

Strength and Durability Properties of Geopolymer Concrete made with Ground Granulated Blast furnace Slag and Black Rice Husk Ash

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Abstract

This paper presents an experimental study on the strength and durability properties of Geopolymer concrete prepared using Ground Granulated Blast Furnace Slag (GGBS) and Black Rice Husk Ash (BRHA). The Geopolymer concrete was prepared with GGBS as the primary binder instead of cement and BRHA was replaced with GGBS at various proportions such as 10%, 20% and 30%. The effect of curing temperature on the compressive strength of Geopolymer concrete was studied in addition to the flexural and split tensile strengths. Studies on the durability performance under Sorptivity, Rapid Chloride Permeability Test and Accelerated corrosion were also made. The test results show that the strength of Geopolymer concrete increases with increase in curing temperature. Addition of BRHA in Geopolymer concrete beyond 10% retards the strength development yet the strengths are well above the target for up to 20% replacement levels. At the same time, addition of BRHA significantly improves the durability with reduced sorptivity and chloride permeability when compared to the control concrete. Higher corrosion initiation and delayed cracking time were observed up to 20% BRHA replacement in Geopolymer concrete.

Keywords: *geopolymer concrete, Ground Granulated Blast furnace Slag (GGBS), Black Rice Husk Ash (BRHA), strength, durability, accelerated corrosion*

1. Introduction

Concrete is the most predominantly used construction material in the world. The basic ingredient of concrete that is Ordinary Portland Cement (OPC) is a major contributor of global warming. The yearly global cement production of 1.6 billion tons is responsible for about 7% of the total CO₂ emission into the atmosphere (Mehta, 2001). The raw materials required for cement production are non-renewable and depleting at a rapid rate. At the same time, a lot of industrial and agro wastes with inherent cementitious properties are produced abundantly but mostly dumped into landfills. Employing such byproducts as alternates for cement has multiple benefits including conservation of environment, sustainability of resources and solving the disposal problem of byproducts. Extensive researches are being carried out to assess the feasibility of utilizing industrial wastes as complete replacement for OPC and generating superior binders from the same. One such successful attempt is Geopolymer concrete which entirely eliminates the use of OPC in concrete production.

Davidovits coined the term ‘geo-polymer’ in 1978 to describe a family of mineral binders that can be produced from the polymerization reaction between an alkaline liquid and a source material containing silicon (Si) and aluminium (Al), which

possess a chemical composition similar to zeolites but exhibiting an amorphous microstructure (Davidovits, 2011). These zeolitic polymers possess high strength in comparison to OPC concrete (Jun and Oh, 2015). The polymerization process is a relatively quick chemical reaction involving alkaline liquids and Si-Al minerals. The end product is a three-dimensional polymeric ring structure comprising of Si-O-Al-O bonds, termed as ‘silicon oxo aluminates’ or ‘sialates’ in short. The general formula describing the chemical composition of the geopolymer is given by, $M_n[-(\text{SiO}_2)_z-\text{AlO}_2]_n \cdot w\text{H}_2\text{O}$, where, M is the alkaline component (like potassium or sodium), the symbol – represents the presence of a bond, n is the degree of polymerization and z is a number 1, 2, 3 etc. up to 32 (Davidovits, 1999). The properties of Geopolymers are mainly dependent on the source materials used (Albitar *et al.*, 2014).

Contrasting to OPC, the principal binders in Geopolymer concrete are not calcium-silicate-hydrates. Instead, an aluminosilicate polymeric gel formed by tetrahedrally-bonded silicon and aluminium with oxygen atoms shared in between acts the binder. However, several studies have revealed that, when sources like GGBS or Metakaolin containing high amounts of soluble calcium silicates are added, calcium dissolution occurs at low alkalinity resulting in the formation of a C-S-H gel in conjunction with the geopolymeric gel (Yip *et al.*, 2005; Yip *et*

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al., 2008; Bernal *et al.*, 2011; Ismail *et al.*, 2014). The geopolymeric gel remains the principal binder phase with small calcium precipitates scattered inside. This co-existence of two binder phases enhances the mechanical strength of the geopolymer concrete (Yip *et al.*, 2005).

