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SELF-Cured Alkali Activated Slag Concrete Mixes-An Experimental Study

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***Abstract*—**Alkali Activated Slag Concrete (AASC) mixes are manufactured by activating ground granulated blast furnace slag (GGBFS) using sodium hydroxide and sodium silicate solutions. The aim of the present experimental research was to investigate the effect of increasing the dosages of sodium oxide (Na_2O, in the range of 4 to 8%) and the activator modulus (Ms) (i.e. the SiO_2/Na_2O ratio, in the range of 0.5 to 1.5) of the alkaline solutions, on the workability and strength characteristics of self-cured (air-cured) alkali activated Indian slag concrete mixes. Further the split tensile and flexure strengths for optimal mixes were studied for each dosage of Na_2O.It is observed that increase in Na_2O concentration increases the compressive, split-tensile and flexural strengths, both at the early and later-ages, while increase in Ms, decreases the workability of the mixes. An optimal Ms of 1.25 is found at various Na_2O dosages. No significant differences in the strength performances were observed between AASCs manufactured with alkali solutions prepared using either of potable and de-ionized water.

***Keywords*—**Alkali activated slag, self-curing, strength characteristics.

I. Introduction

INDIA is the second largest producer of cement in the world and ranks fifth in the CO_2 emission. The cement production in India is increasing rapidly due to increasing industrial activities with large investments in the infrastructure sector, industrial sector and real estate business. The cement industry of India has a current capacity of approx. 325 million tons per annum (MTPA) and is expected to add 30-40 MTPA of capacity in the coming years.

The Government of India is planning to invest around US $ 1025 billion in the 12th Five Year Plan (2013 - 2018) for infrastructure development. To achieve this, huge quantities of cement are required and such production of cement consumes large quantities of naturally occurring materials like chalk, limestone, and clay. The increasing costs of carbon-intensive fuels like coal and other materials in clinker-making in the recent years have been leading to correspondingly escalating prices of cement. Thus, it is essential to find appropriate replacements for Ordinary Portland Cement (OPC). Considering the crucial importance of infrastructure development for the Indian economy, it is now evident that using more durable and less energy intensive construction materials is inevitable for the sustainability of the construction industry.

Continuous depletion of natural resources is forcing the researchers to search for new sources of raw materials that can be used in the production of building materials. The use of Ground Granulated Blast Furnace Slag (GGBFS) - A by-product of iron industry can not only expand the raw-material base, but also reduce emissions of a carbon dioxide into the atmosphere. GGBFS is a rich source of calcium and silica in the amorphous form and thus a perfect initial component for the manufacture of alkali activated binders**.**

Alkali-activated slag (AAS)-based cements are now receiving increasing interest all over the world as an alternative to OPC because of their higher strengths, better durability and lower carbon foot-print [1]-[3]. The main reaction product in them is a C-S-H gel with a low CaO/SiO_2 ratio. This C-S-H has been found to have some structural differences as compared to C-S-H gel present in hydrated OPC paste [4].

Caustic alkali, silicate salts and non-silicate salts of a weak acid can be used as alkaline activators to activate the slag-contents in an AASC mix. Different alkali activators are to be used depending on the choice of the primary or the starting materials [5]. It has been observed that slags get well activated with use of non-silicate salts of a weak acid or water glass, with different silica moduli - a silica modulus of 1.0-1.5 for basic slags, 0.9-1.3 for neutral and 0.75-1.25 for acid slags. They also suggest the use of contents of the order of 3-5.5% of Na_2O by weight of slag. While Bhakharev et al. [6] successfully used concentrations between 7-8%. Fernandez-Jimenez and Palomo [3] mention that increasing the concentration of the Na_2O leads to an increase in the mechanical strengths. A number of other parameters such as curing conditions, water content and Si/Al ratio of initial materials also affect the properties of alkali activated cements and concretes [7]-[9].

Thus the alkali-activation reactions and the consequent strength behavior of AASC mixes are influenced by many variables - such as the chemical and mineral composition of the slag, the type and concentration of activators, pH, type of curing and temperature. However, alkali activation process of slags is still not fully understood and requires further research [10]. Many previous studies have investigated either the mechanical properties or durability of AAS mortars and concrete mixes, which are either heat-cured or water cured. Relatively only a few studies have addressed the activation of AAS systems at ambient, air-cured (self-curing) conditions.

In the present study, the engineering properties of Indian slag-based and air-cured AASC mixes, using various dosages of alkali-activated solutions were investigated. Sodium oxide (Na_2O) dosages at 4%, 6% and 8% of the total weight of slag

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were considered. Sodium silicate solutions with modulus ratios (mass ratio of SiO_2 to Na_2O) of 0.5 to 1.5 were adopted. The alkaline solutions are prepared by dissolving the appropriate quantities of NaOH flakes in sodium silicate solutions. Additionally, required quantities of water were added to the alkaline system in order to maintain a constant effective water/binder ratio of 0.45, taking the water content of the sodium silicate solution also into account.

