



Research article

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Impact of pond ash as fine aggregate on mechanical and microstructural properties of geopolymer concrete

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Abstract. In this research, geopolymer concrete (GPC) was produced using pond ash (PA) as a fine aggregate replacement to river sand (RS). The focus of the study is to investigate the effect of PA as fine aggregate on the mechanical and microstructural properties of GPC. The present study detailed characterization of PA which includes particle size distribution, X-ray diffraction (XRD) analysis, scanning electron microscopy (SEM) analysis, energy dispersive X-ray (EDAX) studies, and chemical analysis including reactive silica (R-SiO₂). PA replaced RS at percentages of 0, 20, 40, 60, 80 and 100% in GPC. Properties like compressive strength, flexural strength, split tensile strength and water permeability of GPC were studied. It was found that compressive strength, flexural strength and split tensile strength of GPC with 80% PA as fine aggregate are, respectively, 26%, 29% and 31% greater compared to concrete with RS as aggregate. The inclusion of reactive sand-like PA increased the R-SiO₂/Na₂O ratio from 3.2 to 5.4 in GPC mixes with up to 80% replacement and contributed to higher compressive strength. SEM studies, R-SiO₂, EDAX and Fourier-transform infrared spectroscopy (FTIR) analysis of GPC confirmed the participation of PA particles in polymerization thereby contributing to higher strength. Utilization of pond ash as fine aggregate provides a valuable addition to GPC in aspects of strength and economics as well as alleviates the environmental problems caused by coal ash ponding.

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1. Introduction

Coal ash (CA) is a by-product from thermal power plant, which includes fly ash (FA) collected through electrostatic precipitator and bottom ash (BA) collected from bottom of the boiler furnace [1]. In most of the thermal power plants, the unused FA and BA are conveyed to landfills by means of water and is termed as pond ash (PA) [2]. Disposal of CA into the ash pond results in environmental problems like pollution of air, soil and water [3]. Hence, it is inevitable to find ways to utilize the ponded ash especially in construction sector to alleviate the previously mentioned environmental problems.

PA encompasses finer reactive particles of FA and coarser less reactive or non-reactive particles of BA [4]. Burnt clay bricks produced by replacing clay with PA have been studied earlier [5]. Sintered aggregates with high water absorption feature can be manufactured by using PA [6]. Presence of coarser non-reactive particles in PA, prompted researchers to use it as replacement to aggregates in cement concrete and higher water demand to achieve required workability was observed in such concretes [7]. PA has been tried as replacement to cement in cement mortar [2] and as raw material in the manufacture of cement by black meal process [8].

Geopolymer technology is emerging as one of the alternative binder to cement when it comes to sustainability and durability aspects [9]. In geopolymer concrete (GPC); an aluminosilicates material is activated by alkaline solution forming a paste which binds coarse and fine aggregate with other unreacted material. The fresh and hardened properties of GPC depend on proportioning and properties of alkaline solution and base material [10]. In earlier studies, coal ashes are used as precursor [11, 12] and as aggregates [13, 14] in making geopolymer composites. In production of geopolymer mortar, PA is used as base material along with ultra-fine slag and the resultant mortar exhibited good durability characteristics [3]. Earlier researchers have tried using PA as fine aggregate and replacement to cement in cement concrete and as base material in geopolymer mortar. PA as fine aggregate in GPC needs to be explored.

During the year, 2018-19, in India 217 million tons (MT) of CA has been generated from 195 thermal power plants. In which 22 % of CA has been disposed as PA [15]. Disposal of CA into the ash ponds results in degrading the fertility of soil and quality of water [3]. Further aggregates which contributes 70 % of the volume of concrete are derived from natural queries and river sand [16]. Increase in the demand of concrete in future, is likely to exploit the non-renewable sources by taking raw materials for cement manufacturing and aggregate production. Geopolymer composites made up of using industrial by-products as both base material and as aggregates is one of the alternative solutions for previously mentioned problems. Hence, the present study focusses on making of geopolymer concrete using FA and ground granulated blast furnace slag (GGBS) as base material and PA as replacement to river sand (RS) at 0,20,40,60 and 100 percentages.

The objective of the present study is to assess the influence of PA as fine aggregate replacement to RS on mechanical properties of GPC. In addition, using scanning electron microscope (SEM) studies, energy dispersive X- ray analysis (EDAX) and Fourier transform infrared spectroscopy (FTIR) analysis microstructure of GPC made up of PA is evaluated.

