

CORROSION STUDIES ON AGRICULTURAL AND INDUSTRIAL RESIDUE BASED GEOPOLYMER CONCRETE USING ELECTRICAL ANALYSER

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Abstract: Geopolymer Concrete (GPC) is a novel concrete which has evolved in recent decades. It uses industrial waste products like fly ash (FA), ground granulated blast slag (GGBS), Rice husk ash (RHA), micro-silica, and red mud etc., from industries, with alkaline liquids to replace cement in concrete by 100%, thereby developing an eco-friendly and sustainable construction material and simultaneously reducing waste disposal problem of fly ash and rice husk ash. Agricultural residue and industrial residue based GPC not only possesses excellent mechanical properties it also has very good durability properties. The durability test conducted in this study using Electrical Analyser. This paper presents the corrosion effect of partial replacements of fly ash with rice husk ash on the properties of geopolymer concrete. The design mix ratio 1:1.4:3.2 chosen for investigation were Geopolymer concrete containing 50% RHA and 50% FA. Various synthesis parameters like alkaline liquid to source material ratio, molarity of NaOH solution, sodium silicate to sodium hydroxide ratio were kept at their optimum values of 0.40, 0.45, 0.50 and 0.55 with 10M & 12M, and 2.5 respectively. In this research study, four important corrosion tests are Open circuit potential test, Impedance test, LPR Sweep test and Custom Sweep test were conducted.

Key words: Fly ash, Rice husk ash, Residue, Alkaline liquid, Molarity

1. INTRODUCTION

1.1 Rice Husk Ash

Rice husk is an agricultural residue which accounts for 21.3% of the 721 million tons of rice produced annually worldwide. The produced partially burnt husk from the milling plants when used as a fuel also contributes to pollution and efforts are being made to overcome this environmental issue by utilizing this material as a supplementary cementing material. The chemical composition of rice husk is found to vary from one sample to another due to the differences in the type of paddy, crop year, climate and geographical conditions.

Burning the husk under controlled temperature below 800 °C can produce ash with silica mainly in amorphous form. Recently, Nair et al.(1) reported an investigation on the pozzolanic activity of RHA by using various techniques in order to verify the effect of incineration temperature and burning duration. He stated that the samples burnt at 500 or 700 °C and burned for more than 12 hours produced ashes with high reactivity with no significant amount of crystalline material. The short burning durations (25 – 360 minutes) resulted in high carbon content for the produced RHA even with high incinerating temperatures of 500 to 700 °C. The Rice Husk ash collected from Jayankondam, Ariyalur district, Tamilnadu paddy field. The chemical composition of Rice Husk ash is available in Table 1.

Table 1 Chemical Compositions of RHA.

Oxide Composition (% by mass)	RHA
SiO ₂	87.41
Al ₂ O ₃	0.62
Fe ₂ O ₃	0.83
CaO	0.79
MgO	0.53
Na ₂ O ₃	0.21
K ₂ O	3.18
LOI	6.03

1.2 Fly Ash

Fly ash is Industrial residue low-calcium (ASTM Class F) dry fly ash and it is collected from Mettur Thermal Power Station, Mettur was used. The fly ash has been stacked in an open space at Mettur as shown in Fig.1. Low-calcium fly ash could be successfully used to manufacture geopolymer concrete when the silicon and aluminum oxides constituted about 80 percent by mass, with the Si-to-Al ratio of about 2. The chemical composition of Fly ash is given in Table 2 and the constituents of geopolymer concrete materials are mentioned in Table 3.

Table 2 Chemical composition of fly ash

Sl.No.	Compound	Percentage (mass)
1.	SiO ₂	52.54
2.	Al ₂ O ₃	26.74
3.	Fe ₂ O ₃	11.12
4.	CaO	1.28
5.	Na ₂ O	0.47
6.	K ₂ O	0.82
7.	TiO ₂	1.57
8.	MgO	0.87
9.	P ₂ O ₅	1.53
10.	SO ₃	1.70
11.	*LoI	1.36

**LoI- Loss on Ignition*



Fig. 1 Fly ash disposal Yard

Table 3 Constituents of Geopolymer concrete (Per 1m³)

