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Geopolymer: Portlandcement Free Binder System From Industrial Wastes

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Abstract: Inorganic polymers, more commonly referred to as geopolymers, are aluminosilicate materials which have desirable physical and chemical properties as a binder, for a diverse range of potential applications, including: construction & infrastructure industries, immobilisation of toxic, hazardous and radioactive wastes and fire resistant composites, with a much smaller Carbon footprint than traditional Portland cements. In this paper, chemical activation of binder made from industrial by-products viz., fly ash and Ground Granulated Blast Furnace Slag (GGBS) by geopolymerisation is discussed. The influences of the concentration of activator (made from mixing sodium hydroxide and sodium silicate solutions) and activator to binder ratio (A/B) on performance characteristics and micro structure of binder are reported. Based on the experimental studies, the optimum Si/Al ratio is found to be 0.50 with maximum strength of binder system of 25 % Fly ash + 75% GGBS cured at room temperature being 65MPa. Applications of Geopolymer technology in the brick production have been made with the optimized mix proportions. The mechanical strength of paver blocks tested and it was found that Geopolymer blocks containing (25% Fly ash + 75% GGBS) with A/B ratio of 0.7 possessed relatively high compressive strength of 35MPa which is much above the desired compressive strength in a building block. The micrographs and the XRD patterns provide an insight into the product morphology of geopolymers. The lower carbon footprint of Geopolymeric composition in comparison with Portland-based cement is quantified in this paper and hence, the application of Geopolymer technology substantially reduces the green house gas emission of the construction industry.

Introduction

Each year, the concrete industry produces approximately 12 billion tonnes of concrete and uses about 1.6 billion tonnes of Portland cement (PC) worldwide. Indeed, with the manufacture of one tonne of cement approximately 0.8 tonnes of CO₂ are launched into the atmosphere. The cement industry accounts for 5-8% of worldwide CO₂ emission¹. In addition, cement industry consumes considerable amounts of virgin materials (limestone and sand) and energy (energy demand about 1700-1800 MJ/tonne clinker), producing each

tonne of PC requires about 1.5 tonnes of raw material¹. In light of these, the scientific community has undertaken to seek new processes, technologies and materials to provide the construction industry with alternative binders. Currently, the potential alternatives to OPC technology include calcium sulpho-aluminate cements, magnesium cements, alkali-activated fly ash, slag and other alumina-silicate materials. During the years 1976–1979, the new binder, namely geopolymer, were originated by Davidovits through inorganic poly-condensation reaction of aluminosilicate oxides with alkali silicates, yielding polymeric Si-O-Al bonds²⁻⁸. There was polymerization of individual aluminate and silicate species, which are dissolved from their original sources at high pH in the presence of alkali metals. The geopolymer is reported to have the general formula $M_n[-(\text{Si-O})_z-\text{Al-O}]_n \cdot w\text{H}_2\text{O}$, where M is the alkali element, ‘-’ indicates the presence of a bond, z is 1, 2, or 3, and n is the degree of polymerization⁹. Theoretically, any alkali and alkali-earth cations can be used as the alkali element (M) in the reaction; however, the majority of the research has focused on the effect of sodium (Na⁺) and potassium (K⁺) ions.