2. Methods

2.1. Materials

The raw materials used in this study were FA, GGBS, PA, coarse aggregate and RS. GGBS was purchased from local manufacturers, Coarse aggregate and RS from local suppliers. FA and PA were procured from Udupi thermal power plant, Karnataka, India. Physical properties of materials are shown in Table 1, Specific surface area of GGBS is 400 m²/Kg and is higher than cement and FA [17], further PA has exhibited greater moisture absorption compared to RS and coarse aggregate. In PA, percentage of particles finer than 600 μ are exceeding the limit prescribed in IS 383-2016 for zone II sand [18] which can be noticed from Fig. 1. Particle size distribution curves in Fig. 2 reveal that FA and GGBS exhibit wide range of particle sizes compared to PA. Chemical composition and Reactive silica (R-SiO₂) of FA, PA and GGBS were determined using gravimetric analysis and is presented in Table 2. It confirms that FA comes under Class F Pozzolan as per ASTM C618 [19] and PA has abundant silica and aluminium oxide. Even though GGBS has less Total silica (T-SiO₂) compared to FA and PA, R-SiO₂ is more in GGBS than other two. Pondering action and presence of BA yielded less R-SiO₂ values for PA than FA as observed from Table 2. X-ray diffraction (XRD) curves shown in Fig. 3 supports R-SiO₂ values, absence of crystalline peaks in GGBS confirms presence of greater reactive or amorphous phases. Hump formation in XRD curve between 20° and 30°, is indication of presence of amorphous phase and is better for FA than PA. Elemental composition of PA (Fig. 4) obtained by EDAX revealed that PA is rich in silica and alumina, same result was observed in the earlier studies [20].

SEM images of cement, GGBS, FA and PA are shown in Fig. 5. Cement particles are angular in shape, while particles of GGBS are in anomalous shape with clear edges and angles [21]. FA particles are spherical [22] and PA composed of both spherical FA particles and agglomerated porous coarser BA particles same is depicted in Fig. 5.

Sodium silicate (Na₂SiO₃) and sodium hydroxide (NaOH) were procured from local supplier. Pellets of NaOH with 98 % purity were used in this research. Chemical composition of silicate was 11 % of Na₂O, 31.25 % of SiO₂, 13.08 % of R-SiO₂ and 56.5 % of H₂O.

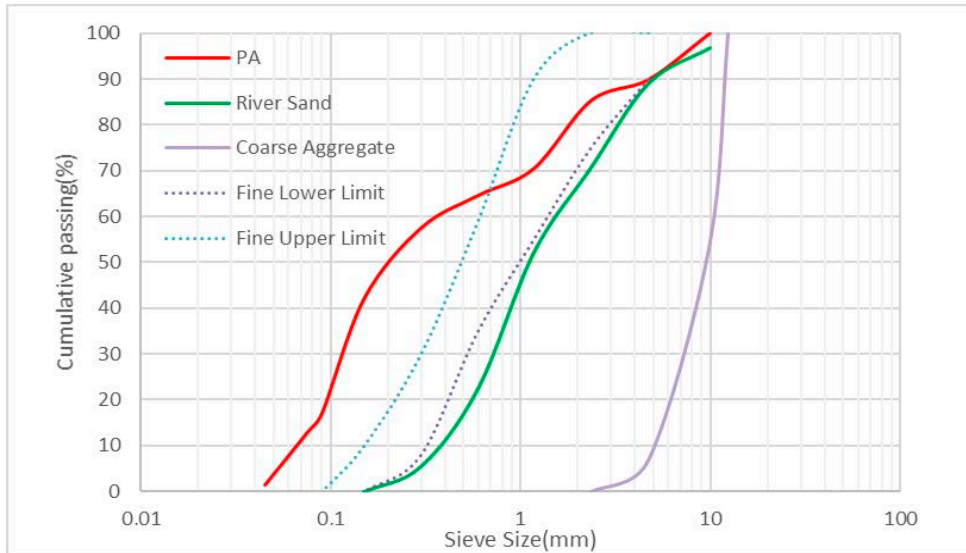


Figure 1. Grain size distributions of aggregates.