Sl. No	Mix ratio	Fly ash (kg)	RH ash (kg)	Fine Agg. (kg)	Coarse Agg. (kg)	NaOH solution		Na ₂ SiO ₃ (kg)	Na ₂ SiO ₃ / NaOH	AAS /FA
						Mass (kg)	Molar			
1.	1:1.4:3.2	221	229	635.8	1454.6	52.0	10M	130.0	2.5	0.40
2.						58.5		146.25		0.45
3.						65.0		162.50		0.50
4.						71.5		178.75		0.55

2. DURABILITY TESTS CONDUCTED USING ELECTRICAL ANALYSER

Corrosion mostly affects steel and concrete structures. Using Electrical analyser supplied by ACM instruments, UK (Fig. 2), all types of corrosion tests can be conducted. In this research study, four important corrosion tests were conducted.

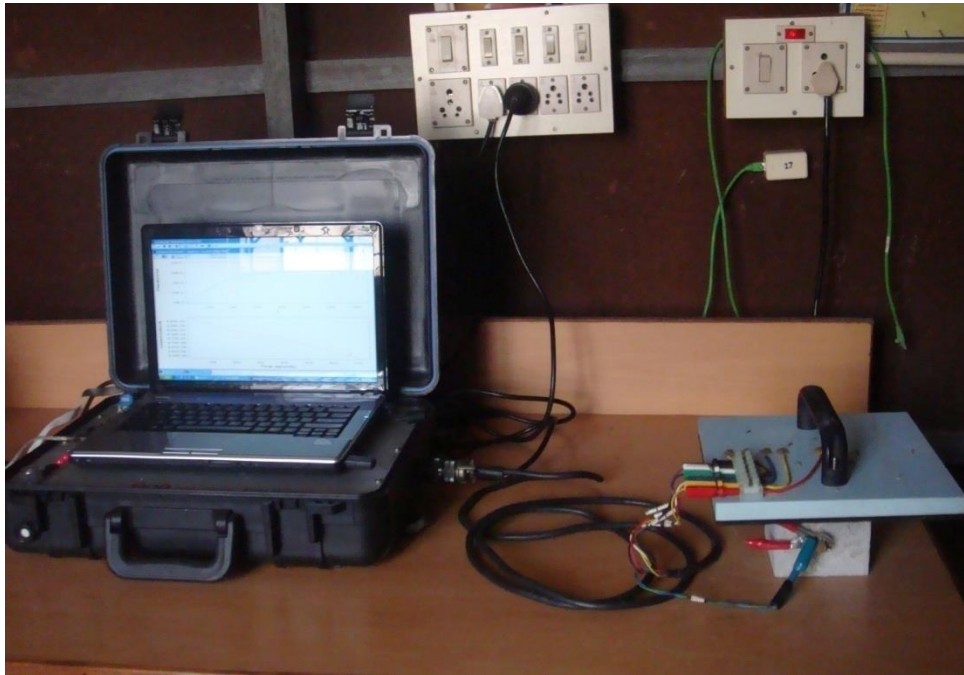


Fig. 2 Electrical Analyser

The geopolymer concrete (Fly ash and Rice husk ash) specimens were cast using different molar solutions of NaOH such as 10M and 12M and various Alkali Activator Solution to Fly Ash(FA) and Rice Husk Ash(RHA) ratios say 0.40, 0.45, 0.50 and 0.55. The normal concrete specimens were also cast with Water to cement ratio 0.40 and the design mix ratio adopted was 1:1.4:3.2 for both the concrete specimens. The following tests were conducted on concrete specimens:

- (i) Open circuit potential test
- (ii) Impedance test
- (iii) LPR Sweep test
- (iv) Custom Sweep test

2.1 Details of specimen

In the experimental study the geopolymer concrete (FA &RHA) specimens and normal concrete specimens were prepared for the durability and corrosion studies as given below:

- i) Cube: 100x100x100 mm with centrally located 8 mm diameter bars
- ii) Cylinder: 100x200 mm with centrally located 8 mm diameter bars

2.2 Preparation of specimen

Initially the dry weights of fly ash, rice husk ash, coarse aggregate and sand were measured as required. The Alkali-Activator Solution was prepared separately according to its NaOH concentration. The fly ash, rice husk ash, coarse aggregate and sand were mixed thoroughly in dry condition and the AAS was added gradually to the mix so that it would not get de-bonded. Then the cleaned mould was placed over the base

plate. The concrete mix was placed inside the mould and it was laid on the vibrator and the concrete was allowed to settle with suitable compaction then the 8 mm dia steel rods were centrally located. Thus, the well-shaped geopolymer concrete cube and cylinder specimen was prepared as shown in Fig.3



