances the pore system and increases compressive strength [27].

Figs. 5a, 5b, and 5c represent the strength development of G100, G75F25, and G50F50 mortar samples after immersion in the acidic solution for the prescribed periods. Clearly sulfuric acid didn't induce a reduction in compressive strength at any particular curing age, compared to the previous test age. Compressive strength increased with age indicating reactivity of GBS and development of polymerization products are enhanced by the closed curing system formed by water in the sulfuric acid solution. This is consistent with data published by Collins and Sanjayan [34] where GBS-based mortar samples cured in water-path developed higher compressive strength compared to similar mortar samples exposed to air and samples wrapped with sealing material.

The relatively high compressive strength of G100 mortars, ranging from 36.24 MPa to 47.1 MPa, at the age of 7-days is due to the high reactivity of GBS, as shown in Table 3 and Fig. 5a. This is very close to

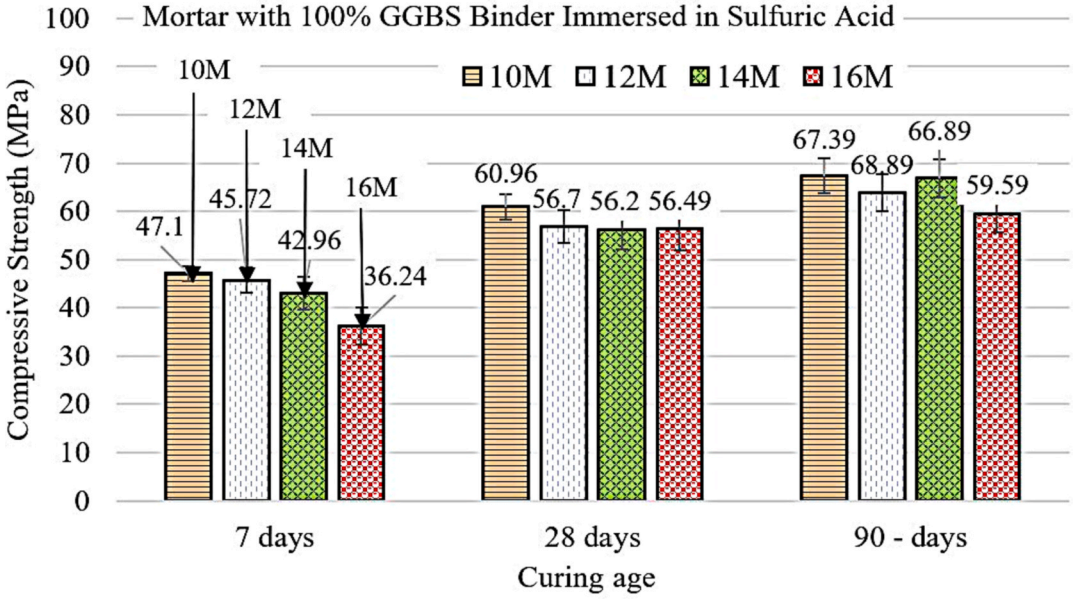

Fig. 5a. Strength development of G100 mortars immersed in10% sulfuric acid.

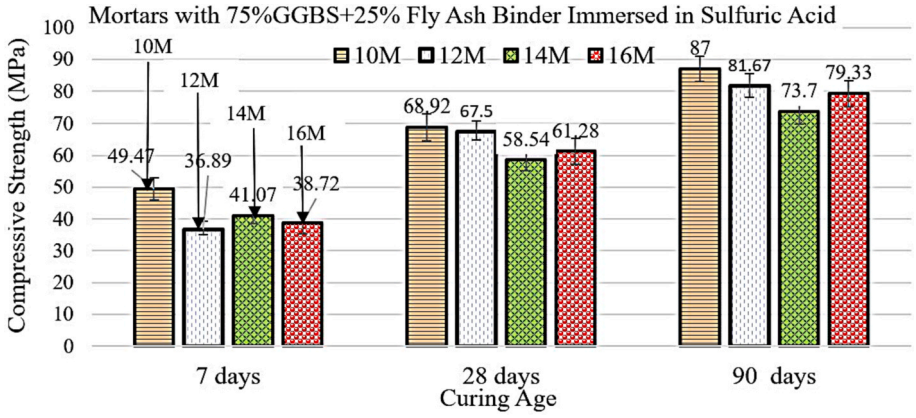

Fig. 5b. Strength development of G75F25 mortar after immersion in sulfuric acid solution.