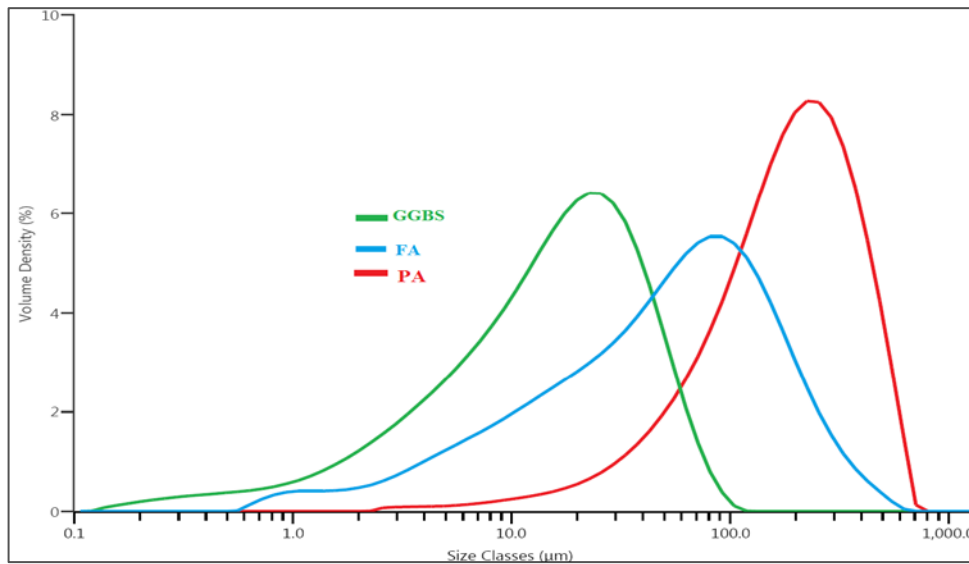


Figure 2. Particle size distribution curves.

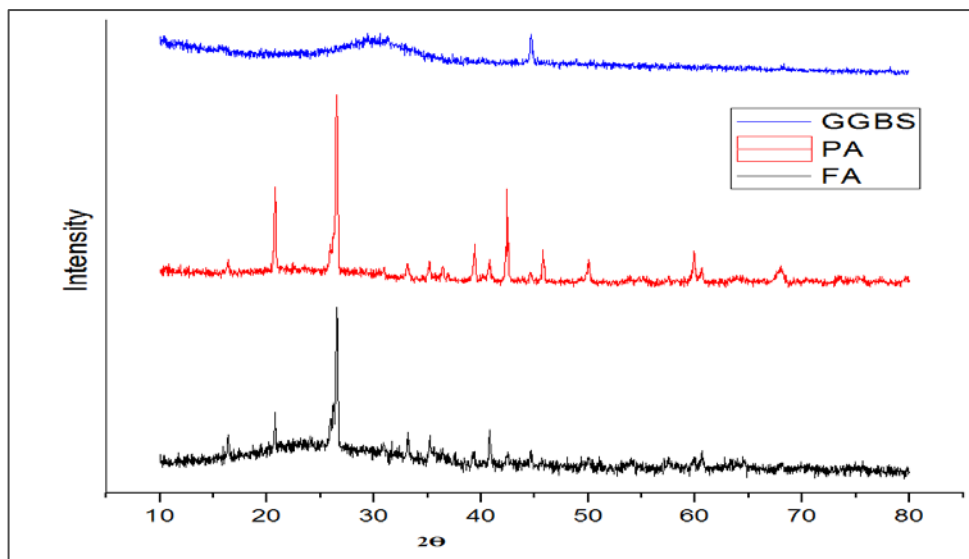


Figure 3. XRD curves of GGBS, PA and FA.

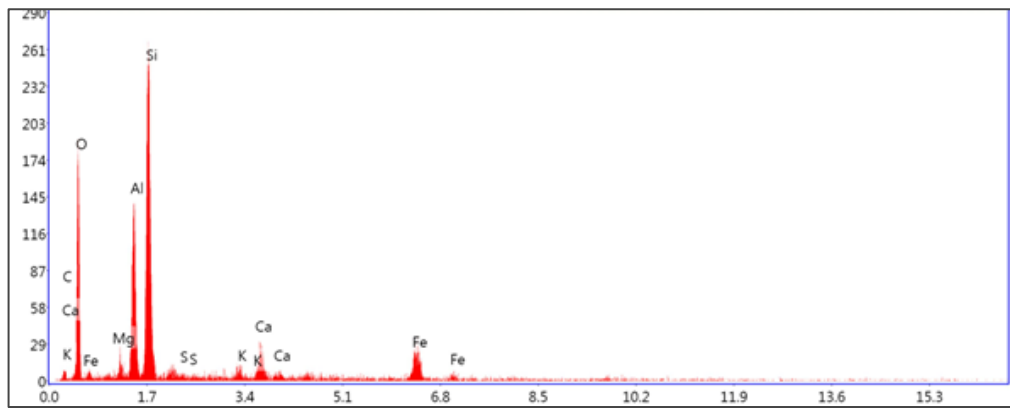


Figure 4. Micrograph of Pond Ash obtained by EDAX analysis.