In Geopolymerisation or mineral polymerisation, $[\text{SiO}_4]^{4-}$ tetrahedra is the fundamental structural unit of these systems. Depending upon the nature of linkage and sharing of Silicon-oxygen tetrahedra, the silicate minerals exist in isolated as well as continuous chain structures. The network formation of $[\text{SiO}_4]^{4-}$ tetrahedra by sharing of oxygen with the adjacent tetrahedra indicates the polymerization structure. Polysialate, polysialate-siloxo and polysialate-disiloxo are the three important oligomeric structures for Polymerization reaction. The amorphous to semi-crystalline three dimensional silico-aluminate structures as geopolymers based on silico-aluminate and further categorised the geopolymers structure based on the ratio of Si/Al (Figure 1.0) was observed by Davidovits. The chain structures of $[\text{SiO}_4]^{4-}$ tetrahedra with cations and neutralizing anions build the mineral polymer framework. These silicate minerals possess excellent rock forming property akin to the calcium-silicate-hydrated structure that typically exists in hydrated Portland cement. Geopolymeric materials with proper mix design and formulations, derived from different silica & alumina source materials can exhibit chemical and mechanical properties superior to ordinary Portland cement (OPC) and are highly cost effective⁹⁻¹². Geopolymerisation of industrial wastes would involve environmental benefits such as the reduction in consumption of natural resources and decrease in the net production of CO₂ since it is estimated that the geopolymer cement synthesis emits 5–6 times less CO₂ when compared with Portland cement¹³. The present study focuses on preparation of functional flyash-GGBS based geopolymers using local resources from India, and evaluation in terms of microstructure, mechanical properties and potential for building blocks production.

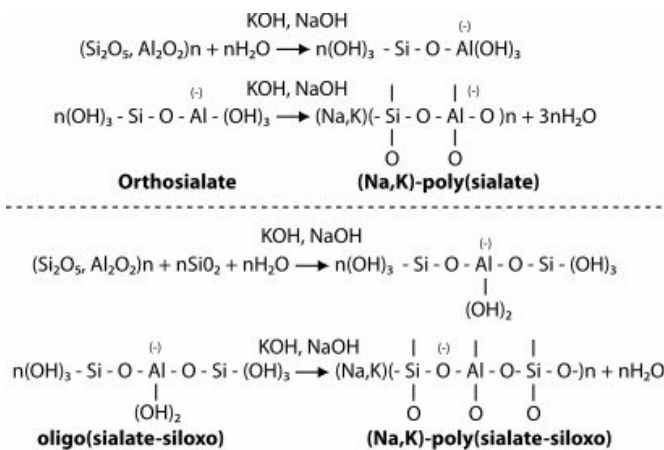


Figure 1.0. Scheme of geopolymeric reactions.

Experimental work.

Geopolymerisation require alkali activator solution (AAS) consisting of hydroxides and silicates of sodium whose concentrations play a major role in determining the rate and quantum of geopolymerisation. It is also very important to note that the ratio of alumina to silica (Si/Al) and Na/Al from ‘geo polymeric source material’ (GSM) such as fly ash, metakaolin, GGBS etc., which are possessing wide compositional range of Si, Al, etc, influence geopolymerisation reactions and thereby the performance characteristics. The properties of fly ash, GGBS and sodium silicate solution used in this investigation are shown in Tables 1a, 1b and 1c. The ratio

of SiO₂ and Al₂O₃ in the fly ash is around 2, suitable for making low CO₂ cements. Locally available river Sand passing through 4.75 mm is used as fine aggregate in the production of geopolymer blocks (Table 1d).

Production of geopolymers: Geopolymeric source materials (Fly ash and GGBS) of varying proportions such as 0%, 25%,50% Fly ash were taken and paste was prepared by mixing with the alkaline activator (made of hydroxide and silicate solutions of sodium). Raw material mix design is shown in Table 2.0. The SiO₂/Na₂O ratios (Ms modulus) of sodium silicate solution (Ms = 2.1 & 2.6) was modified by the addition of NaOH solution to a value near to 0.63-0.65. The other important parameter, weight ratio of activator liquid to binder (A/B) is varied since the performance characteristics is mostly depend upon the liquid quantity used.

The activator solution was prepared by mechanically mixing of sodium hydroxide solution with sodium silicate solution for 1 min and kept for 24 hours. Activator solution and solids were mixed for 15 minutes in an electrically operated mixer. The fresh mix was then poured into moulds (cylinders: 75 φ x 150 mm H, Bricks: 200x100 x100 mm) and were cured in room temperature. At the end of curing period, specimens were removed from the moulds and tested for compressive strength, each time a minimum of 5 specimens were used.

