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An Experimental Investigation of Red Soil Based Geopolymer Mortar Without Portland cement

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Abstract: Vast using of ordinary Portland cement (OPC) for construction purpose caused pollution to the global environment as production of OPC released the significant amount of CO2 to the environment. So now a days it is essential to reduce the global carbon dioxide which is encouraged to the researchers to search an eco-friendly alternative material for sustainable construction. One of these alternative materials for this type of construction is Geopolymer based construction materials such as mortar, concrete and bricks, those are having 100% cement less and increasing popularity towards the sustainable development. This Geopolymer based mortar or concrete can be produced from mineral admixtures, such as fly ash, clay, red mud, ground granulated blast furnace slag, meta-kaolin and silica fume, with user-friendly alkaline-reagents. In this investigation red soil based mortar is described as this is locally available greener and low cost material compared to OPC mortar. This paper is showing the possible application of red soil based Geopolymer mortar in construction industry and to find out its compressive strength, chemical durability, sorptivity, coefficient of water absorption, elevated temperature study, ultrasonic pulse velocity, water absorption and porosity with different molarity and percentage of binder. Moreover composition and microstructure of red soil were characterized by XRD, TGA, SEM and EDAX as well.

Index Terms- OPC, Geopolymer Mortar, Red Soil, Microstructure, Molarity, Performance, Application

1.0 Introduction:

Concrete is the 2nd widely used construction material in the world. The main ingredient used for this production of Ordinary Portland Cement (OPC) which is the most energy intensive construction material and whose production increases approximately 3% annually [1].

The production of OPC releases large amount of carbon dioxide (CO_2) to the atmosphere which is currently contributing to greenhouse gas emissions in global world. Current statistics have shown that one ton of OPC produced almost one ton of CO_2.

Moreover the production of 1 ton OPC requires approximately 1.5 tons of limestone, and considerable amount of both fossil fuel and electrical energy [1]. Hence, it is inevitable to find an alternative material to the existing most expensive and energy consuming OPC. One of this promising alternative for the replacement of OPC with by product material such as red soil based Geopolymer mortar which is one more environmental friendly and locally available material.

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Geopolymer is a type of amorphous aluminosilicate cementitious material. Due to wide range applications of concrete and mortar a considerable research have been shifted towards the development of inorganic Geopolymer.

Several reports can be found in the literature on the synthesis, properties and applications of Geopolymer [2]. The development of this technology is an important step towards the production of eco-friendly concrete. It can be synthesized by poly-condensation reaction of geo-polymeric precursor and alkali poly-silicates known as geo-polymerization process. It is an innovative technology that can transform several aluminosilicate materials (Clay, Red Soil, Fly ash etc.) into useful products called Geopolymer or inorganic polymers [3].In this investigation red soil is a predominant materials for making Geopolymer mortar.

Due to differing mineral sources and refining processes adopted red soil varies in physical, chemical and mineralogical properties, rust hue is an intrinsic property of all red soil, which is caused by the oxidized iron present in the soil. In addition, solid constituents of red soil include mainly iron oxides (mostly hematite), alumina, and some toxic heavy metals (Zhang et al. 2010). It also can be slightly radioactive if the original bauxite contained radioactive minerals [4].

2.0 Experimental Work

2.1 Materials Used:

Red soil: Collected from Karaikudi, Tamil Nadu is used for casting the specimen.

Fine Aggregate: Locally available River sand (local) passing through 4.75mm mm sieve was used.

Binder: Combinations of sodium hydroxide (NaOH) and Sodium Silicate (Na_2SiO_3) are employed to achieve the activation of the Geopolymer mortar. The chemical composition of sodium silicate is: Na_2O-14.922%, SiO_2-43.247% and H_2O -41.83% [5] .The binder concentrations were 8M, 10M, 12M, and 14M with alkaline activator to red soil ratio 0.2, 0.3 and 0.4 were used for the investigation.

Water: Portable water is used with pH 7 to 8.5; Cl^- = 40 ppm, Hardness = 240 ppm and distilled water was used for making binder [5].

2.2 Elemental and Morphological Characterization:

X-ray Diffraction (XRD):

The X-ray diffractograms of red soil-based geopolymers are shown Fig 1. Although red soil is an essentially vitreous material, it contains a series of minority crystalline phases such as quartz (SiO2, JCPDS 05-0492), Kaolinite (3Al2O3.2SiO2, JCPDS 15-0776) and magnetite (Fe3O4, JCPDS 19-0629).

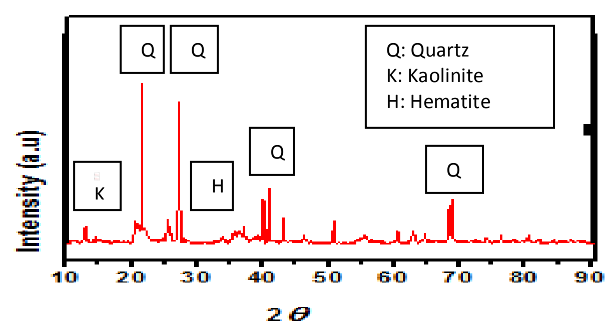

Fig1. X-ray diffractograms of red soil

The basic material of the Geopolymer-based red soil is of a prevailingly amorphous character only seldom containing needle-shaped minority crystals [6].