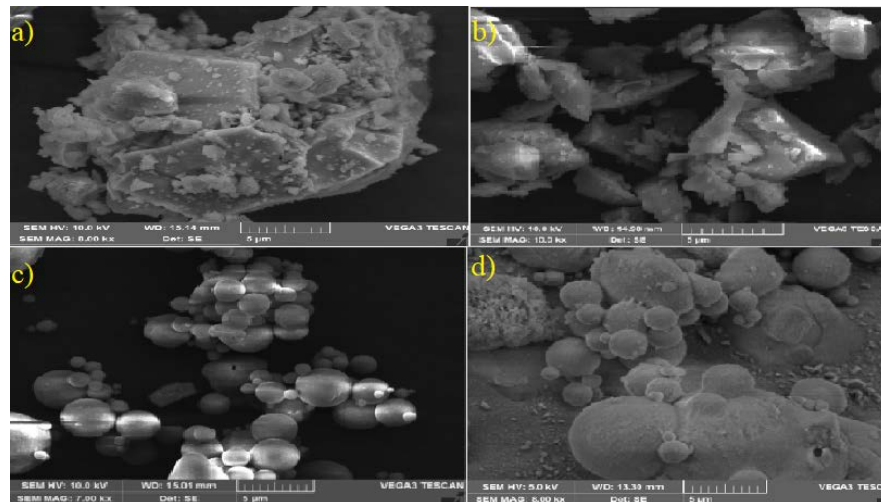


Figure 5. SEM images of a) Cement b) GGBS c) FA d) PA.

Table 1. Physical properties of materials

Properties	PA	River sand	Coarse Aggregate	FA	GGBS
Residue on 45 μ (%)	–	–	–	32	10
Blaine surface area (m ² /Kg)	–	–	–	386	400
Specific gravity	2.45	2.50	2.65	2.10	2.90
Fineness modulus	4.75	2.80	7.80	–	–
Moisture absorption (%)	5.2	1.5	0.1	–	–

Table 2. Chemical composition of GGBS, FA and PA

Oxides (%)	GGBS	FA	PA
LOI	1.41	0.57	5.30
T-SiO ₂	33.80	56.16	58.38
R-SiO ₂	32.80	29.71	20.60
Al ₂ O ₃	17.36	20.16	25.34
Fe ₂ O ₃	1.36	10.34	5.68
CaO	38.30	7.20	2.69
MgO	4.90	2.26	0.98
SO ₃	0.02	0.41	0.06
Others	2.85	2.90	1.57

2.2. Mix Proportions

Geopolymer concrete mix proportioning was carried out by fixing total water content and density of concrete to 202 Kg/m³ and 2400 Kg/m³ respectively. Here, total water content includes water present in sodium silicate and sodium hydroxide solution. Mix proportions for pond ash geopolymer concrete represented in Table 3. RS, PA and coarse aggregates were mixed with FA and GGBS for four minutes in dry state. Later, alkaline solution was added and mixing was continued for five more minutes. Concrete was poured in moulds for testing with proper compaction carried out using vibrating table. All the test samples were cured in ambient temperature at 27 °C till the day of testing.

For concrete casting, alkaline solution was prepared one day prior to casting by mixing 10M sodium hydroxide and sodium silicate solution in the ratio 1: 2.5 respectively.

Table 3. Mix proportions per m³ of geopolymer concrete

Mixes	Replacement of River Sand	Fly ash (Kg)	GGBS (Kg)	River Sand (Kg)	Pond ash (Kg)	Coarse aggregate (Kg)	NaOH (Kg)	Na ₂ SiO ₃ (Kg)
PA0	0			660	0			
PA20	20%			528	132			
PA40	40%			396	264			
PA60	60%	330	83	264	396	990	96	241
PA80	80%			132	528			
PA100	100%			0	660			

2.3. Testing

Workability of GPC was measured using slump cone test as per IS : 1199 – 1959 [23]. Compressive and flexural strength of GPC were tested following the guidelines of IS : 516 – 1959 [24]. IS 5816:1989 [25] specifications were followed in finding the splitting tensile strength of GPC. Cylinder of size 200 mm height and diameter of 100 mm were casted and tensile strength was found out by splitting cylinder at loading rate of 1.2–2.4 MPa/min.

Water permeability of GPC was found out by measuring the water ingress into the cylinder specimens under 5 bar pressure applied for a period of 3 days as mentioned in DIN 1048 [26]. After 3 days, penetration of water is measured by splitting the cylinder along long side using compression machine. Test set up for water permeability shown in Fig. 6.