Workability (Slump) tests and compressive strength tests were conducted for all the candidate AASC mixes. The split-tensile strengths and flexural strengths are however determined only for optimal AASC mixes and are compared with the results for control concrete produced using OPC. In addition to this, effect of using deionized water instead of potable lab water to prepare the alkaline solutions is investigated. The effect of the age of alkaline solution used during the mixing of the AASC mixes, on their strength characteristics, is also studied.

II. Experimental Program

A. Materials

The GGBFS used was procured from JSW Iron and Steel Plant, Bellary, India. The ordinary Portland cement (OPC) 43grade conforming to IS 8112 – 2013[18], was used for reference concrete. The chemical composition and physical properties of the GGBFS and OPC are presented in Table I. The basicity coefficient, K_b [K_b = (CaO + MgO)/(SiO_2 + Al_2O_3)] and the hydration modulus HM [HM = (CaO + MgO + Al_2O_3)/SiO_2], of the GGBFS, based on its chemical composition, were 0.94 and 1.81, respectively.

A mixture of Sodium hydroxide and Sodium silicate solution ($Na_2O.xSiO_2.nH_2O$) is commonly used as an alkaline activator [11]. In this study too, the alkaline activation of the GGBFS was carried out using a solution with commercial grade NaOH flakes (97% purity, density = 2110 kg/m^3) dissolve in a sodium silicate solution (14.7% Na_2O + 32.8% SiO_2 + 52.5% H_2O by mass, and density = 1570 kg/m^3)

River sand conforming to Zone II of IS 383-1983[19] and crushed granite of 20 mm maximum size (MAS) were used as fine and coarse aggregates respectively. The specific gravity of the coarse aggregates and fine aggregates were 2.69 and 2.64 respectively. The water absorption of the coarse and fine aggregate was found to be 0.98% and 2.2%, respectively.

TABLE I
CHEMICAL COMPOSITION OF OPC AND GGBFS (% BY WEIGHT)

Oxide %	OPC	Slag
CaO	63.47	34.78
SiO_2	19.9	33.9
Al_2O_3	4.62	15.13
Fe_2O_3	3.97	0.26
MgO	1.73	11.47
SO_3	2.56	0.34
K_2O	0.57	1.08
Na_2O	0.15	0.78
MnO	0.009	1.26
LOI	1.95	-
Blaine fineness	366 m^2/kg	370 m^2/kg
Specific gravity	3.13	2.9

B. Mix Design and Specimens Preparation

Mixes of OPC based normal concrete with binder contents of 370 kg per cubic meter were designed, using broad guidelines of IS 10262 – 2009 [20]. OPCCs were prepared with the desired W/ dry binder (C) ratio at 0.45. After thorough mixing, the concrete was used to cast cube, prism and cylinder specimens of 10x10x10cm, 15cmx30cm and 10x10x50cm respectively. After initially keeping the moulds for 24 hrs in an atmosphere at 95% relative humidity at 27^{0}C, the specimens were de-moulded and stored in water until testing. The same binder content and water/binder ratio as in OPCC were adopted for alkali-activated slag concretes (AASCs). For alkaline activator solution, mixtures of NaOH and sodium silicate solution, with varying modulus ratios (mass ratio of SiO_2 to Na_2O) of 0.5–1.5, were used. The combined solutions were allowed to cool to room temperature before using them in the mixes. The details of the mix-proportions for the various candidate mixes are given in Table II. The slump values of all the mixes were recorded as per IS: 1199 – 1959 [21]. The ASSC test specimens were de-moulded after 24 hours and were allowed to cure in air at laboratory conditions with 70-80% RH and 25-31^{0}C of temperature until testing. Three cube specimens each were tested for determination of compression strengths at each of 3 days, 7 days, 28 days and 56 days of age, as per IS: 516 – 1959 [22]. The splitting tensile strengths (using cylinders) and flexural strengths (using prism specimens) of the optimal mixes were determined at 7 days and 28 days as per I.S 9399: 1979 [23] and IS: 5816 – 1999 [24] respectively. Mix M1 to M15 represent the mixes prepared by using potable water in alkali activating solution, while MD1 to MD5 represent mixes prepared by using de-ionized water in alkali solution.