Black Rice Husk Ash (BRHA) is an agro-industrial waste generated from rice milling industry. It is obtained by burning rice husk in the incinerator. The ash obtained as a result of this combustion process has a high content of unburnt carbon. Hence the use of BRHA as a construction material is very limited, even though it has high silica content about 90%. But several researchers including Chatweera and Lertwattanaruk (2011) and Piyaphanuwat and Asavapisit (2009) reported that the addition of BRHA in concrete has improved its durability property. In Geopolymer concrete, most of the research works have been made on Flyash based geopolymers and in this present study, industrial waste which is Ground Granulated Blast furnace Slag (GGBS) and the agro waste which is Black Rice Husk Ash (BRHA) are used as source materials for Geopolymer concrete. GGBS was kept as the base material in which BRHA was replaced at different percentages and its effect on the compressive, flexural and tensile strengths and other durability properties like sorptivity, chloride permeability and resistance to accelerated corrosion were studied.

2. Materials Used

2.1 Ground Granulated Blast Furnace Slag (GGBS) and Black Rice Husk Ash (BRHA)

GGBS was obtained from JSW cements limited, Bellari, India. BRHA was obtained from a local rice mill. It was finely grounded in a ball-mill for 30 minutes and passed through 75 μ sieve. The chemical composition of GGBS and BRHA given by SGS India Pvt. Ltd. Laboratory, Chennai, India are given in Table 1.

2.2 Aggregates

Natural river sand conforming to Zone II as per IS 383 (1987) with a fineness modulus of 2.45 and a specific gravity of 2.6 was used as fine aggregate. Crushed granite coarse aggregate conforming to IS: 383 (1987) was used. Coarse aggregate of size 20 mm and below with a specific gravity of 2.87 and fineness modulus of 8.14 was used.

Table 1. Properties of GGBS and BRHA

Sl No.	Property	GGBS	BRHA
1	SiO ₂	31.25%	93.96%
2	Al ₂ O ₃	14.06%	0.56%
3	Fe ₂ O ₃	2.80%	0.43%
4	CaO	33.75%	0.55%
5	MgO	7.03%	0.4%
6	Specific gravity	2.61	2.14
7	Blaine Fineness	4550 cm ² /g	5673 cm ² /g

Table 2. Mix Proportions

Quantities	Mix			
	GP	GPR1	GPR2	GPR3
GGBS (kg/m ³)	394	355	315	276
BRHA (kg/m ³)	0	39	79	118
Coarse Aggregate (kg/m ³)	1201	1201	1201	1201
Fine Aggregate (kg/m ³)	647	647	647	647
Sodium Hydroxide (kg/m ³)	45	45	45	45
Sodium Silicate (kg/m ³)	113	113	113	113
Super-plasticizer (% of binder)	1.5	1.5	1.5	1.5
Water (lit/m ³)	59	59	59	59

Note: GP - Control Concrete GPR1 - 10% BRHA
GPR2 - 20 % BRHA GPR3 - 30% BRHA

2.3 Chemicals

A mixture of Sodium hydroxide and Sodium Silicate was used as the alkaline solution. Commercial grade Sodium Hydroxide in pellets form (97%-100% purity) and Sodium silicate solution having 14.7% Na₂O, 29.4% SiO₂, and 55.6% water by mass were used. The molar concentration of Sodium Hydroxide was kept as 8M and the ratio of Sodium Silicate to Sodium Hydroxide was taken as 2.5. High-range water-reducing naphthalene based super plasticizer was used to improve the workability of fresh concrete. Also extra water about 15% of binder content was added to increase the workability of the concrete.