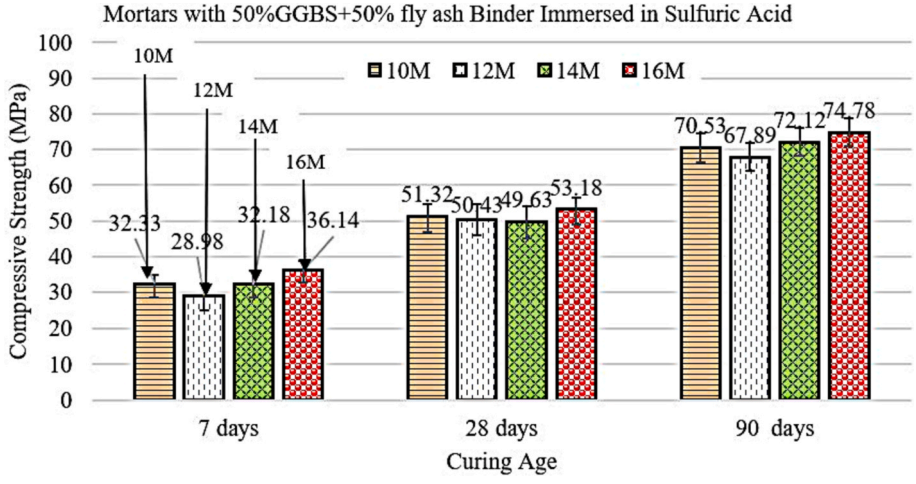

Fig. 5c. Strength development of G50F50 mortar immersed on sulfuric acid solution.

Table 3
Compressive strength development of G100, G75F25, and G50F50 samples immersed in sulfuric acid solution.

	7-day compressive strength (MPa)			28-day compressive strength (MPa)			90-day compressive strength (MPa)		
	G100	G75F25	G50F50	G100	G75F25	G50F50	G100	G75F25	G50F50
10 M	47.1	49.47	32.3	60.96	68.92	51.32	67.39	87	70.53
12 M	45.72	36.69	28.98	56.77	67.5	50.43	63.89	81.67	67.89
14 M	42.96	41.07	32.18	56.29	58.54	49.63	66.89	73.7	72.12
16 M	36.24	38.72	36.14	56.49	61.28	53.18	59.59	79.33	74.78

the range for G75F25 (36.69 MPa to 49.47 MPa) shown in Fig. 5b, but higher than G50F50 (28.98 MPa to 36.14 MPa) shown in Fig. 5c. This means, at the early age of 7 days, replacing 25% of GGBS with fly ash decreases the compressive strength, largely due to the slow reactivity of fly ash. The inherently slow reactivity of fly ash has historically impeded its use as sole alkali-activated binder in synthesis of geopolymer mortar/concrete [35]. At the ages of 28-days and 90 days, G75F25 exhibited higher compressive strength compared to G100 and G50F50 mortars, as shown in Table 3, regardless of NaOH molarity. GBS typically contributes to enhancing compactness of mortars with alkali-activated GBS/fly ash blended binder, but the ratio of GBS-to-fly ash of 3:1 appears to develop optimum strength. Table 3 also shows that at the age of 7-days, the higher the concentration of NaOH, the lower the strength of G100 mortars. In alkali-activated GBS-based mortars, increasing NaOH concentration increases compressive strength up to an optimum concentration, beyond which the strength decreases. Studies have shown that when excess sodium (Na) is present in the solution due to higher NaOH concentration, amorphous sodium-based products form within the C-A-S-H gel and are responsible for the decrease in strength that occurs when alkalinity of the activator solution increases [36].

The highest 28-day compressive strength of all binder combinations and NaOH concentrations occurred with NaOH concentrations of 10 M and 12 M, reaching values 68.92 MPa and 67.5 MPa, respectively. This confirms the ratio of GBS:fly ash of 3:1 as optimum for compressive strength development, and that high GBS content produces higher compressive strength at lower solution alkalinity (NaOH = 10 M or 12 M). The highest compressive strength at the age of 90-day was 87 MPa, developed by G75F25 mix prepared using 10 M NaOH. Clearly, alkali-activated G75F25 mortar is classified as high strength for structural engineering applications. The 90-day compressive strength for this mix ranges from 74 MPa (14 M NaOH) to 87 MPa (10 M NaOH).

Fig. 5a shows that G100 mortars exhibited significantly higher rate of strength development from 7 to 28-days compared to G75F25 and G50F50, but the rate slowed down from 28 days to 90 days. For example, G100 mortar with 16 M NaOH increased in strength by 55.88% from 7 days to 28 days, but increased by only 5.49% from 28 days to 90 days. The observation is consistent for all NaOH concentrations shown in Fig. 5a. Furthermore, at the age of 28 days, the highest compressive strength corresponds to the lowest NaOH of 10 M, which is consistent with the findings of Samantasinghar and Singh [37]. This is because the higher alkalinity of NaOH accelerates leaching of aluminosilicates from the precursor. When alkalis beyond the optimum concentration are present in the solution, they cause excessive leaching of silica, which delays the polymerization process by congealing particle surfaces. Congealing of particle surfaces inhibits further leaching of ions, and ultimately reduces strength [38].