TABLE II
MIX PROPORTIONS OF THE CONTROL AND AASC MIXES

Mix	Cement kg/m^3	GGBFS kg/m^3	Coarse aggregate kg/m^3	Fine aggregate kg/m^3	% Na_2O	Modulus	NaOH kg/m^3	Na_2SiO_3 kg/m^3	Added Water kg/m^3
Mixes Prepared with Potable water									
Control	370	Nil	1251	661			-	-	166.5
M1			1217	643		0.5	14.8	22.6	154.5
M2			1217	643		0.75	12.7	33.8	148.5
M3	Nil	370	1217	643	4	1	10.5	45.1	142.5
M4			1217	643		1.25	8.4	56.4	136.5
M5			1216	643		1.5	6.3	67.7	130.5
M6			1209	639		0.5	22.2	33.8	148.5
M7			1208	639		0.75	19.0	50.8	139.5
M8	Nil	370	1208	638	6	1	15.8	67.7	130.5
M9			1208	638		1.25	12.6	84.6	121.5
M10			1207	638		1.5	9.4	101.5	112.5
M11			1200	634		0.5	29.6	45.1	142.5
M12			1200	634		0.75	25.4	67.7	130.5
M13	Nil	370	1199	634	8	1	21.1	90.2	118.5
M14			1199	634		1.25	16.8	112.8	106.5
M15			1198	633		1.5	12.5	135.4	94.5
Mixes prepared with De-ionized Water									
MD1			1217	643		0.5	14.8	22.6	154.5
MD2			1217	643		0.75	12.7	33.8	148.5
MD3	Nil	370	1217	643	4	1	10.5	45.1	142.5
MD4			1217	643		1.25	8.4	56.4	136.5
MD5			1216	643		1.5	6.3	67.7	130.5

III. RESULTS AND DISCUSSIONS

A. Workability

The results in Table III show that OPC-based control concrete mix is having a very low slump as compared to most of the AASC mixes, all of which have the same w/b ratio. This is due to better dispersion and surface characteristics of the GGBFS particles, which are smooth, absorb little water during mixing and difference in viscosity of AASC compared to OPCC [12], [13]. Although the AASC mixes were highly workable, they showed rapid setting with increasing activator dosage and modulus. These results are in agreement with the results obtained by Adam et al. [14].

B. Compressive Strengths of AASC Mixes

The compressive strength results of OPCC and AASC mixes, at various ages, are also shown in Table III. In general, for a given modulus, as Na_2O dosage increases, strengths of the mixes also increase. It can be observed that for all Na2O dosages, higher strengths at all the four representative ages were recorded at Ms=1.25, which is in agreement with observations of both Wang et al. [5], and Adam [15]. A small reduction in 56-days strengths of M1 and M3 mixes is observed, which may be considered as statistically insignificant and it can be attributed as no significant strength improvement at 56 days. From Table III, one can also observe that there is no significant difference in strengths between mixes prepared with potable and de-ionized water. Hence it can be concluded that use of potable water for preparing alkali solution will not significantly affect the strengths of AASC mixes.

Split Tensile (ft) and Flexural Strengths (fr) of AASC Mixes

The results in Table IV indicate that increase in Na_2O dosage increases the split tensile and flexural strengths of concrete mixes at both 7-days and 28-days. It can be observed that split tensile and flexure strengths are higher than those of OPCC at all dosages which may be attributed to the higher compressive strengths of AASC mixes and also due to stronger bond between the aggregate phase and AAS as compared to the bond with OPC paste. As these candidate AASC mixes, cast with alkaline solutions with optimized modulus, indicate marginally higher ft and fr values as compared to OPC-based control concrete, crack resistance of AASCs could be superior to that of OPCC.

C. Correlation between Different Strengths

1. fc and ft

Srinivas Rao et al. [16] proposed relation between split tensile strength of standard cylinders to compressive strength of standard cubes made up of OPCC as $ft = 0.185\ (fck)^{0.785}$ and hence, ratio of ft / fc ranges from 5.0% to 9.0% for fc of 20 to 80 MPa. From Table IV, it can be observed that, split tensile strength test value of AASC varied from 6.5% to 7% of the compressive strength and is found to be within the above range for OPCC mix.

2. fc and fr

The data from Table IV shows that, the flexural strength, fr, increased with the square root of compressive strength, fc. Thus, the relationship between fc and fr of OPCCs can be assumed conservatively for AASCs also.

3. ft and fr

Table IV shows that flexural strength, fr is higher than the split tensile strength ft, and the ratio of ft/ fr for AASC is found to be in the range of 62.9% to 69.4% which is in a similar range of results as observed by Price, [17] for OPCC.

D. Effect of Age of Alkali Solution on Compressive Strength

In order to study the effect of age of alkali solution on AASCs, mix M4 was selected as a candidate. The alkali solution is stored in a closed plastic container, is stirred daily and then used for activation at different ages of the alkaline solution, as shown in Table V. The results indicate that the variations in strength, with the age of the solution up to 15 days are not significant. Hence it can be said that alkali solution can be used up to 15 days without causing any appreciable reduction in the strength characteristics of the corresponding AASC mixes.