Table 1(a) Chemical Composition of GSMs Table 1(b) Physical Properties of GSMs

Chemical Composition	Fly Ash class F	Blast Furnace Slag Powder
CaO	1.3	40.3
SiO ₂	60.3	43.4
Al ₂ O ₃	25.5	12.5
Na ₂ O	0.4	0.9
K ₂ O	0.8	0.6
MgO	0.8	1.5
Fe ₂ O ₃	7.8	0.3
LOI	1.4	2.1
Specific Gravity	2.21	2.91

Property		GSMs for GPCs	
		Fly Ash	GGBS
Specific Gravity		2.25	2.91
Fineness (Blaine)	m ² /kg	330	425
Bulk density	kg/m ³	1005	1360
Residue on 45 micron sieve	%	35	ND
CEF, k at 28 day		0.93	0.95
LOI	%	1.4	1.8
Glass content 85(BS:6699)	%		96

Table 1(c) Properties of SSS Soln

Particular	1:2.00	1:2.60	1:3.20
Specific Gravity	1.56 - 1.66	1.52 - 1.55	1.38 - 1.41
Baume	51 - 55	47 - 49	39 - 41
Na ₂ O	15.50 - 16.50%	11.50 - 12.50%	8.50.95.0%
SiO ₂	31.0 - 33.50%	31.0 - 33.50%	28.0 - 30.0%
Weight Ratio	2.0+ /-0.5	2.60+ /-0.5	3.20+ /-0.5

Table 1(d) Properties of Aggregates

Source	River Sand	Crushed Granite Aggregate
Type	Fine Aggregate	Coarse Aggregate
Specific Gravity	2.61	2.72
Fineness Modulus	2.73	6.68
Bulk density kg/m ³	1540	1720
Water absorption (%)	0.83	1.2
Flakiness index	NA	18.72
Elongation Index	NA	36.27
MSA, mm	4.75	20

In order to get the idea of morphology, the reaction products were examined by scanning electron microscopy (SEM) with Quanta 200 FEG instrument under 15 kV accelerating voltage conditions. Environment Scanning Electron Microscopic (ESEM) mode was used for high resolution morphological surface analysis.

The elemental analysis through the Energy Dispersive X-Ray spectrometry (EDX) was done to know the crystalline information via electron Back Scattered Detection (BSD) system attached with microscope.

Preparation of Blocks

The manufacturing process of geopolymer building brick consisted of: raw material preparation, activator addition and mixing, casting and demoulding followed by curing. The process is considered as a green process, useful to manufacture of cold setting building products. Quarry dust is used as fully replacement of fine aggregate for one set of blocks (Table 5.0). Weight quantities of powdery ingredients (viz., FA and GGBS) were poured in a pan mixer; the aggregate was added and mixed thoroughly till homogenous mix is obtained. Then liquid alkaline activator was added to the dry mix and mixing was continued to get a uniform mix. A Compressed Block Making machine was used to mould the Blocks. Curing of blocks by storing them at ambient temperatures till testing for compressive strength in the particular interval.

Results and Discussion

Compressive strength (Tables 3.0 and 4.0)

The formation of Geopolymer requires reactive precursor materials and a high concentration of alkaline activator solution which induce the dissolution of silicon and aluminium ions to form mobile precursors and then precipitation of solid phases. Generally, minerals which dissolve readily when concentration of NaOH is increased, as compared to KOH, but higher compressive strengths were reported when KOH was used. This is because of larger K ion favour the formation of large silicate oligomer with aluminium hydroxide $[(Al(OH)_4]^-$. Palomo *et al.*³ suggested that an excess of OH^- concentration in the system can lead to decrease in mechanical strength. Increasing the activator concentration beyond a certain alkali content (depending on mineral, activator and curing conditions), may not result in further increase in strength, and detrimental effects such as efflorescence and brittleness resulting from the effects of high free alkali in the product have been reported.