Figure 6. Test setup for Water permeability.

3. Results and Discussion

3.1. Fresh property

Increase in PA replacement to RS, resulted in decreasing of workability of GPC which can be noticed from Table 4. Since PA contains porous bottom ash particles, these porous bottom ash particles absorb water and cause water deficiency for lubrication of particles [27–29]. In addition, as depicted in Fig. 1

particles finer than 600 μ in PA are considerably greater in quantity than in RS. Presence of these finer particles in PA demands extra water for wetting their outer surface. Thus, increase in PA content in GPC drops the workability. Further, handling of GPC became difficult at 80 % and 100 % replacement as it turns into more cohesive. In order to restore the workability, naphthalene based superplasticizer (SP) i.e. Conplast SP 30 was used in PA 80 and PA 100 mixes. As a result of this slump increased from 200 to 250 mm and 50 to 185 mm in 80 % and 100 % replacement mixes respectively (Fig. 7).

3.2. Compressive strength of GPC

Compressive strength results of GPC at 3, 7 and 28 day curing for different PA content are depicted in Fig. 8. It can be noticed that as the RS replacement by PA increases, the compressive strength increases till 80 % replacement. Meanwhile, 4 to 5 MPa reduction in strength was noticed for 100 % replacement when compared with 80 % replacement. However, at all curing periods compressive strength of pond ash GPC has shown greater strength irrespective of replacement level compared to control mix i.e. 100 % RS geopolymer concrete. 80 % replacement of river sand by PA contributed for 26 % increase in strength compared to control mix. In addition to FA and GGBS, it appears that PA aggregates also participated in polymerization by leaching out silica and alumina from it. Y. Zaetang et al., have reported similar phenomena while using bottom ash as aggregate in geopolymers [13].

3.3. Flexural strength, splitting tensile strength and water permeability of GPC

Flexural strength, splitting tensile strength and water permeability tests were carried out for control and 100 % PA mixes, respective results are shown in Table 5. 100 % PA mixes exhibited greater flexural and splitting tensile strength at 28-day curing period compared to control mix. PA particles participation in polymerization reaction contributed for 29 % and 31 % improvement in flexural and splitting tensile strength respectively.

Water penetration depth in 100 % PA mix specimen was 12 mm, whereas in control mix it was observed as 20 mm. Water permeability test clearly indicates 100 % PA mix exhibited greater resistance to water penetration than control mix.



Figure 7. Slump in 100 % pond ash replacement a) without SP and b) with SP.

Table 4. Flow / slump of geopolymer concrete

Pond ash content	Slump/Flow values (mm)	T ₅₀₀ (Second)
0 %	690	3
20 %	650	4
40 %	590	49
60 %	545	62
80 %	200	-
80 % with SP	250	-
100 %	50	-
100 % with SP	185	-

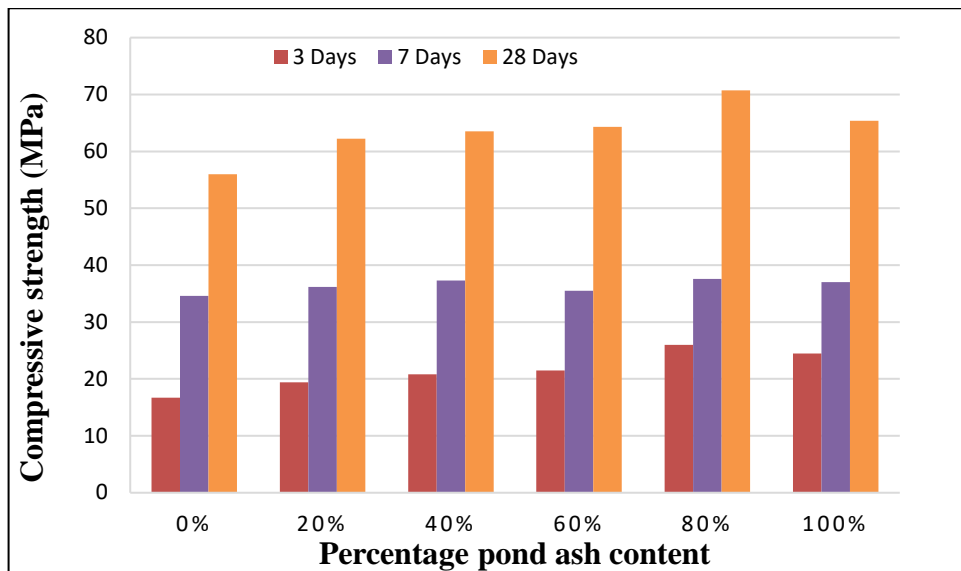


Figure 8. Compressive strength of GPC.