Fig. 5b shows the strength development of G75F25 mortars immersed in sulfuric acid solution. Due to the high GBS content (75%), mixes with 10 M NaOH activator (the lowest solution alkalinity in the study) developed the highest compressive strength at the ages of 7, 28, and 90-days. G75F25 also developed higher compressive strength at each age, compared to G100 mortars (Fig. 5a), as shown in Table 3. For example, G75F25 mortar developed 68.92 MPa at the age of 28 days, compared to 60.96 MPa for G100 mortar. In addition, G75F25 mortar developed 87 MPa at the age of 90 days, compared to 68.89 MPa for G100 mortar. The rate of strength development from 7 days to 28 days, and from 28 days to 90 days for G75F25 mortar is higher than G100 mortar. For example, the minimum increase in compressive strength of G75F25 mortars from 28 days to 90 days was 21% (Fig. 5b), while the maximum increase in compressive strength of G100 mortar was 18.83%. This substantial increase in compressive strength fof G75F25 indicates that immersion in sulfuric acid solution at an early age (24 h after casting) provides a closed and protective medium through water in the solution, which supports strength development.

Therefore, replacing 25% of the GBS with fly ash was highly effective in enhacing the compressive strength, especially at the ages of 28 days and 90 days. The superior compressive strength development when the GBS:fly ash ratio is 3:1 is consistent with the data reported by Aiken et al [19].

Fig. 5c shows the strength development of G50F50 mortars immersed in sulfuric acid solution. The highest 7-day compressive strength of G50F50 is 36.14 MPa, smaller than or equal to the corresponding strengths of G75F25 and G100. Due to the high fly ash content in G50F50, the highest compressive strength at the age of 7 days occurred when the NaOH concentration was also the highest (16 M). Despite the relatively lower compressive strength at the age of 7 days for G50F50 compared to G100 and G75F25 (Table 3), the rate of strength development increased signficantly from 7 days to 28 days and from 28 days to 90 days. For example, when NaOH concentration was 10 M G50F50 developed 37.43% higher strength at the age of 90 days compared to 28 days, while G75F25 increased by 26.23%, and G100 increased by 10.55%, during the same period.

When alkali-activated mortars are exposed to strong acid, such as the 10% sulfuric acid, leaching was reported in the literature as a potential concern [39]. However, in the present study, there was no decrease in compressive strength with curing age. The enhanced resistance is due to the favorable closed environment provided as early as 24 h after casting. In addition, studies by Li and Peethamparan [39] indicated that alkali-activated mortars that use GBS as binder exhibited higher resistance to leaching compared to OPC- based mortar. In the present study, the minimum GBS content was greater than or equal to 50%.

3.3. Compressive strength of G100, G75F25, G50F50 mortar cured in air

It is of interest to observe the effect of ambient conditions, without special curing arrangements, on the strength development. It is also of interest to evaluate the effect of NaOH alkaline activator molarity on compressive strength development at ambient conditions. The alkaline activator solution preparation and mortar mixing procedures were described earlier. Mortar samples were demolded 24 h after casting and left in the laboratory subjected to air at temperature of 22 ± 2 °C and 70% RH, until compression or splitting tensile strength test day. Figs. 6a, 6b, and 6c show continuous increase in compressive strength with age for each of the three binder sets (G100, G75F25, and G50F50).

As shown in Fig. 6a, G100 samples developed a high 1-day compressive strength ranging from 28.7 to 31.3 MPa, but the effect of NaOH concentration was insignificant, as polymerization process was still in the early stages. At the age of 7 days, compressive strength of G100 mortar jumped rapidly to 42.75 MPa when the concentration of NaOH alkaline activator was 10 M NaOH activator mixes, to 52.3 MPa

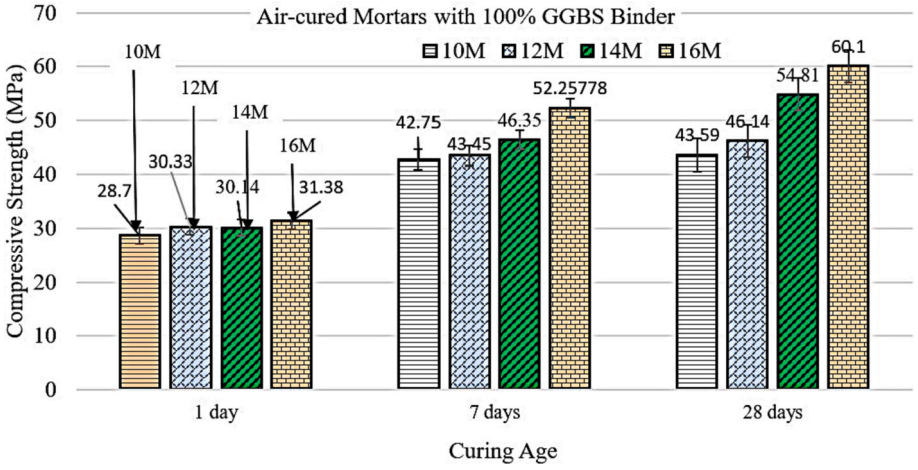

Fig. 6a. Compressive strength of air-cured G100 mortars.