Table 3. Effect of Molar ratios of SSS on compressive strength

Size of the Specimen (mm) Plastic Disposal Moulds $75 \phi \times 150$: GSM/AAS ratio=0.5.
Alkali activator solution(AAS) = mixture of Sodium hydroxide and silicate solutions

Mix Id	Molar Ratio	Flyash in GSM (%)	Mix ratios GSM : Sand : CA	Slump mm	Density kg/m^3	Comp Strength MPa		%Reduction	
						7d	28d	7d	28d
G1	2.1	0	1:1.5:2.5	80	2402	38	39		
G1a	2.6	0	1:1.5:2.5	68	2394	28	32	26	18
GF1	2.1	25	1:1.5:2.5	60	2436	36	38		
GF1a	2.6	25	1:1.5:2.5	46	2401	25	29	31	24
GF2	2.1	50	1:1.5:2.5	48	2359	27	33		
GF2a	2.6	50	1:1.5:2.5	40	2397	19	24	30	27

Table 4. Effect of A/B ratio on compressive strength of GPC matrix

Size of the Specimen (mm) 200x100 x100: Molar ratio of sodium silicate solution: 2.4.
Alkali activator solution (AAS) = Mixture of sodium hydroxide and silicate solutions
B=GSM, A=AAS

Mix Id	A/B Ratio	Fly ash (%)	Mix Ratios (GSM : Sand : CA)	Slump (mm)	Density (kg/m^3)	Comp Strength (MPa)		% Reduction	
						7 day	28 day	7 day	28 day
G50	0.5	0	1:1.9:2.8	72	2439	61	68		
G55	0.55	0	1:1.9:2.8	42	2395	52	52	15	24
GF50	0.5	25	1:1.9:2.8	54	2401	57	63		
GF55	0.55	25	1:1.9:2.8	28	2384	42	46	26	27

The most important property of sodium silicate is the molar ratio of SiO_2 to Na_2O . Silicates are commercially produced in the $\text{SiO}_2:\text{Na}_2\text{O}$ ratio in the range of 1.5–3.2. In general, high ratio silicates (i.e.3.2) are most suitable for chemical bonding since it is the siliceous portion of the silicate that reacts with cations to form polymeric compounds. But, in geopolymer technology, due to the importance of maintaining a high alkalinity, it has been recommended that lower molar ratio silicates are generally preferred for activation of sources of alumina and silica. In the present work, industrial sodium silicates with $\text{SiO}_2/\text{Na}_2\text{O}$ ratios of 2.1 and 2.6 were used to form geopolymers from GGBS and its combination with Fly Ash. It was found that 100% GGBS activated by alkali activator solution shows higher compressive strength due to its high calcium oxide content; 28 day strengths recorded were 39 MPa and 32 MPa for Ms of 2.2 and 2.6 respectively. However the addition of fly ash to GGBS, to an extent of 50% did not alter the compressive strength very much. This is an indication of better utilization of fly ash particles in forming the reaction product.

Compressive strengths are found to decrease with increase in activator-to-binder ratio(0.5 to 0.55) even though a higher activator-to-binder ratio effectively contains a higher amount of the activating alkali metal ions and silicates. This might be due to the increased capillary porosity created in the reaction product because of the increase in water portion in the activator solution. In the present study, test data shows that the activator concentration has a greater influence on the compressive strength of geopolymer concretes with fly ash. While main basis of compressive strengths of the fly based Geopolymer concretes the formation of sodium aluminosilicate gel, but, for geopolymer with GGBS, some calcium bearing binder compounds (which may not be geopolymeric in nature) also could be forming, even when activator concentrations are very low. This could be a reason for increased strengths of GGBS based matrix at lower activator concentrations as compared to fly ash based matrix.