Table 5. Results of flexural strength, splitting tensile strength and water permeability

Pond Ash Content	28 days Flexural strength (MPa)	28 days Tensile strength (MPa)	Maximum water depth (mm)
0%	3.1	2.6	20
100%	4.0	3.4	12

3.4. Effect of R-SiO₂ on strength of GPC

R-SiO₂ in an aluminosilicates material is the non-crystalline, a fraction of total silica which have the ability to react with lime yielding increased contents of C-S-H gel [30]. R-SiO₂ in fly ash is the critical factor in strength contribution [31, 32]. R-SiO₂ values of GPC mixes used in this study are shown in Table 6. As RS which is inert when replaced by PA, the total R-SiO₂ content in GPC increases due to the contribution from PA as it contains about 20 % of R-SiO₂. Table 6 also shows ratio of R-SiO₂/Na₂O for different mixes used in the study. As can be seen increase in the ratio increases compressive strength of concrete and same can be noticed from Fig. 9. It is further observed that increase of ratio from 3.2 to 4.3 and then from 4.3 to 5.4 can yield an increase in strength of about total 14 MPa which is significant. Such an increase in strength can also be obtained by increasing either FA or GGBS in the binder. However, such an attempt would have cost implications. Earlier study on geopolymerization revealed, availability of more R-SiO₂ improves the geopolymerization reaction [33] and same results are observed in present study.

Table 6. R-SiO₂ values of GPC mixes

Mixes	R-SiO ₂ in Kg				Total R-SiO ₂ (Kg)	R-SiO ₂ /Na ₂ O	Compressive strength 28 days
	Fly ash	GGBS	Pond ash	Na ₂ SiO ₃			
PA0			0		157	3.2	56.0
PA20			27		184	3.8	62.2
PA40	98	27	54	32	211	4.3	63.5
PA60			82		239	4.9	64.3
PA80			109		266	5.4	70.7
PA100			133		290	5.9	65.4

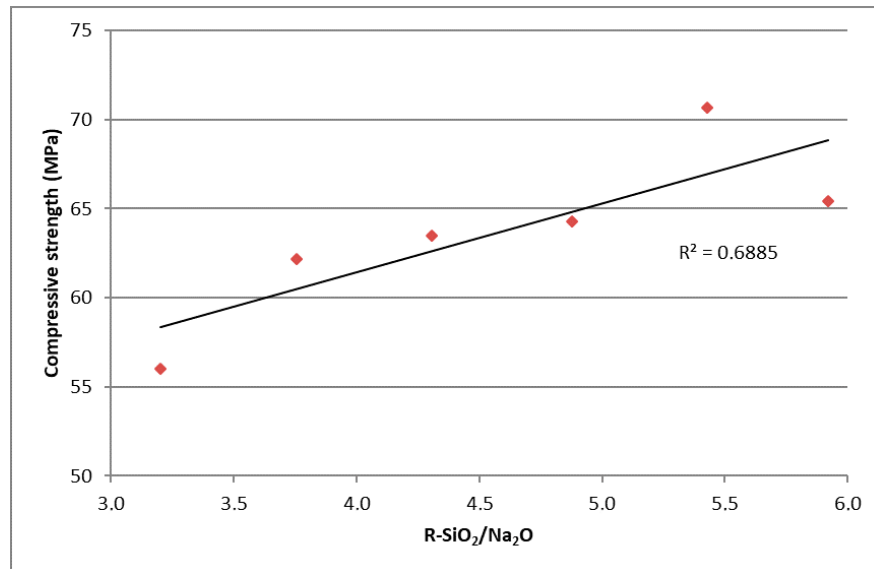


Figure 9. Effect of reactive silica on compressive strength of GPC.

3.5. Microstructure analysis of GPC

Earlier studies on microstructure analysis of fly ash based geopolymers revealed that, polymerization involves three stages a) chemical attack on fly ash surfaces and gradual development to form holes b) bi-directional alkaline attack in case of larger ash particles as sometimes their holes are filled with smaller particles c) product formation both inside and outside the spherical surface till particle of ash is completely consumed [34]. SEM images shown in Fig. 10 indicates that both FA and some portion of PA involved in polymerization and product formation both inside and outside the spherical surfaces as observed in studies done by Hassan. A et al. [34]. It is also observed that dense matrix in case of PA80 and PA100 samples along with PA and FA particles embedded in it.