Fig. 3 Cylinder and cube specimen with the steel insertion

3. OPEN CIRCUIT POTENTIAL TEST

For conducting open circuit potential test, concrete cube specimens of size 100 mm x 100 mm x100 mm had been cast with 8 mm diameter torsteel rod inserted at the centre up to a depth of 70 mm. Geopolymer concrete with different AAS/FA ratios had been considered along with normal concrete for this test. The schematic diagram of Electrical analyser connection is shown in Fig. 4. The cube specimens were placed in the Electrical analyser, the rebar was connected to working electrode-1 and the reference electrode and also auxiliary electrode directly connected to Guard ring was placed over the specimen.

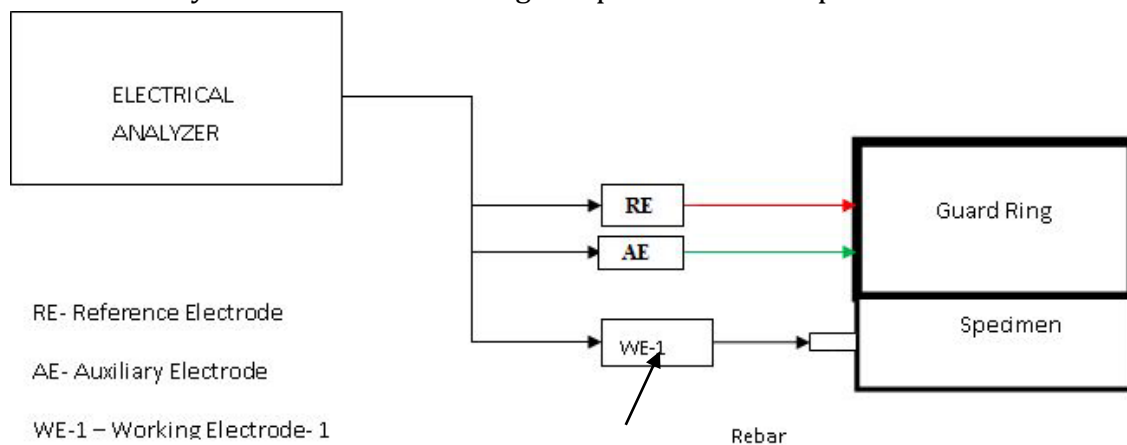


Fig. 4 Schematic Diagram of Electrical Analyser

The function of Working Electrode (WE-1) is to pass the voltage in to the rebar embedded in concrete. The Auxiliary Electrode (AE), converts the passed voltage in to the required current and the current spreads the entire specimen. The Reference Electrode using the converted current, locate the corrosion/weak points available in the specimen. The output is given as a graph showing time Vs potential.

From the graph, voltage ratio has been calculated by the machine. The experiments were conducted for Geopolymer concrete specimens and cement concrete specimens using Electrical analyser.

3.1 Connections of specimen

The cube specimens are placed in the Electrical analyser, the rebar is connected to working electrode -1 and the reference electrode and also auxiliary electrode are directly connected to Guard ring and when the analyser is switched on the process starts. The electrode connections are indicated in Fig. 5

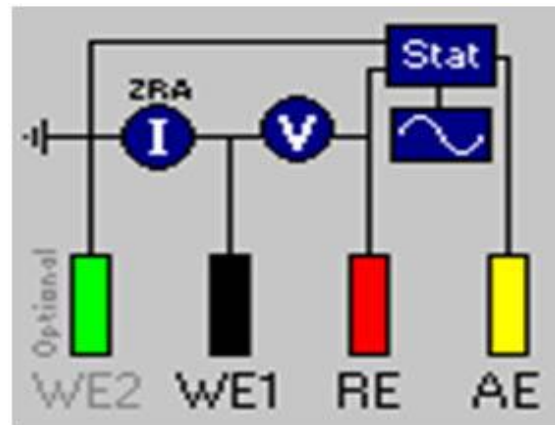
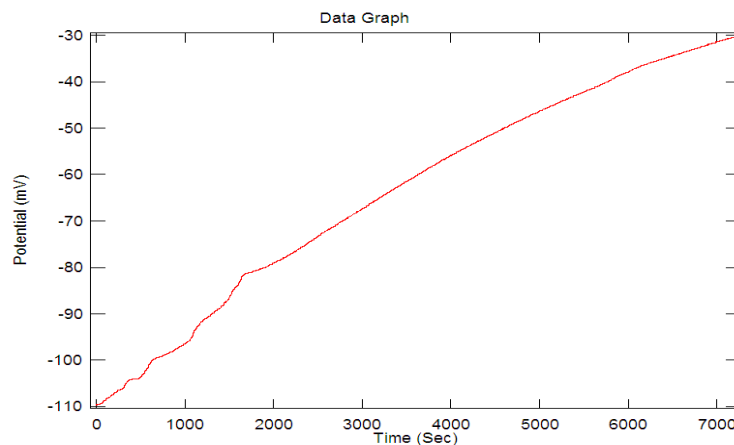