3. Mix Proportions

Since there are no codal provisions available for the mix design of geopolymer concrete, the density of Geopolymer concrete was assumed as 2400 Kg/m³ and other calculations were made based on the density of concrete as per the design given by Lloyd and Rangan (2010). The combined total volume occupied by the coarse and fine aggregates was assumed to be 77%. The alkaline liquid to binder ratio was taken as 0.40. Target strength of 40 MPa was fixed considering as a regular strength concrete. The mix proportions were taken as per Table. 2.

4. Preparation of Test Specimens

The materials for the mixes were weighed and first mixed in dry condition for 3-4 minutes. Then the alkaline solution which is a combination of Sodium hydroxide and Sodium silicate solution and super-plasticizer were added to the dry mix. Then some extra water about 15% by weight of the binder was added to improve the workability. The mixing was continued for about 6-8 minutes. After the mixing, the concrete was placed in steel mould by giving proper compaction. Precautions were taken to ensure uniform mixing of the ingredients. Two types of curing were used for the specimens to be tested for the compressive strength. One set of cubes was oven cured and the other set was cured under ambient condition. For oven curing, the cubes were placed in the oven at 60°C and 90°C respectively for 8 hours. Then the cubes were de-moulded and kept in the oven at the

respective same temperatures for another 8 hours. Then they were taken out and allowed to cure in the room temperature till the required day of testing. For the ambient curing, the cubes were kept in room temperature after casting and de-moulded after 1 day and further cured in the room temperature till the day of testing. The cubes were then tested at 3, 7 and 28 days from the day of casting. For all other tests, the specimens were adopted the oven curing procedure at 60°C.

5. Tests Conducted

5.1 Compressive Strength Test

The compressive strength of the geopolymer concrete was tested as per IS 516:1959. Cube specimens of size 150 mm were cast for each proportion and tested for their compressive strength at the ages of 3, 7 and 28 days. All the specimens were tested using Compression Testing Machine (CTM) of 2000 kN capacity under a uniform rate of loading of 140 kg/cm²/min until failure and the ultimate load at failure was taken to calculate the compressive strength.

5.2 Split Tensile Strength Test

The split tensile strength test was carried out as per IS 5816:1999. Cylindrical concrete specimens of size 150 mm diameter and 300 mm height were cast. The specimens were then tested for their splitting tensile strength using Universal Testing Machine (UTM) at the ages of 3, 7 and 28 days.

5.3 Flexural Strength Test

The flexural strength of the geopolymer concrete was carried out as per IS 516:1959. Beams of size 150 mm × 150 mm × 700 mm size were cast and then subjected to the flexural strength test using Universal Testing Machine (UTM) at the ages of 3, 7 and 28 days.

5.4 Sorptivity Test

The sorptivity test was done in accordance with ASTM C1585-04. Sorptivity is the measure of the capillary force exerted by the concrete pore structure which causes the fluids to be drawn inside the body of the concrete. Concrete slices of 100 mm diameter and 50 mm thickness were used for the test. The sides of the specimen were waxed and sealed with a plastic sheet and then the initial mass of the specimen was taken. The specimen was then kept in a tray with 2 to 5 mm of the depth being immersed in water. The mass of the specimen was then measured at 1 minute, 5 minutes, 10 minutes, 20 minutes, 30 minutes, 1 hour, 2 hours, 3 hours, 4 hours, 5 hours and 6 hours after taking out and blotting off the excess surface water. Sorptivity value was calculated using the formula $s = I/t^{1/2}$, where s is sorptivity in mm/min; t is the elapsed time in min; and I is the cumulative absorption given by $I = \Delta m/Ad$ where Δm is the increase in mass, A is the surface area of specimen through which water penetrates and d is the density of the medium, i.e. water. The cumulative absorption values were plotted against the

square root of time and sorptivity was given by the slope of the best fitting line of the plot.