Microstructure of Geopolymeric paste

Scanning electron microscopy (SEM) was used to understand the morphology of the reaction products in geopolymeric pastes. **Figs. 1, 2, and 3** show the micrograph of geopolymeric pastes made using GGBS with 0%, 25% and 50% fly ash respectively as the Geopolymeric source material and activated by sodium hydroxide and sodium silicate solution with Ms of 2.1 and A/Bratio of 0.50. Fig 1.0 shows the micrograph of Geopolymer having 100% GGBS, where individual particles of GGBS cannot be isolated. The reaction product formed is much more compact, homogeneous and uniform. The activator solution has reacted with geopolymer source materials to form dense microstructure leading to increased compressive strength. During the reaction, soluble phases of fly ash are dissolved by NaOH to release alumina and silica. These phases react with activator solution further and condense on the surface of fly ash particles in the form of the aluminosilicate gel shell¹⁴⁻¹⁷.

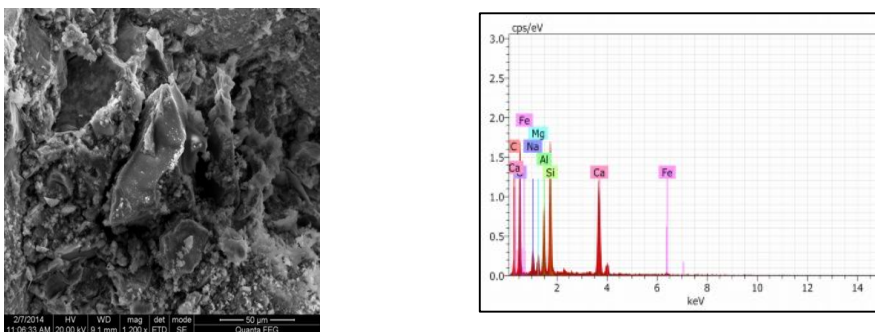


Fig.1 SEM Image of (100%GGBS) Mortar mix and its corresponding elemental analysis

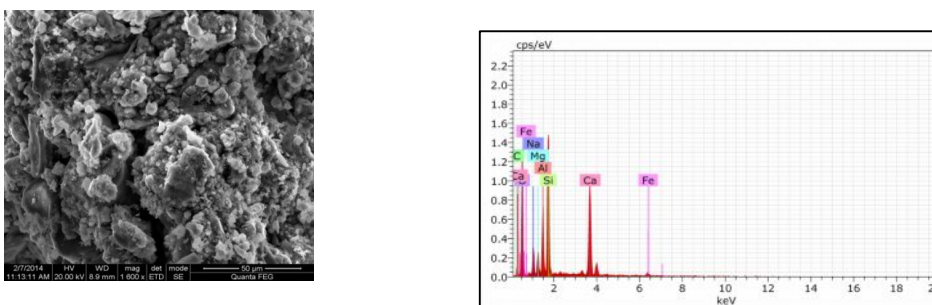


Fig. 2 SEM Image of (75%GGBS) Mortar mix and its corresponding elemental analysis

In the present study, the SEM photograph of the specimens containing fly ash precursors along with GGBS show the formation of gel covering the fly ash particles. In the specimen having 25% fly ash, not much of unreacted fly ash particles are seen proving that considerable reaction has taken place. As the fly ash content increased to 50%, microstructure shows thin layer of reaction products around the fly ash particles reflecting the degree of reaction to be moderate. EDS analysis showed that calcium content is found to be more in GGBS system (100 % ggbs:10.0) comparatively than combination with fly ash.(specimen with 25%-8.0 50%FA-11.0)