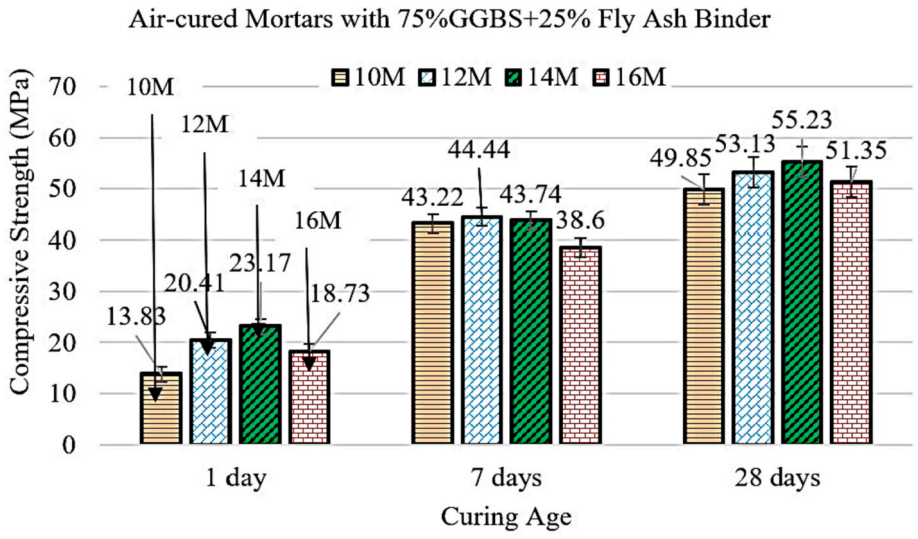

Fig. 6b. Compressive strength of G75F25 Mortar.

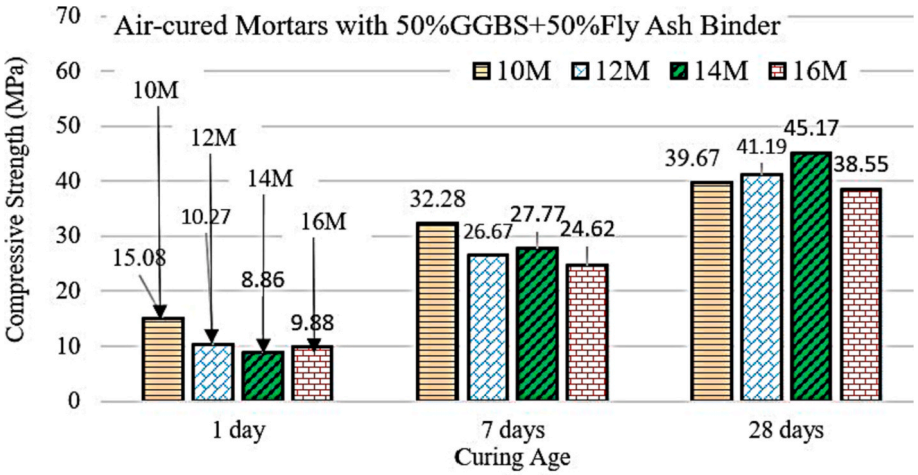

Fig. 6c. Compressive strength of G50F50 mortar samples.

when NaOH molarity was 16 M. This represents a significant increase in compressive strength of 43.26% to 66.54%. The rapid increase in compressive strength of G100 from 1 day to 7 days is due to the high reactivity of GBS and the availability of mixing water to sustain the reaction. However, the rate of strength development from 7 days to 28 days decreased significantly to the range of 1.96% to 18%, depending on

alkaline activator concentration. There are two possible and related reasons for the decrease: 1) curing in open system (exposed to air) permits loss of moisture from the exterior surfaces, and 2) the high GBS content may have caused initiation of shrinkage related microcracks [16]. However, the 28-day compressive strength of mortar with 16 M NaOH alkaline activator was 60.1 MPa, which is typically regarded as high strength for practical purposes.

Dissolution of fly ash during polymerization is much slower than GBS which is likely to affect the early strength development [12]. Therefore, Table 4 and Fig. 6b show that the G75F25 mix developed much lower 1-day compressive strength than G100, ranging 13.83 to 23.17 MPa, depending on concentration of NaOH alkaline activator solution. However, at the age of 7-days, the difference between compressive strength of G100 and G75F25 decreased significantly. Table 4 and Fig. 6b indicate that the percentile increase in compressive strength of G75F25 from 7 days to 28 days is 15.34% to 26.27%, depending on activator solution molarity. Comparing Fig. 6b to 6a, it is observed that the rate of strength development from 7 days to 28 days of G75F25 is higher than G100 mortars. Similar to the observations in the previous section of this article, replacing 25% of GBS with fly ash (G75F25) enhanced the rate of strength development. This may be due effective pore filling and densification of the matrix, or possible activation of fly ash by GBS in G75F25 at this optimum ratio of GBS to fly ash (3 to 1). It is important to consider that high GBS contents also offers enhanced chloride binding capacity, but causes substantial decrease in setting time [16].

