

Effect of dry mix alkali activated slag binder composite properties cured in ambient condition

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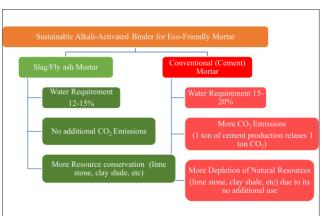
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Graphical abstract



Abstract

A dry mix (solution-free) alkali-activated slag (a primary cementitious material) and/or fly ash based binder composition is developed that can be cast in-site and consequently cured at ambient temperature. Mortar specimens were cast by mixing slag and or fly ash, river sand, powder form alkaline activators (NaOH 14M, varying solids percentage of Na₂SiO₃) and water were thoroughly mixed in fabricated equipment. The dry density (28 days) of all specimens showed greater than 2200 kg/m³. The compressive strength (28 days) of all mixes was resulted in higher than 40 N/mm². Among all the four mixes, a mix F103 with 90% slag, 10% fly ash, 30% Na_2SiO_3 and 14M NaOH at 3, 7 and 28 days curing showed overall higher compressive strength. It is due to fewer solids content of Na₂SiO₃. The experimental results indicated that solution free studied binder composite can be developed under ambient conditions eliminating other curing types without compromising in the strength. The studied sustainable mixes require only 12-15 percent water which is less compared to regular used mixes. Thus reduced water

quantities can be achieved thereby protects the reduction in volume of water bodies, environment hazards, reduces CO_2 emissions due to use of industrial by-products as main binders.

Keywords: Alkali activated; alkali activated binder composite; alkali activated slag; ambient curing; sustainable; industrial by-products

1. Introduction

Concrete placed second only to water as the most widely utilized construction material on the planet. A normal concrete mix contains around 10-15% cement, 60-75% aggregates (including coarse and fine aggregates), and 15-20% water (Steven and Michelle 2011). Cement mortar, on the other hand, is made from cement, sand, and water and is used for various applications, such as wall and slab plastering, bedding material to bond bricks and stones, and crack treatment. Cement mortar performs similarly to concrete in terms of effectiveness, compatibility, and durability. Due to rapid urbanization and industrialization demand for cement, as well as its natural ingredients such as limestone, clay, silica stone, water, etc. has increased. This led to over-exploitation of its ingredients, on the other hand, manufacturing and utilization of cement in construction activities led to the emission of greenhouse gases mainly carbon dioxide (CO₂). As per the report of Oss, 1 ton of cement releases 0.9 ton of CO₂ on hydration and causing an environmental hazard. Hence, it has given rise to sustainability issues and become imperative to look for alternatives to cement. Supplementary Cementitious Materials (SCM's) such as Ground Granulated Blast Furnace Slag (GGBFS or Slag), fly ash blended with cement mitigates the reduction of CO₂ (Oss 2005)⁻ Cement blended with 30-70% by mass of ground granulated slag reduces overall CO₂ emissions of about 90-390 kg per ton of cement. Fly ash, 15-35% by mass blended with cement