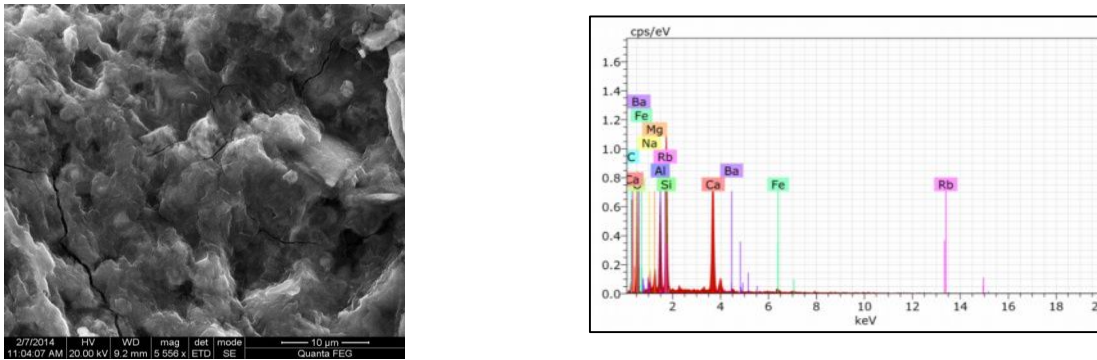


Fig. 3 SEM Image of (50%GGBS) Mortar mix and its corresponding elemental analysis

Production of geopolymeric paver blocks

Compressive strengths of Geopolymer paver blocks with and without quarry dust as replacement material for sand, tabulated in Table 5.0 were determined as per the guidance given in IS: 1077-1992. Geopolymer blocks with quarry dust have low dry density and excellent strength compared to blocks without quarry dust. The 28 day compressive strength of 35MPa is much above that needed in a building block. As the % of fly ash content increases, the strength level decreases. The preparation of geopolymeric paver blocks is shown in Fig 5.0

Fig 5.0 Units of GPC Paver Blocks



(a) Paver Block Mould (b) Casting of Paver Block (C) Curing of GPC blocks

Table 5: Details of GPC Paver Blocks

Nominal weight of the sample: 3.5 kg. ; Nominal Size of the block: 20x9.5x9cm;
 Cross sectional area = 190cm²; Material used for each was about 170 kg

S No	%FA in GSM	%Sand in FAg	Mix proportions (by weight)				Compressive strength	
			GSM	Fag	CA	AAS	MPa	% Increase due to Sand
1	0	0	1	4.82	0.61	0.70	25.2	
2	0	24	1	4.73	0.62	0.70	33.2	32
3	25	0	1	4.82	0.61	0.70	25.6	
4	25	24	1	4.73	0.62	0.70	38.1	49
5	50	0	1	4.82	0.61	0.70	15.1	
6	50	24	1	4.73	0.62	0.70	15.1	0

Parameters for cost and ecological studies¹⁸

On the basic data given in **Table 6**, computations for comparative ecological evaluation of geopolymeric and ordinary Portland cement concrete mixes was made for a typical specimen samples. (**Table 7.0**).

Embodied Energy (EE): This is the net energy required to bring the material under consideration to the present level of utilization. Since most of the energy requirement in the present world comes from burning of fossil fuels, low EE value of any material and operation is always welcome as it leads to lower contribution to the global warming on account of reduced production of green house gases. Since the strength of material is also important, the EE involved in achieving the strength in the concrete can be quantified by dividing EE of the concrete by the compressive strength. This factor, EE/f_{c28} , would have units of MJ/MPa and a lower value would indicate more efficient use of energy from structural considerations.

Embodied Carbon Dioxide Emission (ECO_{2e}):Emission may be due to the CO₂ coming out as a by-product in production technology for the materials involved or it could be due to the type of fuel used to produce energy for various operations of material production, processing and its utility. In case of conventional concretes, the major amount of embodied CO₂ is due to Portland cement. In the present case, the CO₂ emission is expressed in terms of kg of CO₂ emission involved for production of 1 m³ of concrete. This factor ECO_{2e} is expressed in kg of CO₂ per m³ of concrete, i.e. kgCO_{2e}/m³ and strength combined parameter, i.e., ECO_{2e} per unit strength is calculated as ECO_{2e}/f_{c28} . A lower value of ECO_{2e}/f_{c28} must be preferred obviously.