Despite the slow start of the G75F25 mix category, the 28-day compressive strength of the mix was generally higher than G100 for NaOH concentrations of 10 M, 12 M, and 14 M NaOH, as shown in Table 4.

At the ages of 1, 7, and 28 days, compressive strength of G75F25 increased with the molarity of the alkaline activator NaOH up to 14 M. However, when the concentration of NaOH activator was 16 M, compressive strength of G75F25 mortar samples decreased at each of the three test ages, indicating 14 M is the optimum concentration. The existence of an optimum NaOH concentration is consistent with the findings of Samantasinghar and S. P. Singh [37]. During the polymerization process, the OH^- ions emanating from the sodium hydroxide activator solution serve as catalyst and stimulate dissolution of Si^{4+} and Al^{3+} ions from the aluminosilicate precursor(s). Furthermore, the Na^+ cation from the sodium hydroxide solution balances the charge deficit of the matrix [40]. At low alkaline concentration, leaching is slow and this weakens the polymeric structure and decreases the strength. Conversely, when alkalinity is high, the leaching is comparatively higher, leading to high compressive strength. Rattanasak [38] indicated that the presence of alkalis in greater amounts than the optimum concentration increases leaching of silica which congeals particle surfaces and slows down the polymerization process.

The slow reactivity of fly ash is more evident in G50F50 mortar samples than G75F25. The one-day compressive strength ranged from 8.86 MPa to 15.08 MPa, lower than the G100 and G75F25 mixes. The highest compressive strength of 15.08 MPa at the age of 1-day, shown in Fig. 6c corresponds to NaOH molarity of 10 M, the lowest of the four molarities evaluated in this study. Although geopolymerization reaction is slow at lower NaOH concentrations, higher NaOH concentrations may not increase compressive strength due to excessive leaching of silica, as described earlier [37]. Comparing Figs. 6b and 6c, it can be seen that for each molarity, the compressive strength of G50F50 at the age of 7 days and 28 days is lower than G75F25, due to the higher fly ash content in G50F50. However, the highest 28-day compressive strength of the G50F50 mix is 45.17 MPa, which is classified as high strength concrete. As shown in Fig. 6c, the increase in compressive strength of G50F50 from 7 days to 28 days ranged from 22.89% to 62.68%, depending on NaOH concentration. This is higher than the corresponding rate for G100 (Fig. 6a) and G75F25 (Fig. 6c). Therefore, increasing fly ash content from 0 to 50%, increased the rate of strength development of air-cured samples for all activator molarities from 10 M to 16 M. It is hypothesized that GBS with its fast reactivity serves as fly ash activator after one day of casting, accelerating the rate of strength development in GBS-fly ash blended binders.

3.4. Splitting tensile strength and its relationship to the 28-day compressive strength

The splitting tensile strength and compressive strength of air-cured mortar samples was determined experimentally, 28-days after casting. Mortar samples were kept in casting molds for 24 h, then removed and left exposed in the laboratory at 22 ± 2 °C and 70% RH. Splitting tensile strength and the corresponding 28-day compressive strength are shown in Table 5 for G100, G75F25, and G50F50 mortar samples. The difference in characteristics of GBS and fly ash reaction products motivates the need for developing the relationship between tensile and compressive strengths separately for samples prepared using each of the three binders individually, then seeking the relationship for G100, G75F25, and G50G50 combined. Eq. (2) is a prediction model for splitting tensile, f_{ct}, as a function of 28-day compressive strength f_c^n, that is often used in building codes and design standards [21].

$$f_{ct} = k f_c^n \tag{2}$$

where: k and n are data-dependent parameters obtained from regression analysis.

In order to assess the influence of GBS and fly ash on the relationship between tensile and compressive strengths of geopolymer mortar, regression analysis was conducted for the individual tensile strengths of G100, G75F25, and G50F50 mortar samples. The mathematical models investigated include Eq. (2) as well other relationships as discussed in following paragraphs.

Regression analysis using G100 data in Table 5 showed that several mathematical expressions demonstrated strong relationships between splitting tensile strength and the 28-day compressive of the mixes with 100%GBS binder. It is to be noted that this observation is subject to the parameters of this study, such as the liquid-to-binder ratio, range of NaOH concentrations, and sodium silicate/sodium hydroxide ratios. One of the successful models is the 2nd degree polynomial shown in Fig. 7a with high coefficient of correlation $R^2 = 0.971$. The standard model used by many building codes and represented by Eq. (2) provided slightly lower coefficient of correlation. The G100 data in Table 5 indicate that the splitting tensile strength ranges from a minimum of 8% to maximum of 9% of the corresponding 28-compressive strength.

Table 4
Compressive strength of samples cured in air for 1, 7, and 28 days.