Oxide compositions of GPC tested samples obtained through EDAX analysis are presented in Table 7. In geopolymerization $\text{SiO}_2/\text{Na}_2\text{O}$ and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ ratio indicates the availability of sodium ions for activation and dissolution of aluminosilicates [35]. In present study maximum strength is achieved for $\text{SiO}_2/\text{Na}_2\text{O}$ value 5.85 and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ value 0.78 which is corresponding to 80 % PA GPC. It is also noticed that $\text{SiO}_2/\text{Na}_2\text{O}$ value increased with introduction of PA in concrete indicating the dissolution of more aluminosilicates.

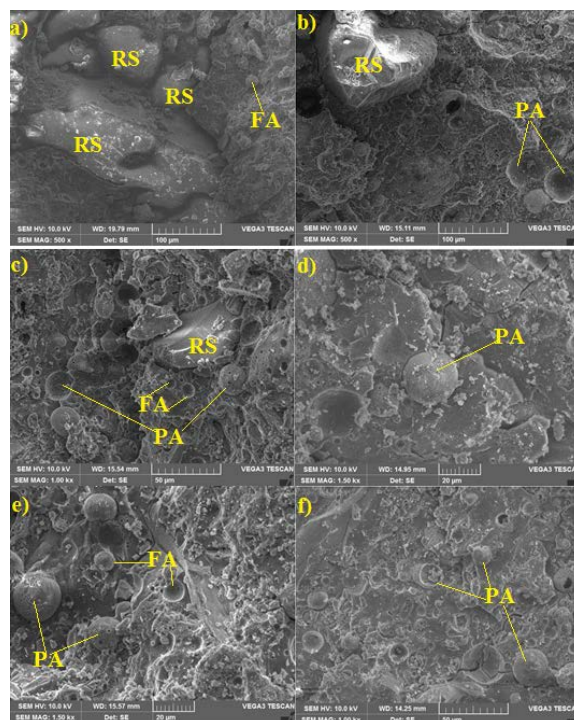
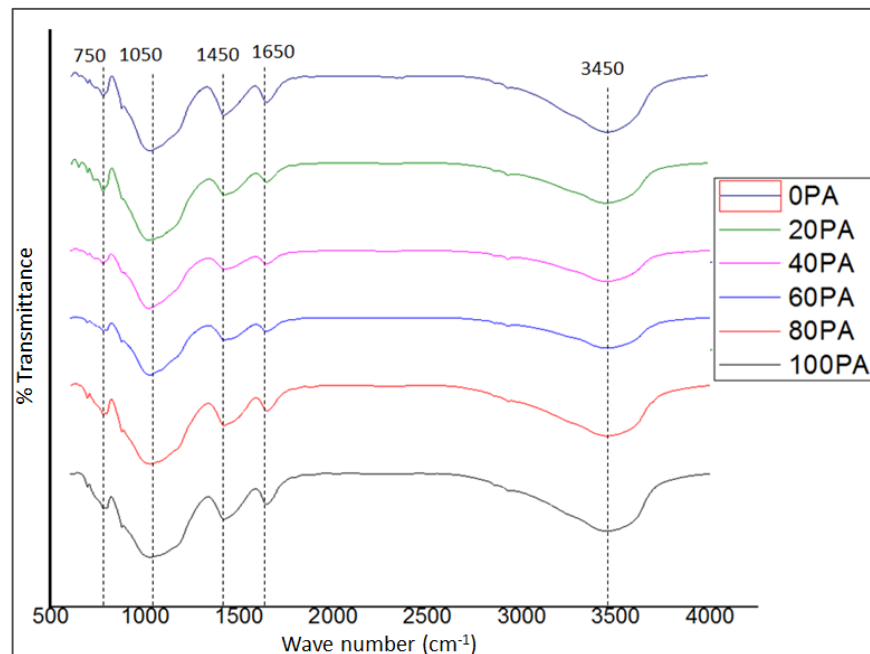


Figure 10: SEM images of 28 days tested samples
a) PA0 b) PA20 c) PA40 d) PA60 e) PA80 f) PA100.