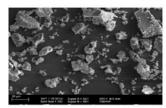
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resulted in decreased emissions of CO₂ by 45-127 kg per ton of cement (Sector Policies and Programs Division et al. 2010). Alkali solution-based geopolymer cement concrete was developed using metakaolin, the use of this composite in major projects raises the project's cost (Davidovits 1994). The methods and ingredients of geopolymer concrete manufactured with expensive materials like meta-kaolinite, amorphous silica, and/or diatomaceous earth, might result in increased project costs (Barsoum and Aaron 2008). The alkalinity and setting temperature affected the mechanical strength of solution mixed geopolymer compositions. The specimens were heat cured to 90°C to obtain compressive strengths of 65 to 70.9 N/mm², making the technique unsuitable for insitu construction (Davidovits et al. 2008). The solution mixed fly ash geopolymer concrete specimens are heated to around 50°C to obtain high compressive strength. It was also mentioned that when the temperature rises, compressive strength rises as well (Young et al. 2009). On the other hand, heat curing has the drawback of not being suitable for large-scale in situ construction. The samples were made of paste obtained by mixing 40-100% slag, 40-60% pumice, 0.1-25% hydrated lime by weight of cementitious component and water. The samples made with a composition of 50% slag, 50% pumice and 10% lime cured in the water bath for 24 hrs. at a temperature of 42.2°C (180°F) attained a higher compressive strength of 2.9 N/mm² compared to other mixes. However, with a mandatory water bath curing, in situ construction activities can be challenging to implement (Chatterji et al. 2017). Solution mixed geopolymer mortar or concrete is time-consuming and causes a delay in project schedules (Varma and Qayyumi 2011). The mechanical properties of various types of geopolymer fly ash concrete by mixing with alkali solutions are primarily influenced by the fly ash source and its properties (Erez and Loya 2016). The process for making alkali-activated foamed concrete without the need for additional foaming agents was identified (Dennis and Malone 1997). The melamine water with borate can be used for producing alkali-activated binder without cement. Since the specimens are cured by melting and synthesizing process which makes it not viable for in situ construction activities (Yang and Kyu 2009). The cement substituted with grounded blast furnace slag up to 70% showed optimal compressive and tensile strength. The increase in the GGBFS/Slag content showed an increase in the compressive strength (Hawileh et al. 2017). Using GGBFS/Slag in concrete reduces unit production, increases workability and rheological characteristics decreases chloride ion penetration, and enhances abrasion resistance (Erdog et al., 2016; Rafeet et al., 2017). Ambient cured geopolymer concrete and mortar specimens containing 70% fly ash and 30% slag had compressive strengths of 55 N/mm² to 63 N/mm², respectively (Nath and Sarker, 2014). Fly ash and cement mixed geopolymer concrete specimens with used foundry sand up to 60% replacement of river sand (ambient cured) had compressive and splitting tensile strengths of 43% and 20% greater, respectively than control mix geopolymer (Bhardwaj and Kumar 2018). In geopolymer

binder composites, polymerization plays a substantial role in imparting the strength, whereas, specimens of cementbased binder composites are cured in water for hydration reaction, which then gives them strength. CO₂ emissions will occur as a result of the hydration process, which must be reduced. Cement is proposed to (OPC-Ordinary Portland Cement) replace fully with SCM's such as GGBFS, a by-product of the steel manufacturing industry (Mendoza et al. 2015; Yuksel 2018) and fly ash, a byproduct of thermal power plants (Snezana and Jelena 2018). The effect of plant fibres on Alkali Activated Slag (AAS) was studied and reported that AAS with alkali treated wheat straw fibre of 1% volume showed optimum flexural and compressive strengths compared to increased fibre content from 1 to 5%. The increased strength at 1% fibre content was due to filling the inner cracks and at higher dosages due to agglomeration of fibres decreased strengths was noticed (Zhu et al. 2023). The bond strength of newly formed Alkali Activated Concrete (AAC) consisting 50% slag and 50% fly ash mix with older AAC concrete is maximum compared to high slag and low fly ash or vicewarsa, wherein a decline in bond strength was observed by the authors. The maximum strength was due to the mutual transfer of hydration products at the interface of old and new AAC (Zhang et al. 2022). The AAS activated with Sodium Carbonate (Na₂CO₃) and Calcium hydroxide (Ca(OH)₂) exhibited 60% greater compressive strength due its dense mix compared to NaOH activated AAS (Zhou et al. 2021). The chloride binding capacity of AAS activated with only water glass was much higher than only NaOH activated and hence reduces the risk of corrosion in former (Zhu et al. 2022). In AAS mortar, when 20% slag was replaced with cement recorded higher compressive strength and reduced 2 years drying shrinkage and loss of weight (Wang et al. 2023).