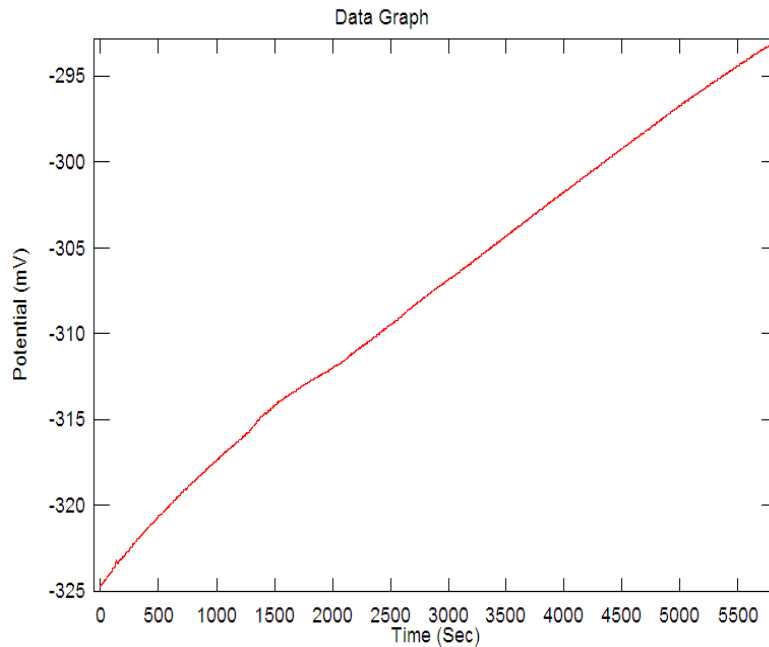
Fig. 5 Electrode connections

The experiments were conducted on geopolymer concrete specimens and cement concrete specimens using Electrical analyser. The Open circuit potential graphical representations are given in Fig.6 and 7.



Delta V (mV) - 0.0285, Average V (mV) - 8.7381

Fig. 6 Open Circuit Potential – Geopolymer Concrete



Delta V (mV) – 0.1980, Average V (mV)-69.86

Fig.7 Open Circuit Potential – Normal Concrete

There is not much variation in the graphical representations of normal concrete and geopolymer concrete. The delta voltage and average voltages are directly given by the Electrical Analyser. The open circuit potential measurement results are given in Table 4. The tendency of any metal to react with an environment is indicated by the potential it develops in contact with the environment.

Table 4 Open circuit potential test results

Sl.No.	Descriptio n	AAS/FA	Voltage ratio	
			10M	12M
1.	GPC	0.40	-0.0832	-0.1013
2.	GPC	0.45	-0.0905	-0.1056
3.	GPC	0.50	-0.0963	-0.1080
4.	GPC	0.55	-0.0992	-0.1167
5.	NC	0.4	- 0.1155	

NC- Normal Concrete, GPC- Geopolymer Concrete, Sr – Alkali Activator Solution/Fly ash

In reinforced concrete structures, concrete acts as an electrolyte and the reinforcement will develop a potential depending on the concrete environment, which may vary from place to place. The principle involved in this technique is essentially measurement of corrosion potential of rebar with respect to a standard reference electrode, such as saturated calomel electrode (SCE), copper/copper sulfate electrode (CSE), silver/ silver chloride electrode etc. The detection and measurement of corrosion in concrete structures are essential. Although there are several methods for the diagnosis, detection and measurement of corrosion in reinforcing steel, there is no consensus regarding which method assesses corrosion levels in reinforced concrete structures most accurately. Monitoring of open circuit potential (OCP) is the most typical procedure to the routine inspection of reinforced concrete structures.