	1-day compressive strength (MPa)			7-day compressive strength (MPa)			28-day compressive strength (MPa)		
	G100	G75F25	G50F50	G100	G75F25	G50F50	G100	G75F25	G50F50
10 M	28.7	13.83	15.08	42.75	43.22	32.28	43.59	49.85	39.67
12 M	30.33	20.1	10.27	43.45	44.44	26.66	46.14	53.13	41.2
14 M	30.14	23.17	8.86	46.35	43.74	27.77	54.81	55.23	45.17
16 M	31.38	18.17	9.88	52.26	38.61	24.62	60.1	51.35	38.55

Table 5
Splitting tensile strength for air-cured samples and corresponding 28-day compressive strength (SD = standard deviation).

	G100				G75F25				G50F50			
	Comp. Strength (MPa)	SD	Splitting Tensile Strength (MPa)	SD	Comp. Strength (MPa)	SD	Splitting Tensile Strength (MPa)	SD	Comp. Strength (MPa)	SD	Splitting Tensile Strength (MPa)	SD
10 M	44.98	1.95	3.75	0	44.45	2.014	3.075	0.32	41.65	2.05	3.45	0
	42.94	1.59	3.6	0	51.8	0.99	3.45	0	43	2	3.375	0.32
	42.85	0.72	3.6	0	53.3	2.91	3.75	0	34.35	2.1	3.3	0
12 M	40.27	2.85	3	0.42	50.3	2.74	3.825	0.11	40.6	1.5	3.525	0.11
	45.19	2.64	4.05	0	55.7	1.52	4.05	0	45.05	0.41	3.15	0
	52.95	4.47	4.5	0.11	53.4	1.92	4.2	0	37.93	5.92	3.075	0.11
14 M	51.68	3.92	4.5	0	44.3	0.96	3.6	0	47.3	3.18	3.3	0
	58.047	3.95	4.8	0	55.8	2.52	3.9	0	46.8	2.16	3.6	0
	54.69	1.72	4.5	0	65.6	2.19	4.65	0	41.25	3.06	3.45	0
16 M	60.67	1.71	4.95	0	48.3	1.59	3.6	0	40.8	2.35	3.45	0
	58.97	1.69	4.8	0	52.7	4.61	3.75	0	38.7	2.79	3.3	0
	60.68	0.9	4.8	0	53.05	3.1	3.975	0.11	36.15	1.63	3.15	0

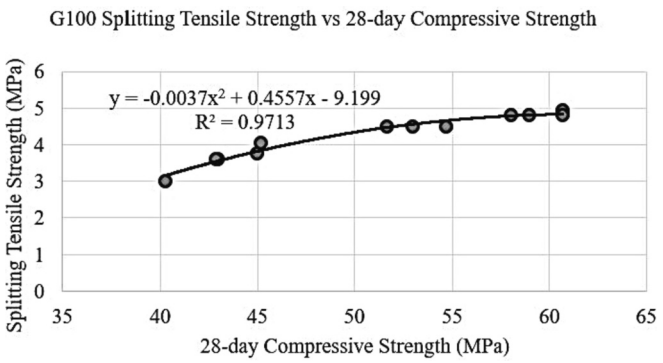

Fig. 7a. A 2nd degree polynomial shows a strong mathematical relationship between splitting tensile strength and compressive strength for mortars with 100%GBS binder.

The effect of fly ash binder on the relationship between splitting tensile strength and compressive is evaluated for G75F25 and G50F50 mortars. Based on the data in Table 5, the best fit model for the splitting tensile strength of G75F25 mixes according to Eq. (2) is given by $f_{ct} = kf_c^{0.84}$ with a coefficient of determination $R^2 = 0.75$. Several other mathematical models were evaluated, producing R^2 values less than or equal to 0.75. The best fit using a 2nd degree polynomial is shown in Fig. 7b, with $R^2 = 0.75$, far lower than coefficient of correlation obtained with 100% GBS mortars. The lower coefficient confirms the fact that polymeric products based on reaction of fly ash are structurally different from GBS.

G75F25 data in Table 5 indicate that for this mortar type the splitting tensile ranges from a minimum of 7% to a maximum of 8% of the corresponding 28-day compressive strength.

Fig. 7c shows a 2nd degree polynomial representing a splitting tensile strength prediction model for G50F50 mortars, with $R^2 = 0.181$. Clearly, increasing the fly ash content to 50% weakens the 2nd degree polynomial prediction model significantly as indicated by the R^2 value. Regression analysis was conducted using several models including, but not limited to power and exponential relationships with R^2 values far less than 1.0, similar to the 2nd degree polynomial. Table 5 shows that for mortar with 50% fly ash, the minimum splitting tensile strength was 7% and the maximum was 9.6% of the corresponding 28-day compressive strength.

The strong relationship between tensile and compressive strengths for mortars with 100%GBS and weaker correlation for mortars with fly ash indicate that polymerization products and pore system of GBS and fly ash are distinctly different. Further research is needed to understand the correlation between splitting tensile strength and compressive strength of mortar with alkali-activated GBS-fly ash binders.