There exists a dearth of comprehensive research dedicated to dry mixing with ambient curing on alkali activated slag and fly ash binder composite. In research community this type of study otherwise named as onepart alkali activation. However, this study is aimed to address the research questions such as firstly, dry mixing (solution free) of AAS/Alkali Activated Slag and or Fly ash (AASF) composite. Secondly, degree of quality control of developed composite mixes on par with conventional mixes as per standards (ACI 363.2R-98 1998). Thirdly, strength of developed mixes cured in ambient conditions. Based on the research questions, this study is aimed to develop a solution-free alkali-activated slag and/or fly ash based binder composition that can be cast in-site, avoiding solution mix and consequently cured at ambient temperature thereby water and heat curing are eliminated. The composite mix is prepared with varying percentages of slag and or fly ash as cementitious ingredients, river sand, powder form alkaline activators (SS and SH) and water are mixed in fabricated equipment to ensure thorough mixing. Then the mortar mixture is placed in moulds and left to ambient curing to gain strength thereof. The cured (hardened) specimens were measured for dry density and compressive strength, as in majority situations mortar is subjected to compression,

hence it's natural to test for its compressive strength. The studied mixes may require less water compared to normal mixes. This reduction in water use during the production of mortar composites not only conserves water resources but also helps mitigate environmental hazards.



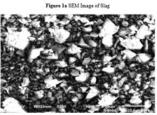
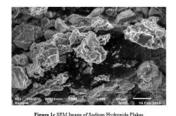


Figure 1b SEM Image of Fly a



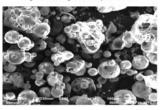


Figure 1d SEM Image of Sodium Silicate Powde

Figure 1. Scanning Electron Microscopic Images

2. Materials and properties

2.1. Ground Granulated Blast Furnace Slag / GGBFS / Slag

The slag used as one of the primary sources of aluminosilicate material is from Vizag Steel Plant (VSP), Visakhapatnam, India conforming to IS 455, IS 16714 and IS 10289 (Indian Standard 10289, 1987; Indian Standard 16714, 2018; Indian Standard 455, 1989) Figure 1 shows the microscopic image wherein the particles are angular due to extreme milling, and the mechanical and chemical properties are given in Table 1 and 2 respectively.

Table 1 Mechanical Properties of Cementitious Materials



Figure 2a Mixing Bowl



Figure 2b Mixing Blade



Figure 2c Heavy Duty Drilling Machine

Figure 2. Fabricated Equipment

2.2. Fly ash

The fly ash was used as an optional secondary source of aluminosilicate material is from a thermal power plant, Visakhapatnam, India conforming to IS 3812 (Indian Standard 3812 Part-1, 2013). The microscopic image is shown in Figure 1 indicating round shape. The chemical properties of fly ash are given in Table 2.

Tests conducted	Slag
Normal consistency	32%
Soundness	4 mm
Fineness modulus	0.14
Specific gravity	2.32

2.3. Sand

It was conforming to IS 2720 (Indian Standard 2720 Part-3/Sec-2, 1980) and IS 2386 (Indian Standard 2386, 1963, Part-1; Indian Standard 2386, 1963, Part-3) with 2.54 Specific gravity and 2.27 mean grains size.

2.4. Alkaline Activators

The alkaline activators, in powder form and are prepared from the combination of Sodium Silicate (SS or Na₂SiO₃) and flakes form Sodium Hydroxide (SH or NaOH) and which is considered 40% of the total binder composite. The ratio of SS to SH is adopted a fixed value as 2.5 (Nath 2014), a mass of solids in 14M SH is adopted as 33.53 kg/m³ and varying percentages of solids in SS that is 10%, **Table 2** Chemical Properties of Slag and Fly ash 30% and 50% with the mass of water varying according to the percentage of the mass of SS solids are adopted. The microscopic images of SS and SH are shown in Figure 1. From the SEM image of SH, the particles are flaky and in agglomerate form whereas SS particles are round in shape.

Parameter	Percentage (%)					
	Slag	IS 12089 (1987)	Fly ash	IS 3812 (Part 1)- (2013)		
Si O ₂	36.23	-	1.49	35 (Min)		
Al ₂ O ₃	18.91	-	35.82	-		
Fe ₂ O ₃	-	-	6.72	-		
Ca O	38.64	-	0.94	-		
Mg O	7.32	17 (Max)	0.71	5 (Max)		
K ₂ O	-	-	1.11	-		
SO ₃	-	3.5 (Max)	-	3 (Max)		
Na ₂ O	-	-	0.07	1.5 (Max)		
Fe O	1.65	-	-	-		
Mn O	0.58	5.5 (Max)		-		
Ti O ₂	-	-	1.86	-		
P ₂ O ₅	-	-	0.42	-		
Loss on Ignition (LoI)	-	4 (Max)	1.02	5 (Max)		
Glass Content	-	85 (Max)	-	-		
Moisture Content	18.56	-	-	2 (Max)		

3. Experimental Program

3.1. Mixing Methodology

The mixing of studied binder composite is done in fabricated equipment, consists of three components as shown in Figure 2.