The result indicates that the voltage ratio is less in geopolymer concrete. The 10M specimens give low corrosion risk when compared to other specimens as per ASTM guide lines. For the geopolymer concrete for 12M voltage ratio indicates moderate corrosion risk and also normal concrete specimens moderate corrosion risk.

4. IMPEDANCE TEST

The Impedance test method measures the corrosion rates using I_{corr} values and also determines the corrosion points available inside the specimens. The schematic diagram and the electrode connections of Electrical analyser are available in Figs.2 and 5. Impedance Z is the ratio of A.C. voltage to A.C. current. An alternating voltage of about 10 to 20 mV is applied to the rebar and the resultant current and phase angle are measured for various frequencies. The response to an A.C. input is a complex impedance that has both real (resistive) and imaginary (capacitive or inductive). The component Z' and Z'' as shown in Fig.8.

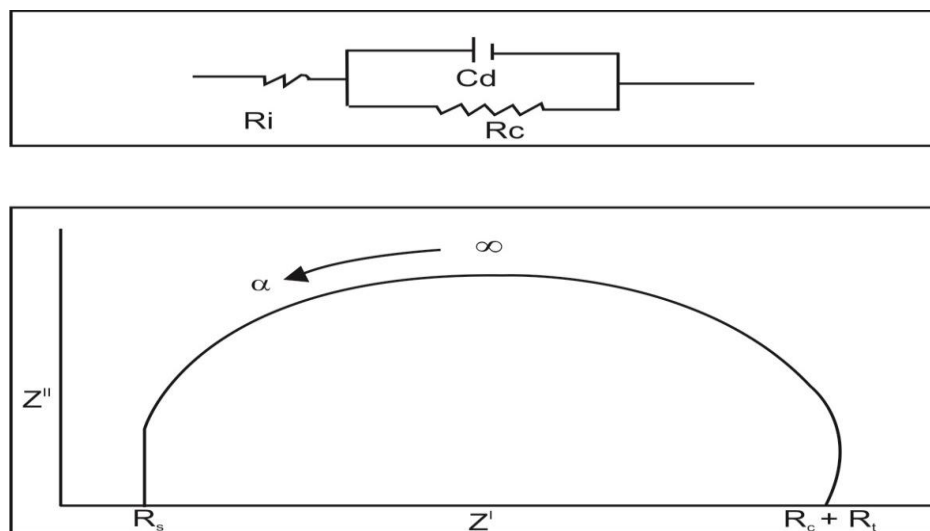


Fig.8 Nyquist Plot for steel in concrete

4.1 Calculation of Corrosion rate

The corrosion rate is calculated by the machine using the following formulae:

$$I_{corr} = B/R_p \quad \text{-----} \quad (1)$$

$$\text{Corrosion rate} = 0.129 \times I_{corr} \times EW / dA \quad \text{-----} \quad (2)$$