5.5 Rapid Chloride Permeability Test

The Rapid Chloride Permeability Test (RCPT) was done in accordance with ASTM C1202-97. This test is the rapid measurement of the electrical conductance of the concrete with respect to its resistance against chloride ion penetration. Cylindrical specimens of size 100 mm diameter and 50 mm thick were used for this test after 28 days from the date of casting. A potential difference of 60 V was maintained across the ends of the specimen for about 6 hours. One end was mounted to a cell containing 3% sodium chloride solution and the other end was mounted to a cell containing 0.3 N sodium hydroxide solution. The current passing through the specimen was measured for every 30 minutes till 6 hours. The chloride contamination and temperature were also monitored. From the values of current and time, the total charge passed through the specimens was calculated in coulombs and related to the resistance of the specimen against chloride ion penetration. The greater the total charge passed, the more is the permeability of the specimen.

5.6 Accelerated Corrosion Test

The accelerated corrosion test was conducted on cylindrical specimens of 100 mm diameter and 200 mm height with a 14 mm diameter steel rod embedded centrally so that equal cover was maintained on all the sides. The specimens were oven cured at 60°C for 48 hours after casting and then further cured in room temperature for 28 days. The cylindrical specimens were then placed in individual containers each containing 4% sodium chloride solution. The level of the solution was maintained at 75 mm from the bottom of the specimen throughout the test. A stainless steel plate was placed in each container which acted as the cathode whereas the steel rod embedded in concrete served as the anode and they were connected to a constant DC supply of 6V so that the setup acted as an electrochemical cell. The voltage was maintained constantly throughout the test and current passing through each specimen was measured daily. At the instance of the appearance of first crack on each specimen, the specimens were stopped off the current supply and removed from the test setup respectively. Once the test was completed for all the specimens, a graph was plotted for the current passed in mA versus time in days to determine the corrosion initiation and propagation periods.

6. Results and Discussion

6.1 Compressive Strength

The compressive strength test results of the geopolymer concrete on 3, 7 and 28 days are shown in Table 3. Figs. 1 to 3 represent the variation of compressive strength of the specimens at different curing temperatures.

The effect of BRHA on the compressive strength of the Geopolymer concrete at different curing temperatures is discussed.

Geopolymer concrete made with 100% GGBS was kept as the control specimen. It is observed there was reduction in the compressive strength for all mixes with the addition of BRHA when cured under ambient condition. The addition of silica rich BRHA causes a rise in the unreactive silica content which further leads to inappropriate increase in the SiO_2/Al_2O_3 ratio of the geopolymer thus creating a negative effect on the compressive strength as reported by Duxon *et al.* (2005). At the same time, for the oven cured specimens both at 60°C and 90°C, at 10% BRHA replacement, the compressive strength was slightly higher and comparable with that of the control mix. This is due to the positive influence of curing temperature on the setting and hardening of geopolymers (Nazari *et al.*, 2011). The higher temperature enables greater dissolution of Si and Al ions and thus the formation of a stronger polymer chain as mentioned by Kusbiantoro *et al.* (2012). The other mixes showed reduced compressive strength than the control specimen. The addition of silica rich BRHA increased the SiO_2/Al_2O_3 ratio incongruously which inhibited the Geopolymer reactions and the subsequent gelation process, inspite of the temperature increase. Hence the decreasing trend in strength is observed for the increase in BRHA proportion. Another possible reason as observed by Kusbiantoro *et al.* (2012) could be due to the differences in the solubility degree between GGBS and BRHA, as the replacement of GGBS with BRHA remarkably decreases the dissolution and polycondensation rate of aluminosilicate precursors.