- a) Mixing Bowl
- b) Mixing Blade
- c) Heavy Duty Drilling Machine

The mixer blade is attached to the drilling machine, and the dry components are poured into the bowl so that the blade is fully submerged. To avoid spilling while mixing, the material should not fill more than half of the bowl.

The following is the mixing process used to combine the components to produce a 1:3 solution free alkali activated slag based binder composite and same is also shown in Figures 3 and 4 (Nath 2014).

- At least one part of any of the SCM's such as slag and/or fly ash, must be completely mixed dry by being turned over and over, backwards and forwards, many times until the mixture is of a uniform colour for about 3 min as shown in Figure 4a.
- 2. For 2 minutes, completely combine solids of SH powder of desired molarity with solids of SS powder of required quantity as shown in Figure 4c.
- Mix the dry ingredients (slag or fly ash and sand) well for 2 minutes with solids of SH powder and SS powder respectively as shown in Figure 4d.

- Mix the dry components (step 3) well for another 4 minutes after adding the appropriate amount of water.
- This indicates dry ingredients (step 3) and water (step 4) mixed separately forms a solution free binder.
- Ensure a homogenous mix; if not, add any more water needed depending on the water-to-alkali activated binder ratio and thoroughly mix for 3–4 minutes and later place in moulds and kept in curing till testing as shown in Figure 4f and 4g.

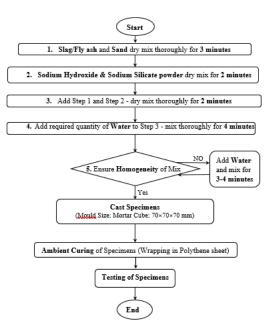


Figure 3. Dry Mix Design Flowchart



Figure 4. Mortar Mix Procedure Steps Follows Arrow Mark

3.2. Difference between Solution mix binder and solution free (studied mix) binder

In alkali activated and geopolymer mortar or concrete, the solutions of SH and SS were stirred together for around 30 minutes before being combined with the other dry binder components. This increases the reactivity of combined solutions and imparts strength to the binder (Nath 2014). Whereas in the studied mix, the dry components (slag and/or fly ash, sand and powdered form solids of SH and SS) are mixed as per the mentioned procedure. As SH is alkaline in nature reacts with powdered form solids of SS and thus reacts by liberating heat without the presence **Table 3** Details of Mix Proportion and Ingredients of all in Mixes

water. Later water is added to the dry components for further geopolymerization which imparts strength to the studied binder composite mix. The studied mixes required 12-15 percentage of water which is less compared to regular conventional mixes as shown in Table 3. Thus reduction in water quantity used in manufacturing of mortar composite can be achieved thereby protects the reduction in volume of water bodies, environment hazards. One of the author's stated that "if the limitations of one-part method is overcomed, it holds significant potential for mass scale production and as a packaged material it may be distributed" (Lanjewar *et al.* 2023).

3.3. Specimen preparation

To prepare a binder composite of F01mix (100% slag, 0% Fly ash, 100% sand, alkaline activator with 10% solids of SS and water). The F01 mix constituent compositions, as indicated in Table 3, are completely mixed according to the mixing technique, and then the fresh mix is kept in three layers in a mortar mould (70 mm each side), each layer being compressed with a 16 mm diameter tamping rod. Thereafter, moulded samples were vibrated for 2 minutes on a table vibrator to ensure adequate mortar compaction. After compaction, the top surface was levelled by using the trowel then fresh mix specimen moulds for 24 hours were covered with a polythene sheet and stored at room temperature (to allow setting and hardening). The cubes were demoulded once the mortar has hardened and left to cure at room temperature until the day of testing, as shown in Figure 4. A similar procedure was adopted for the rest of the mixes that is F103, F105 and F201 (Srinivasula et al. 2018). For all the mixes, the parameters such as binder composite ratio kept as 1:3, alkali to binder ratio as 0.4, SS to SH ratio as 2.5 and NaOH of 14M are kept constant. In the similar process per mix 3 specimens were cast for each curing period which accounts to total 36 specimens for all mixes.