B is the Stern–Geary constant, Stern–Geary range of 10–30 mV

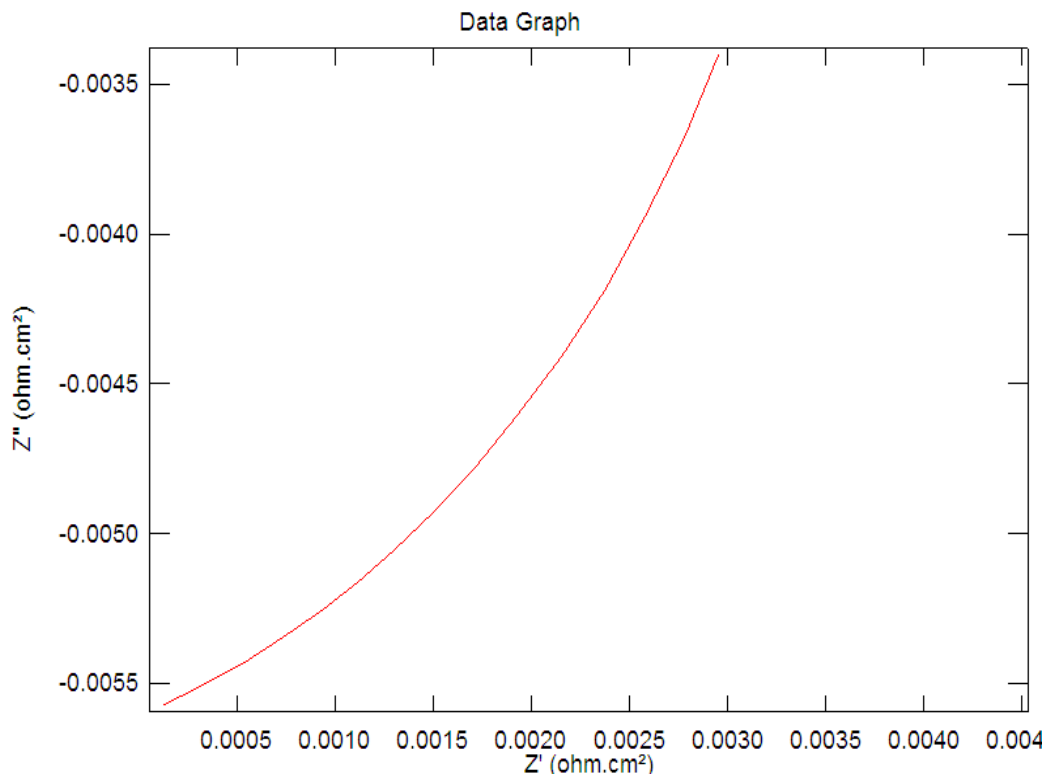
R_p = Polarization Resistance

$E.W$ =equivalent weight of the corroding species in (g).

A =exposed surface area of the reinforcing steel,

d = the density of the reinforcing steel, in g/cm³

The Impedance test graphical representations are shown in Fig. 9 and the impedance test results are shown in Table 5.



Corrosion Rate (mm/year)-0.04628, Corrosion rate (mils/yr) – 1.8215808

Fig.9 Impedance test - Geopolymer Concrete

Table 5 Impedance test Results

Sl.No.	Description	AAS/FA	Corrosion rate (mm/year)	
			10M	12M
1.	GPC	0.40	2.9246	3.345 6
2.	GPC	0.45	3.5642	3.711 2
3.	GPC	0.50	3.6123	3.854 2
4.	GPC	0.55	3.9876	4.213 5
5.	NC	W/C=0.40	8.7650	

NC- Normal Concrete, GPC- Geopolymer Concrete, Sr – Alkali Activator Solution/Fly ash

For the geopolymer concrete (Fly ash and Rice husk ash) specimens 10M and 12M specimens corrosion rates ranges between 1mm/year to 3mm/year and so it is termed as low risk corrosion as detailed in ASTM D2776 and G59 -2009 (2). For the normal concrete specimens, corrosion rate becomes 7 mm/year to 10 mm/year and so it is called moderate risk corrosion.

5. LPR SWEEP TEST

The Linear polarization resistance sweep method measures the instantaneous corrosion rates as compared to other methods by which metal loss is measured over a finite period of time. Instantaneous means that each reading on the instrument can be translated directly into corrosion rate.

The experiment can be completed in a matter of minutes and the small polarization from the corrosion potential does not disturb the system. This permits rapid rate measurements ASTM G102-2010 (3) and it can be used to monitor corrosion rate in various process streams. The LPR data enables a more detailed assessment of the structural condition and is a major tool in deciding the optimum remedial strategy to be adopted. It is thus imperative that the LPR measurements obtained are accurate. In LPR measurements the reinforcing steel is perturbed by a small amount from its equilibrium potential. The LPR sweep test results are given in Table 6.

Table 6 LPR Sweep test results

Sl.No.	Description	AAS/FA	Corrosion rate (mm/year)	
			10M	12M
1.	GPC	0.40	0.8543	1.2712
2.	GPC	0.45	1.1340	1.4309
3.	GPC	0.50	1.1856	1.6352
4.	GPC	0.55	1.3119	1.8257
5.	NC	W/C =0.40	5.5481	

NC- Normal Concrete, GPC- Geopolymer Concrete, Sr – Alkali Activator Solution/Fly ash

For the geopolymer concrete specimens for 10M and 12M specimens So it is low risk corrosion and corrosion rate, lies between 1 mm/year to 3 mm/year and so it is medium risk corrosion. ASTM G102-2010. The normal concrete specimens corrosion rate lies between 3 mm/year to 7 mm/year. Hence it is moderate risk corrosion. The corrosion rates for geopolymer concrete gave good results compared to normal concrete and hence the geopolymer concrete can be utilized in steel and concrete structures.