However, it can be seen that the BRHA added Geopolymer

concrete did exceed the target strength of 30 MPa and attained substantial 28th day strengths around 70.72 MPa for 10% addition and 51.46 MPa for 20% BRHA when cured at 60°C. When the curing temperature was increased to 90°C, there was only slight increase in the 28th day strength up to 72.34 MPa for 10% addition and 52.66 MPa for 20% addition. The ambient cured specimens obtained 28th day strengths of 48.44 MPa for 10% BRHA addition. The general trend of higher strengths can be attributed to the presence of additional calcium silicate hydrate gel along with the predominant aluminosilicate gel which has a compacting effect on the microstructure of geopolymer (Kumar *et al.*, 2009). The 30% BRHA added specimens did not achieve

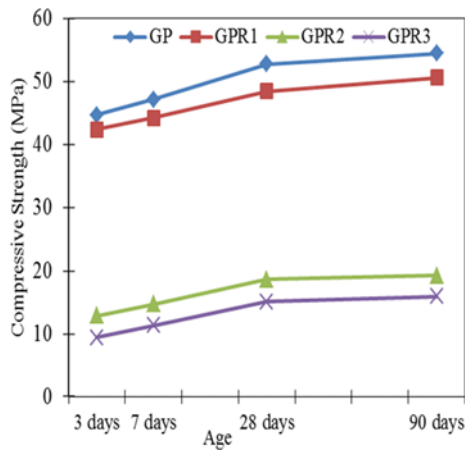


Fig. 1. Compressive Strength of Geopolymer Concrete cured at Ambient Temperature at Different Ages with Different BRHA Content

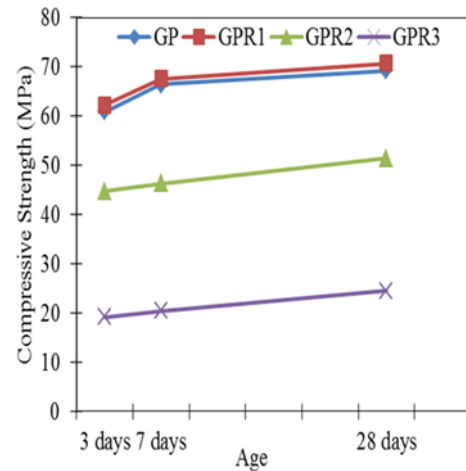


Fig. 2. Compressive Strength of Geopolymer Concrete cured at 60°C at Different Ages with Different BRHA Content

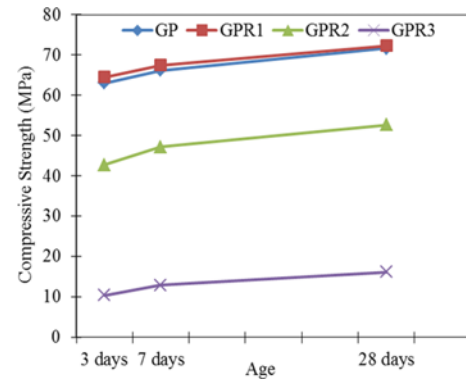


Fig. 3. Compressive Strength of Geopolymer Concrete cured at 90°C at Different Ages with Different BRHA Content

Table 3. Compressive Strength of Geopolymer Concrete at Different Curing Temperature

Mix	Compressive strength at different curing temperatures (MPa)								
	60°C			90°C			Ambient		
	3 days	7 days	28 days	3 days	7 days	28 days	3 days	7 days	28 days
GP	60.9	66.5	69.28	63.04	66.19	71.63	44.67	47.16	52.79
GPR1	62.3	67.6	70.72	64.53	67.45	72.34	42.38	44.25	48.44
GPR2	44.72	46.32	51.46	42.7	47.2	52.66	12.89	14.73	18.67
GPR3	19.24	20.48	24.52	10.4	12.9	16.1	9.41	11.33	15.11

any significant strength in all three curing conditions.

6.2 Split Tensile and Flexural Strengths

The results of splitting tensile and flexural strengths of the Geopolymer concrete at 3, 7 and 28 days are given in Table 4 and the variation of the split tensile strength and flexural strength for the various mixes are shown in Fig. 4 & Fig. 5 respectively.