Mix ID	Sodium Silicate			Ingred	ients (kg/m³)		
	Solids	Slag	Fly ash	Sand	Sodium Hydroxide Solids	Sodium Silicate Solids	Water
	(%)	(G)	(F)	(S)	(SH)	(SS)	(W)
F01	10	733.48	-	1173.128	33.506	20.9566	238.905
F103	30	660.132	73.348	1173.128	33.506	62.868	196.992
F105	50	660.132	73.348	1173.128	33.506	104.789	138.314
F201	10	586.784	146.696	1173.128	33.506	20.9566	238.905

Note:

F01 – Indicates Slag (G) 100% by weight, Fly ash (F) 0% by weight, Sodium Silicate Solids 10% by weight.

F103 – Indicates Slag (G) 90% by weight, Fly ash (F) 10% by weight, Sodium Silicate Solids 30% by weight.

F105 – Indicates Slag (G) 90% by weight, Fly ash (F) 10% by weight, Sodium Silicate Solids 50% by weight.

F201 – Indicates Slag (G) 80% by weight, Fly ash (F) 20% by weight, Sodium Silicate Solids 10% by weight.

4. Results and discussion

4.1. Dry density

Figure 5 shows the dry densities of the 3, 7, and 28-day cured mortar specimens. It was evident from the figure that all of the mixes, at all stages of curing, exceeded the required dry density of 2200 kg/m³. However, at 3 and 28

days, F105 mix had a dry density of more than 2400 kg/m³, which was 11.42% and 9.56% greater than normal dry density, respectively. It is due to addition of fly ash and higher solids percentage of Na₂SiO₃. The dry density for all composite mixes varies from 2216.57 kg/m³ to 2458.39 kg/m³ which is in acceptable limits of ordinary Portland cement recommended in ACI 318 (ACI 318-11,

2014). In comparison to standard dry density, all types of mixtures showed improved dry density values at all ages of curing.

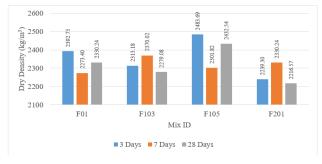


Figure 5. Dry Density of all in Mixes

4.2. Compressive Strength

As shown in Figure 6, the compressive strength of all mixtures increased consistently throughout the curing process. Interestingly, the compressive strength of all in mixtures at 28 days was greater than 40 N/mm². Whereas mix F103 (which contains 90% slag, 10% fly ash, and 30% Na₂SiO₃) had a greater compressive strength of 54.33 N/mm², which was 15.21%, 17.73%, and 19.90% higher than the other mixes. Even though, F103 and F105 contain 10% fly ash but a higher percentage of SS in F105 that is 50% Na₂SiO₃ caused a reduction in strength. In other author studies it is concluded that a compressive strength of 54 N/mm² is achieved for slag and fly ash mix (Ghafoor *et al.* 2021). It was concluded that ambient cured solution free alkali activated binder mortar composition were able **Table 4** Compressive Strength ± Standard Deviation of all in Mixes

to produce successfully, whose strengths are higher than standard compressive strength.

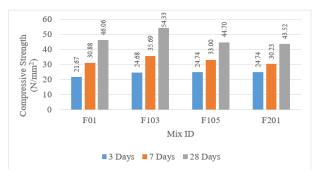


Figure 6. Compressive Strength of all in Mixes

The average compressive strength and its standard deviation of all in mixes at all ages of curing are as shown in Table 4. From the table, it was observed that for all in mixes at all ages of curing with the addition of fly ash and increment in solids percentage of Na₂SiO₃, the standard deviation decreased supporting the studies conducted (Wardhono *et al.* 2015). It indicates that quality control of all mixes were good and due to same with in test and overall strength variation results is low (ACI 363.2R-98, 1998). It was noticed that mixes F1O3 and F1O5 the initial 3 days and 7 days compressive strength variation is low. Strength at 28 days for F1O3 is 17.7% higher than F1O5, it is due to less solids quantity of Na₂SiO₃ and the same mix recorded overall higher strengths compared to remaining mixes at all ages of curing.