Table 7. Oxide composition of GPC tested samples obtained by EDAX

Compound	PA0	PA20	PA40	PA60	PA80	PA100
Na ₂ O	12.64	11.65	11.07	10.68	9.55	10.03
MgO	3.55	3.33	3.81	3.83	3.20	3.20
Al ₂ O ₃	10.79	10.78	12.96	11.36	12.13	13.65
SiO ₂	58.82	59.26	57.9	58.17	55.96	59.47
K ₂ O	1.46	0.92	1.51	1.09	1.35	0.85
CaO	10.3	11.36	9.80	12.07	14.65	9.91
TiO ₂	1.38	1.07	1.40	1.58	1.53	1.24
Fe ₂ O ₃	1.07	1.62	1.55	1.23	1.63	1.66
SiO ₂ /Na ₂ O	4.65	5.08	5.23	5.44	5.85	5.92
Na ₂ O/Al ₂ O ₃	1.17	1.08	0.85	0.94	0.78	0.73
CaO/SiO ₂	0.17	0.19	0.16	0.2	0.26	0.16
SiO ₂ /Al ₂ O ₃	5.45	5.49	4.46	5.12	4.61	4.35

**Figure 11. FTIR graphs of GPC.**

FTIR analysis on GPC was conducted and respective graphs are presented in Fig. 11. The absorption bands corresponding to wave numbers 3450 cm⁻¹ and 1650 cm⁻¹ are indicative of vibrations of O-H and H-O-H bonds in water molecules [36]. The C-O asymmetric stretching is denoted by vibration bands located roughly around 1450 cm⁻¹ [37]. Spectra bands situated approximately around wavenumber 750 cm⁻¹ represents crystalline phases of quartz [36]. The Si-O-T (T is Si or Al) asymmetric vibration is indicated by bands of FTIR spectrum with wave numbers ranging around 1050 cm⁻¹ and degree of geopolymerization is determined from this bond [38]. Higher absorption at 1050 cm⁻¹ records high rate of geopolymerization [36] and in Fig. 11 it can be noticed that GPC mixes with PA have shown greater absorption at 1050 cm⁻¹ than GPC with RS. The absorption is significant beyond 60% replacement indicating the contribution of PA in polymerisation reaction.

3.6. Cost analysis of pond ash geopolymer concrete

Cost analysis of concrete clearly indicates; apart from alkaline solution fine aggregate play a major role in fixing the cost of concrete. Keeping quantity of solution constant, thereby replacing the RS with PA reduces cost of concrete per cubic metre to greater extent. As observed from Table 8, the cost of control mix made up of RS was around rupees (Rs) 9000/- and it got reduced to Rs 7000/- as PA replaces RS totally. It is conclusive that PA as fine aggregate in GPC can outperform RS in both cost and strength aspects.

Table 8. Cost of Pond Ash geopolymer blocks and concrete

Material	Unit cost (Rs/Kg)	0% PA		100% PA	
		Kg/m ³	Total Cost (Rs)	Kg/m ³	Total Cost (Rs)
Fly ash	2.5	330	825	330	825
GGBFS	2.5	83	206	83	206
Pond ash	1.0	–	–	660	660
River Sand	4.0	660	2640	–	–
Coarse Aggregate	1.0	990	990	990	990
Alkaline Solution (NaOH + Na ₂ SiO ₃)	15.0	268	4022	268	4022
Water	1.0	165	165	165	165
Total			8,848/-		6,868/-

4. Conclusion

PA was replaced to RS at different percentages in making geopolymer concrete and its mechanical properties were assessed. Based on the aforementioned studies following conclusions are made

1. Replacement of river sand by pond ash resulted in stiffening of concrete, especially at 80 and 100 % replacement. However, it can be restored by using suitable super plasticizers.

2. In addition to Fly Ash and GGBS, finer reactive pond ash particles also actively participated in geopolymerization process by leaching out silica and alumina. Thus compressive strength, flexural strength and split tensile strength of GPC with 80 % pond ash as fine aggregate are 26 %, 29 % and 31 % greater compared to concrete with river sand as aggregate. Pond Ash GPC unveiled greater resistance to water permeability than GPC made up of river sand.

3. Inclusion of reactive sand such as PA increases the ratio R-SiO₂/Na₂O from 3.2 to 5.4 in GPC mixes with up to 80 % replacement and contributes for higher compressive strength up to 14 MPa. SEM studies on GPC revealed that both FA and PA involved in reaction product formation resulting in dense matrix. Ratio SiO₂/Na₂O which is indicative of dissolution of aluminosilicates in GPC is higher i.e. 5.85 for GPC with 80 % PA as evidenced from EDAX results. FTIR curves confirmed participation of reactive silica of PA in geopolymerization by showing significant absorption rates.

Utilization of pond ash as fine aggregate provides value addition to GPC in aspects of strength and economics. In addition, environmental problems caused due to the land filling of pond ash will be alleviated. Keeping pond ash participation in polymerization process, research on GPC with pond ash as precursor needs to be explored.

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