When comparing the 10% BRHA replaced mix GPR1 with control mix GP, there is a slight improvement in both split tensile and flexural strengths. The heat curing of the specimens along with increased SiO₂/Al₂O₃ ratio and higher fineness of BRHA particles might have assisted the dissolution of ions and polycondensation mechanism of the geopolymer framework. Also, the co-existence of the secondary calcium silicate hydrate phase along with the primary geopolymer phase is another reason for the improvement in the mechanical strength as observed by Yip *et al.* (2005). But with further increase in BRHA content, the split tensile and flexural strength seem to get decreased. Similar results were reported by Jing Liu *et al.* (2014) while using palm oil fuel ash as a binder for Geopolymer

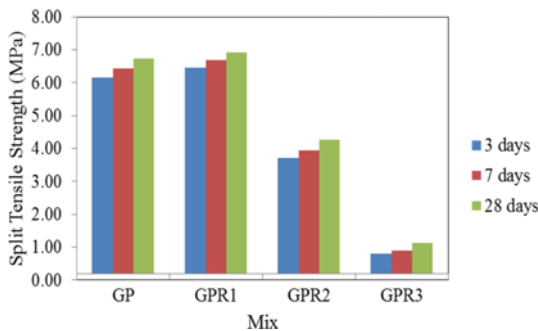


Fig. 4. Split Tensile Strength of Geopolymer Concrete

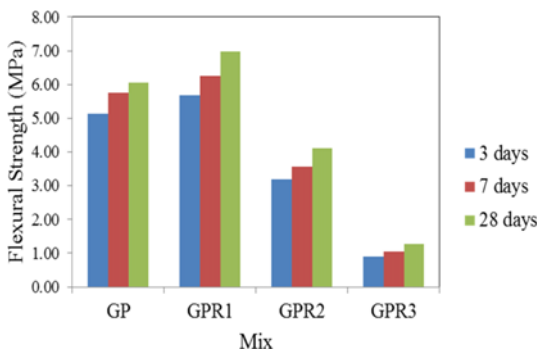


Fig. 5. Flexural Strength of Geopolymer Concrete

Table 4. Split Tensile and Flexural Strengths

	Split tensile strength (MPa)			Flexural strength (MPa)		
	3 days	7 days	28 days	3 days	7 days	28 days
GP	6.16	6.43	6.74	5.13	5.75	6.06
GPR1	6.47	6.69	6.92	5.69	6.26	6.98
GPR2	3.72	3.94	4.26	3.18	3.57	4.12
GPR3	0.81	0.89	1.12	0.91	1.05	1.27

concrete. BRHA particles possess a different silicate structure and when the SiO₂/Al₂O₃ ratio exceeds very high, the kinetics of the polymerization mechanism is inhibited due to the difference in solubility of GGBS and BRHA and thus the production rate of geopolymer gel is reduced as deduced by Kusbiantoro *et al.* (2012).

6.3 Sorptivity

The absorption values plotted against square roots of time are shown in Fig. 6. The slope of the absorption curve for each mix gives the corresponding sorptivity value and the variation of sorptivity for the different mixes of geopolymer concrete is shown in Fig. 7.

It can be found that the addition of BRHA decreases the sorptivity to a minimum value at 10% replacement. The fine BRHA particles produce a micro-filler effect by arresting the pores and result in a more compact concrete structure thereby reducing the liquid penetration property as inferred by Sampaio *et al.* (2003). The sorptivity value is slightly higher for 30% replacement of BRHA. It could be that the excessive BRHA content has affected the structural compactability of geopolymer concrete by hindering the development of a denser aluminosilicate polymer gel. This might have enhanced the capillary suction. Similar trends were reported for RHA substituted OPC concrete by Oyekan and Kamiyo (2011) and Abdelalim *et al.* (2004).