Mix Id	Measured Compressive Strength ± Standard Deviation (N/mm ²)				
	3 days	7 days	28 days		
F01	21.66 ± 0.84	30.88 ± 0.79	46.06 ± 0.77		
F103	24.68 ± 0.51	35.69 ± 0.45	54.33 ± 0.27		
F105	24.73 ± 0.24	33.00 ± 0.2	44.7 ± 0.2		
F201	24.73 ± 0.24	30.23 ± 0.13	43.52 ± 0.13		

4.3. Influence of Sodium Silicate solids on compressive strength

The influence of amount of SS was investigated with the mixes F103 and F105 (10% fly ash and 90% slag) with 30% and 50% Na₂SiO₃ respectively and F201 (20% fly ash and 80% slag) with 10% Na₂SiO₃. Figure 6 also shows the variation of compressive strength with the variation of Na₂SiO₃. For mixes F103 and F105, at 7 and 28 days of ambient curing, the compressive strength gradually decreased with the increase in the Na₂SiO₃ (30% and 50%). It was observed that, a marginal increase of 3 days strength is observed for mix F105 compared to F103 which is considered negligible. The increase of SS quantity caused increase in their solids quantity and that caused reduction in the water quantity and finally led to decreased compressive strength.

4.4. Relation between Dry Density and Compressive Strength

Figure 7 describes the relationship between dry density and compressive strength at 28 days. Even though the compressive strength of F103 mix was higher than all other mixes, the dry density was lower than F01 and F105 and substantially higher than the F201 mix, as seen in the figure. The higher strength of F103 was may be due to overall optimum solids percentage of SS and SH with considerable quantity of water for further geopolymerization imparting strength. Even though mix F105 achieved higher dry density due to higher quantity of solids with less quantity of water that lead to reduced reactions of SS and SH and caused low strength.

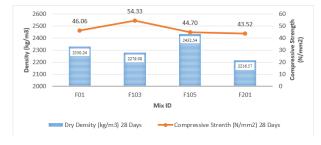


Figure 7. Relation between Dry Density and Compressive Strength

5. Conclusions

Based on the present study, the following conclusions are drawn

- At all ages of curing, the dry density of all types of binder composition mixtures was higher than the specified dry density (2200 kg/m³).
- The dry density of the F105 mix after 28 days was shown greater value of 2432.54 kg/m³ due to addition fly ash and higher percentage of solids of Na₂SiO₃.
- At 3, 7 and 28 days of curing, the compressive strength of all types of mixes increased consistently, and the compressive strength of all mixes at 28 days was greater than 40 N/mm².
- The 28 days compressive strength of binder composite mix F103 (90% Slag, 10% Fly ash, 100% Sand, 30% Na₂SiO₃ and 14M NaOH) was 15.21%, 17.73% and 19.90% respectively higher than the rest of the mixes (F01, F105 and F201). It is due to less percentage of Sodium Silicate solids and high water content.
- The 28 days compressive strength of binder composite F105 is lesser than F103, it is due to the high percentage of Sodium Silicate that caused a reduction in strength.
- The standard deviation of all in mixes at all ages of curing decreased due to addition of fly ash and increment in solids percentage of Na₂SiO₃. It indicates that quality control of all mixes were good and due to same with in test and overall strength variation results is low.
- The 28 days compressive strength of ambient cured solution free slag and or fly ash mixed binder composite with constant NaOH and varying Na₂SiO₃ at 10, 30 and 50% showed higher than 40 N/mm².
- The mix design of dry mix free alkali-activated slag/fly ash binder composite was developed successfully with 28 days compressive strength of above 40 N/mm² under ambient curing conditions.
- Thus studied mix is a sustainable mix due to use of less water quantity, reduced CO₂ emissions, reduction in depletion of lime, clay, shale, etc due to use of slag/fly ash as binder composites.

Data Availability

The data used to support the findings of this study are included in the article.

Conflicts of Interest

The authors declare that there is no conflict of interest.

Ethics Approval

All experimental procedures of the proposed research work were in accordance with the ethical standards.

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