Table 6. Data on ingredients for ecological calculations:

	EE	ECO _{2e}	Cost
Ingredient	MJ/kg	kgCO _{2e} /kg	Rs/kg
Fly ash	0.1000 ²²	0.010 ²²	1.00
GGBS	1.6000 ²⁴	0.0830 ²⁴	3.00
OPC	4.7980 ¹⁹	0.9300 ¹⁹	5.00
SHf	20.5 ²⁰	3.2 ²¹	20.00
SSS	10.2 ²¹	2.0 ²¹	12.00
DW	0.2500	0.001	2.00
Water	0.200 ²²	0.0008 ²²	0.01
SP	11.47 ²²	0.600 ²²	50.00
Sand	0.0810 ²⁴	0.0051 ²⁴	1.00
CA	0.0830 ^{23,24}	0.0048 ^{23,24}	1.00
Processing	0.15 ²⁵	0.003 ^{26,27}	1.00

Table 7.0 Ecological analysis of building blocks . (Typical Geopolymeric blocks prepared in the present study is compared with OPC.

28 day Concretes strength (fc 28 d); Embodied Energy (EE),
Embodied carbon-di oxide emission (ECO_{2e})28 day Concretes strength (fc 28)

S. No	Parameter (unit)	Geopolymeric mixes%FA in GSM			OPC 43	Remarks.
		0	25	50		
1.	EE (MJ/m ³)	1829	1658	1492	2453	Lower value is preferred.
2.	ECO _{2e} (kgCO _{2e} /m ³)	214	204	196	442	
3.	Cost (Rs/m ³)	4361	4227	3891	4246	
4.	fc28(MPa)	66	60	55	43	Higher value is preferred.
5.	EE/f _{c28} (MJ/MPa)	28	28	27	31	
6.	ECO _{2e} /f _{c28} (kgCO _{2e} /MPa)	3	3	4	5	
7.	Cost/f _{c28} (Rs/MPa)	66	70	71	66	

Cost (Rs/m³):

Construction involves very high level of investment in any society, and hence, the cost of production of the concrete is very important. Sometimes had higher costs on unit volume basis, but when viewed in terms of cost/strength, the GPC mixes were acceptable since the cost involved for production of unit strength was lower

Conclusion

This paper has provided extensive experimental results and analysis on the compressive strengths and microstructure of cement-free binder pastes and/or concretes containing either GGBS or combination with fly ash as the sole binder and activated using NaOH and sodium silicate solutions of different concentrations. The following conclusions are made based on this study:

(i) The user-friendly geopolymerization conditions applied to the system fly ash/slag/Na-silicate/H₂O yielded geopolymer bodies resembling concrete product, acts as a binder cured at room temperature. (ii) Synthesized Geopolymer have shown excellent mechanical strength compared to that of ordinary Portland cement. Compressive strengths of Geopolymer mortar were found to increase with increase in activator concentration and decrease in activator-to-binder ratio. The Geopolymer mortars made using GGBS as the starting material showed higher compressive strengths compared to those made with fly ash because of the self-cementing property of GGBS and higher binder content used. Geopolymer mortars made using GGBS and fly ash had similar compressive strength when activated using higher concentration of the activator. At lower concentrations of the activator, GP concretes with GGBFS showed higher compressive strength of structural grade concretes of order starts from 30MPa - 60Mpa. (iii) Building blocks made of Geopolymer mix proportions having 25% FA & 75% GGBS with partial replacement of sand with quarry dust showed comparatively high compressive strength than that of ordinary Portland cementitious system. (iv) Microstructural observations revealed the formation of alkali aluminosilicate gel as a shell around the fly ash particles whereas the reaction product was more homogeneously distributed in pastes made using GGBS. The reaction product shell formed around the fly ash inhibits further activation, resulting in incomplete utilization of the fly ash particles. (v) Highly favorable environmental impact is evident.

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