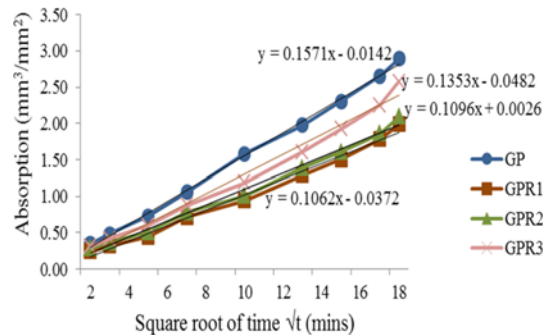


Fig. 6. Absorption Vs Square Root of Time

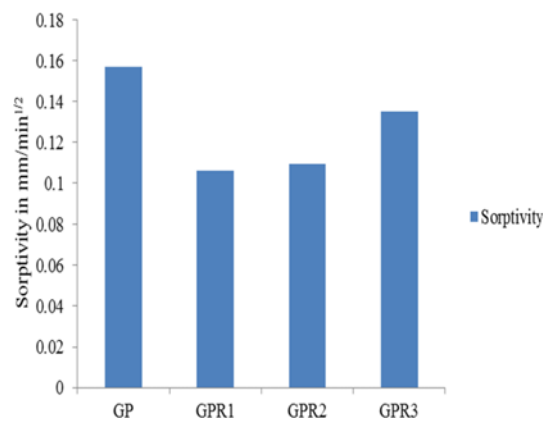


Fig. 7. Sorptivity of Geopolymer Concrete

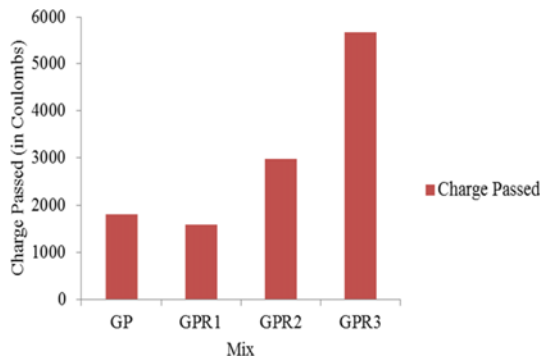


Fig. 8. RCPT of Geopolymer Concrete

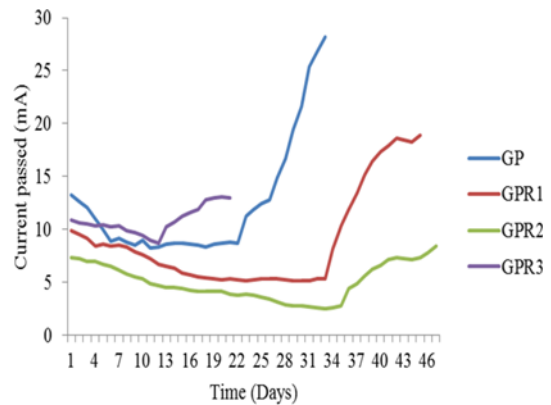


Fig. 9. Variation of Current Passed with time

6.4 Rapid Chloride Permeability Test (RCPT)

The test results of the Rapid Chloride Permeability at 28 days for the Geopolymer concrete mixes are given in Fig. 8.

The total charges passed through the control, 10%, 20% and 30% BRHA replaced specimens in terms of Coulombs are 1813.2, 1597.5, 2977.5 and 5675.1 respectively. There is a reduction in the charge passed for 10% BRHA replaced specimen when compared to the control specimen. Then it tends to increase with further addition of BRHA. As per the classification given by ASTM C1202, the charges passed through the specimens signify the chloride penetrability as low for control as well as 10% BRHA replaced specimens. It is moderate for 20% and high for 30% BRHA replacement levels respectively. One of the key factors influencing the concrete permeability is the pore structure. BRHA replacement at a suitable level reduces the chloride ion penetration of geopolymer concrete which is characterized by the micro filler effect of the fine BRHA particles. However, BRHA being silica rich, its excessive addition leads to an inappropriate $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio. This affects the dissolution mechanism of Si and Al ions and the subsequent rate of polycondensation leading to incomplete aluminosilicate geopolymer matrix. The structural compactability of resulting Geopolymer may not be dense which is believed to have caused higher ion transport through the concrete.