6. CUSTOM SWEEP TEST

The Custom Sweep method or Tafel Extrapolation Method measures the instantaneous corrosion rates. This technique uses data obtained from cathodic and anodic polarization measurements. Cathodic data are preferred, since these are easier to measure experimentally. In this method, the total anodic and cathodic polarization curves corresponding to hydrogen evolution and metal dissolution are superimposed as dotted lines. It can be observed that at relatively high applied current densities and the applied current density corresponding to hydrogen evolution have become virtually identical. To determine the intercept corrosion rate from such polarization measurements, the Tafel region is extrapolated to the corrosion potential. The i_{corr} values are calculated by the Electrical Analyser using the following equations:

$$i_{corr} = \frac{\alpha\beta}{2.3(\alpha + \beta)} \frac{\Delta i}{\Delta E} \quad \text{-----} \quad (3)$$

$$i_{corr} = \frac{\alpha\beta}{2.3(\alpha + \beta)} \frac{1}{R_p} \quad \text{-----} \quad (4)$$

where $\Delta E/\Delta i$ = slope of the polarization curve = Polarization Resistance = R_p .
 α and β = Cathodic and Anodic Tafel constants.

The results of custom sweep test are given in Table 7

Table 7 Custom sweep test results

Sl.No.	Description	AAS/FA	Intercept Corrosion rate (mm/year)	
			10M	12M
1.	GPC	0.40	0.9368	1.4110
2.	GPC	0.45	1.1047	1.8905
3.	GPC	0.50	1.1362	2.1098
4.	GPC	0.55	1.3469	2.8653
5.	CC	W/C=0.40	4.9524	

NC- Normal Concrete, GPC- Geopolymer Concrete, AAS/F.A – Alkali Activator Solution / Fly ash

The Fly ash and Rice husk ash based geopolymer concrete specimens 10M and 12M intercept corrosion rate lies between 1 mm/year to 3 mm/year as detailed in ASTM G102-2010, so it is termed as medium risk corrosion. The normal concrete specimens corrosion rate lies between 3mm/year to 7mm/year and so it is moderate risk corrosion. The intercept corrosion rates for geopolymer concrete gave good results compared to normal concrete.

7. CONCLUSIONS

Fly ash and Rice husk ash is equally (Each 50%) added and found to be superior to other supplementary materials like slag, and silica fume. Flyash and Rice husk ash based geopolymer concrete used in this study is efficient as a pozzolanic material. Due to low specific gravity of RHA(4) which leads to reduction in mass per unit volume, thus adding it reduces the dead load on the structure. Used of Fly ash and Rice husk ash helps in reducing the environment pollution during the disposal of excess Fly ash and Rice husk ash. Cement is costly material, so the partial replacements of these materials by Rice husk ash (5) reduces the cost of concrete. Based on the results presented above, the following conclusions can be drawn:-

1. In Open Circuit Potential test, the geopolymer concrete (Fly ash and Rice husk ash) 10M specimens give low corrosion risk when compared to other specimens. For the geopolymer concrete for 12M voltage ratio indicates moderate corrosion risk.
2. During Impedance test, the geopolymer concrete (Fly ash and Rice husk ash) specimens 10M and 12M specimens corrosion rates ranges between 1mm/year to 3mm/year and so it is termed as low risk corrosion.
3. The Linear polarization resistance (LPR) sweep method measures the instantaneous corrosion rates

in the geopolymer concrete (Fly ash and Rice husk ash) specimens 10M and 12M specimens corrosion rates ranges between 1mm/year to 3mm/year and so it is termed as low risk corrosion.

4. The Fly ash and Rice husk ash based geopolymer concrete specimens 10M and 12M intercept corrosion rate lies between 1 mm/year to 3 mm/year, so it is termed as medium risk corrosion.
5. The above all corrosion tests results were conducted in normal concrete specimens are moderate corrosion risk, so the fly ash and rice husk ash based geopolymer concrete specimens are more durable than the normal concrete specimens.

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