Fig. 7d shows a 2nd degree polynomial model, developed based on regression analysis using all the splitting tensile strength data in Table 5, including G100, G75F25, and G50F50. The coefficient of corelation for this model is R^2 is 0.74, influenced as discusssed eariler by the fly ash content in G75F25 and G50F50.

The standard model given by Eq. (2) for all binder combinations and NaOH molarities from 10 M to 16 M, gives $f_{ct} = kf_c^{0.8}$ with $R^2 = 0.7204$. Clearly, the traditional model of Eqn. (2) doesn't provide a better fit than the 2nd degree polynomial.

Based on Table 5, it can be stated that the splitting tensile strengh of mortar with alkali-activated GBS/fly ash blended binder, where the minimum slag content is 50% of total binder, has a minimum value of 7% and maximum of 9.6% of the corresponding 28-day compressive strength. The results are consistent with published data on alkali-activated GBS-fly ash mortar [41]. Farhan et al [42] indicated that OPC-based mortars develop equivalent splitting tensile strength to fly ash-based and GBS-based alkali-activated mortars.

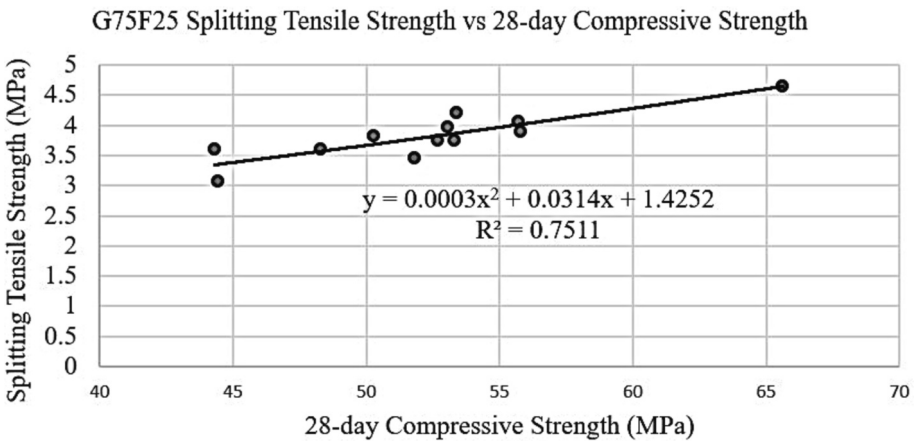

Fig. 7b. 2nd degree polynomial relationship between splitting tensile and compressive strength of G75F25 mortars.

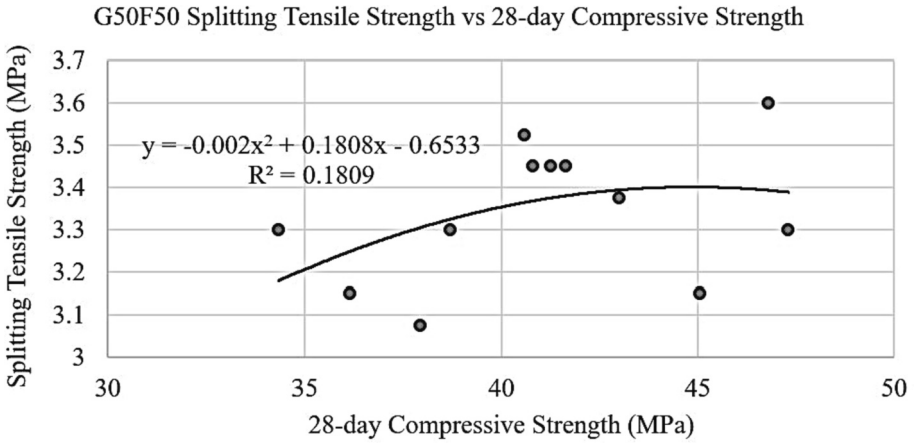

Fig. 7c. 2nd. degree polynomial relating tensile strength and compressive of G50F50 mortar.

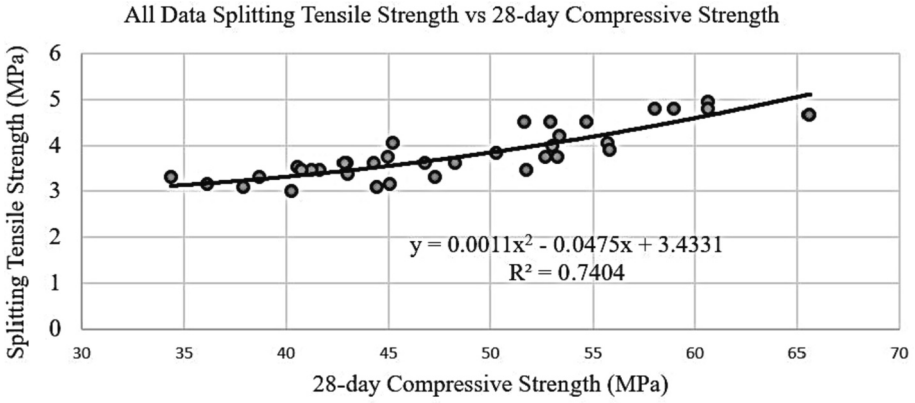

Fig. 7d. Relationship between splitting tensile strength and compressive strength for all binder types.