TABLE III
WORKABILITY AND COMPRESSIVE STRENGTH OF OPC AND AASC MIXES

Mix	Slump mm	Compressive strength f_c (MPa)			
		3 days	*7 days*	*28 days*	*56 days*
Control	20	17.7	31.8	45.3	48.0
M1	140	22.5	31.4	36.2	35.1
M2	110	27.6	32.9	39.1	40.3
M3	95	30.9	38.4	43.7	42.7
M4	85	37.0	46.1	55.8	57.9
M5	65	31.5	41.2	53.5	55.2
M6	135	36.2	42.7	41.4	50.5
M7	110	39.1	41.5	43.6	54.1
M8	90	41.4	57.9	58.4	59.7
M9	80	43.6	56.8	62.4	65.2
M10	55	42.5	52.8	59.3	62.6
M11	90	49.1	56.8	70.1	70.7
M12	65	50.1	63.5	71.5	75.4
M13	35	54.1	73.1	82.7	84.9
M14	20	55.3	71.4	83.2	90.3
M15	15	58.1	67.3	72.5	77.4
MD1	140	22.0	30.2	34.4	34.4
MD2	110	28.6	31.0	39.2	38.7
MD3	100	29.8	38.7	44.8	44.8
MD4	85	38.1	45.9	55.0	56.6
MD5	60	31.0	42.1	55.5	55.5

TABLE IV
SPLIT TENSILE, FLEXURAL STRENGTH AND CORRELATION BETWEEN STRENGTH CHARACTERISTICS OF CONCRETES

Mix	Split Tensile strength f_t(MPa)		Flexural strength f_r (MPa)	
	7days	*28 days*	*7 days*	*28 days*
Control	2.2	3.4	3.6	5.2
M4	3.1	3.9	5.2	6.2
M9	3.9	4.3	6.2	6.6
M14	4.7	5.4	7.1	7.9
	Correlation between f_t and f_c f_t/f_c (%)		***Correlation between f_r and f_c*** f_r/f_c (%)	
Control	6.9	7.5	11.3	11.5
M4	6.7	7.0	11.3	11.1
M9	6.9	6.9	10.9	10.6
M14	6.6	6.5	9.9	9.5
	Correlation between f_t and f_c $f_t/\sqrt{f_c}$		***Correlation between f_r and f_c*** $f_r/\sqrt{f_c}$	
Control	0.39	0.51	0.64	0.77
M4	0.46	0.52	0.77	0.83
M9	0.52	0.54	0.82	0.84
M14	0.56	0.59	0.84	0.87
	Correlation between f_t and f_r f_t/f_r(%)			
	7 days		*28 days*	
Control	61.1		65.4	
M4	59.6		62.9	
M9	62.9		65.2	
M14	66.2		68.4	

TABLE V
EFFECT OF AGE OF ALKALI SOLUTION ON COMPRESSIVE STRENGTH

Age of alkali Solution (days)	Mix	Compressive strength (MPa)	
		7- day strength	*28-day strength*
1	M4	46.1	55.8
5		46.2	55.1
10		44.8	55.0
15		43.6	55.2
30		40.4	50.0

IV. CONCLUSIONS

The results of this initial investigation indicate:

1. Slags exhibit selectivity towards the modulus Ms of alkali activators. For a given sodium oxide dosage, increase in Ms decreased the workability of the AASC mixes; however they recorded much higher workability than OPC-based concrete mix. But, at higher dosages, quicker setting was observed almost in all the AASC mixes. Use of Sodium silicate solution with Ms = 1.25 provided the highest compressive strength exceeding that of OPC concretes of the same w/b ratio.
2. Strengths of AASC mixes depend on the modulus of the solution and concentration of alkalis. The compressive strengths of AASC mixes are higher at both early and later ages, such strengths far exceeding that of OPC concretes of the same w/b ratio. Sodium silicate solution with Ms = 1.25 provided the highest compressive strength, split tensile and flexure strengths up to 8% of Na_2O dosage.
3. The correlation between different strength characteristics of OPCC and AASC shows that, the relationship between fc & fr, fc & ft and ft & fr similar to those adopted for OPCCs can be assumed conservatively for AASCs also.
4. Use of potable water instead of de-ionized water for alkali activating solutions do not yield significant differences in strength development characteristics of AASC mixes.
5. Alkali activating solution prepared using potable water, stored in a closed container, and stirred daily can be used up to 15 days, without any significant changes in compressive strengths.
6. Alkali activated slag concretes develop sufficient strength even under self-curing condition thus leading to saving of water. Self-curing is of great benefit especially in arid and desert regions, which suffer from severe scarcity of water resources and also there is saving in electricity as there is no necessity of thermal curing, which not only reduces cost but also make AASC more environmental friendly.

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