6.5 Accelerated Corrosion

The variation of current passed through the specimens in mA with time in days is shown in Fig. 9.

It can be seen that the current profile starts steadily at the beginning and then shows a declination with time. This is due to the formation of a passive layer by the Geopolymer matrix which delays the effect of the applied voltage to accelerate the corrosion process. This is confirmed by Olivia and Nikraz (2011). After a certain period, there is a sudden increase in the current. This particular point of time is called as the corrosion initiation period which signifies the onset of corrosion process in the concrete. The migration of chloride ions increases with time and when it reaches the critical chloride content of the steel bar used, de-passivation occurs and it gets reflected by a sudden increase in current. The initiation period for the control concrete is 23 days

and the specimen showed first crack at the 33rd day. For 10% BRHA replacement, the initiation period is increased to 34 days and first crack was observed at the 45th day. The initiation period is 36 days for 20% BRHA replacement and it showed first crack only on the 47th day. For 30% BRHA replacement, the initiation period is reduced to 13 days and the specimen showed its first crack at the 23rd day. It is evident from the results that suitable addition of BRHA increases the corrosion initiation period and delays the eventual cracking. The fine BRHA particles facilitate the effective dissolution of Silica and Alumina ions and formation of a stronger Geopolymer matrix. Subsequently they account for the development of a more resistive passive layer against the attack of chloride ions. However excessive BRHA could have a negative effect on corrosion prevention as it retards the development of a stronger Geopolymer matrix and thus the passive layer around reinforcement is weakened as a result.

7. Conclusions

The experimental results show that it is possible to produce geopolymer concrete possessing substantial strength and durability using GGBS and BRHA. The following conclusions can be further drawn from the present study.

1. Oven cured specimens showed higher compressive strength than the ambient cured specimens. While comparing oven curing at a temperature of 60°C and ambient curing, the strength increase at 28 days was 45% for 10% BRHA replacement and nearly three times for 20% BRHA replacement. The 10% BRHA replaced specimens still exceeded the target strength of 40 MPa under ambient curing. Further increase in curing temperature to 90°C had only a moderate increase in the compressive strength. Hence oven curing at 60°C could be optimum for the Geopolymer concrete.
2. Addition of BRHA beyond 10% had a retarding effect on the compressive strength. Although upto 20% replacement, the target compressive strength was surpassed and strength as high as 51 MPa was reached at 28 days. The split tensile and flexural strengths showed a trend similar to that of the compressive strength with respect BRHA proportion.

3. The test results showed that there was a decrease in sorptivity about 47% for 10% BRHA replacement and 43% for 20% BRHA replacement in comparison with the control specimens.
4. The Geopolymer concrete specimens showed good resistance to chloride permeability. The chloride permeability level was low for 10% BRHA and it was moderate for 20% BRHA.
5. The incorporation of BRHA in Geopolymer concrete increases its corrosion resistance. The corrosion initiation period was 34 days for 10% BRHA and 36 days for 20% BRHA while it was only 23 days for the control specimen. The fine BRHA particles account for a greater dissolution of silica and alumina ions and a stronger passive layer formation.
6. Addition of BRHA beyond 20% is not beneficial in Geopolymer concrete. The 30% BRHA replaced specimens neither achieved significant strength nor proved durable.
7. The strength results show that the optimum proportion of BRHA that can be used in Geopolymer concrete is 10% in case of ambient curing and 20% in case of oven curing, considering target strength of M30. From the durability studies, it can be seen that the Geopolymer concrete performed remarkably well in sorptivity, chloride penetration and corrosion resistance upto 20% BRHA replacement. These results are very important in the development of such innovative concretes which completely omit OPC in their production. It aids the utilization of alternate materials like GGBS and BRHA to produce binders. Since these materials are essentially industrial by-products, it also means a solution to their disposal problem.

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