4. Conclusions

This study evaluated the effect of immersing mortar with alkali-activated ground granulated blast furnace slag (GBS) and fly ash blended binders in sulfuric acid solution (pH = 1.0), on compressive strength development and stability of mass. Samples were immesed in acidic solution 24 h after casting. Three binder compositions were considered including 50%GBS + 50%fly ash, 75%GBS + 25% fly ash, and 100% GBS. GBS and fly as where activated using alkaline solution consisting of a mixture of sodium hydroxide and sodium silicate. Strength development of samples cured in air was also assessed. Mathematical models relating splitting tensile and 28-day compressive strength of mortar samples cured in air-were proposed and validated. The main findings can be summarized as follows:

- Visual and manual inspection of mortar samples with binder consisting of alkali-activated GBS or GBS-fly ash combinations, did not detect any signs of damage or softening, after immersion in 10%

sulfuric acid solution for 90 days. Instead, geopolymer mortar samples gained mass and increased in compressive strength from one age to the other, regardless of binder composition or activator solution molarity. This is attributed to the existence of closed and protective environment provided by water in the sulfuric acid solution, which enhanced the development of geopolymerization products and densified the pore system. Water in the acidic solution also protected the samples from shrinkage cracking caused by the high GBS content ($\geq$ 50%), which sustained the increase in mass and strength. Published literature documented visible damage along with decrease in compressive strength of alkali-activated mortar/concrete in which fly ash content is high (60% to 100% of the binder). In this study, the maximum fly ash was $\leq$ 50% of the alkali-activated GBS-fly ash binder.

- Mortar with 50% fly ash and 50% GBS blended binder, that was immersed in sulfuric acid, developed the lowest compressive strength at the early age of 7 days, compared to mortar with 25% fly ash (G75F25) and mortar without fly ash (G100). However, the difference in compressive strength between the three binder compositions was very small at higher activator solution molarity (16 M). This is because the activation of fly ash is enhanced at higher solution alkalinity.
- Fly ash accelerated the rate of strength development of alkali-activated mortar immersed in sulfuric acid at later age, from 28 days to 90 days, regardless of sodium hydroxide concentration. The highest rate of increase in compressive strength from 28 days to 90 days occurred when fly ash content was 50% (the largest), followed by mortar samples with 25% fly ash. The slow reactivity of fly ash did ot inhibit, but delayed its contribution to compressive strength development. For the same reason, the highest rate of increase in compressive strength of air-cured mortar from 7 to 28 days, occurred in samples with 50% fly ash, followed by samples with 25% fly ash.
- Mortars samples with 75% GBS and 25% fly ash binders (G75F25), that were immersed in sulfuric acid solution exhibited the highest compressive strength at the ages of 28 days and 90 days, compared to G100 and G50F50. G75F25 developed 87 MPa when NaOH concentration was 10 M, which is the highest compressive strength in all of the 36 mixes. It appears that GBS:fly ash ratio of 3:1 represents the optimum value for the development of polymerization products that are denser and more compact, compared to other binder compositions. The pattern is also true for air-cured mortar samples, but at only at NaOH molarities of 10 M, 12 M, and 14 M. At NaOH molarity of 16 M, the highest strength was developed by G100, followed by G75F25, then G50F50. This may be due the dissolution of silica in fly ash (G75F25 and G50F50) at the higher molarity of 16 M, causing a congealing effect on particles and impeding further reaction.
- Splitting tensile strength of alkali-activated mortar with GBS binder (G100) exhibted a strong correlation ($R^2 = 0.97$) with the 28-day compressive strength through 2nd degree polynomial, as well as other mathematical relationships. The coefficient of correlation decreases signficantly ($R^2 = 0.75$) when 25% of GBS is replaced with fly ash, and decreases even further when 50% of GBS is replaced with fly ash, regardless of the mathematical mode. The standard square-root relationship, typical in many international codes does not offer the strongest correlation between splitting tensile strength and compressive strength, regardless of binder composition. The minimum splitting tensile strength was 7% of the corresponding 28-day compressive strength, while the maximum tensile strength was 9.6% of the 28-day strength for air-cured mortar samples with fly ash conent ranging from 0% to 50% of the GBS-fly ash binder combinations.

CRediT authorship contribution statement

Osama A. Mohamed: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Writing – original draft, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The author gratefully acknowledges the financial support of Abu Dhabi University, Office of Research and Sponsored Programs (ORSP), Grants No 19300643 and 19300521. The authors also gratefully acknowledge the financial support through ASPIRE Award for Research Excellence under the Advanced Technology Research Council – ASPIRE.

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