Scanning Electron Microscopy (SEM):

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The scanning electron microscopy (SEM) of red soil was illustrated as shown in Fig 2.It has shown that soil particles are spongy in shape and have a wide range of diameter.

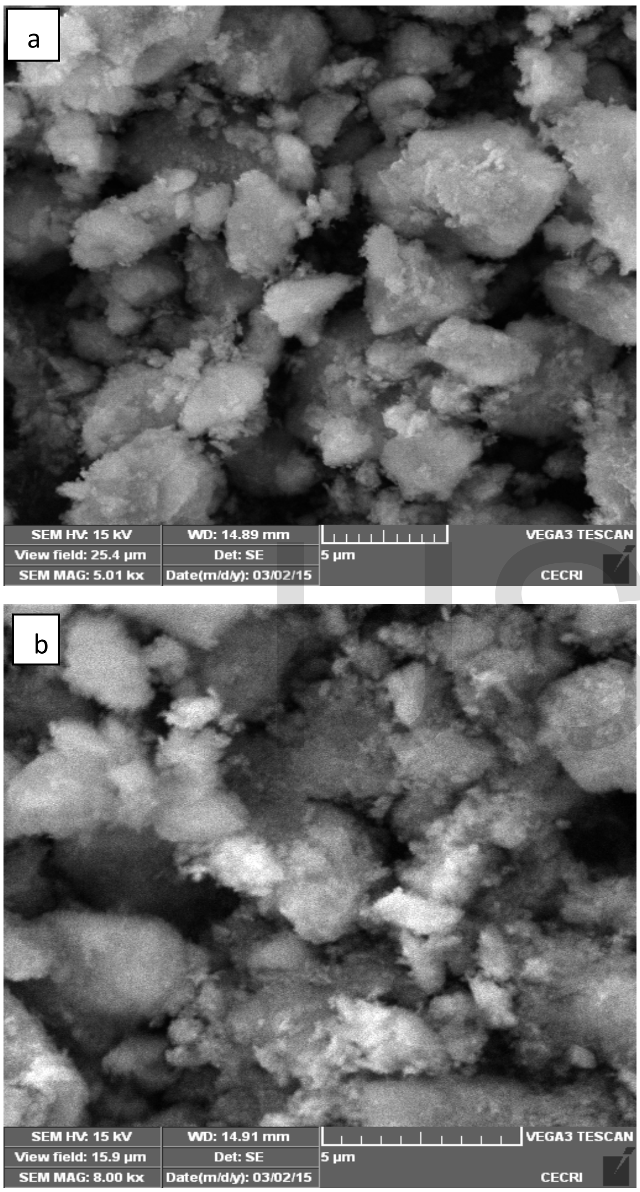
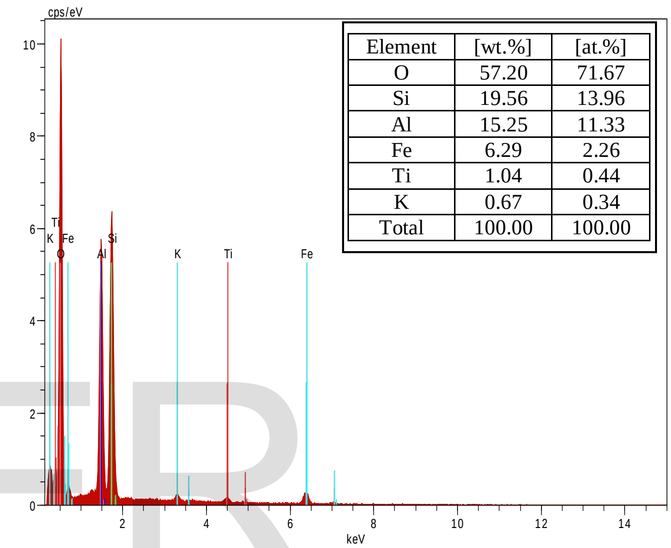


Fig2**.** SEM micrographs of red soil with different magnification a) 5.01kx and b) 8.00kx

Energy Dispersive X-ray (EDX):

To analyze the purity of red soil particles elemental analysis was performed by Hitachi S-3000H EDX instrument. The sample was taken on a carbon coated copper grid and placed inside the evacuated chamber and analyzed. The red soil component by SEM-EDX method has the same result from the X-ray Diffractometer (XRD) method which is shown in Fig.3. From the figure it has been observed that red soil is having more than 90% of aluminosilicate materials and can be used for making Geopolymer mortar.



Element	[wt.%]	[at.%]
O	57.20	71.67
Si	19.56	13.96
Al	15.25	11.33
Fe	6.29	2.26
Ti	1.04	0.44
K	0.67	0.34
Total	100.00	100.00

Fig3. EDX spectrum of red soil

Thermal Gravimetric Analysis (TGA):

In this TGA test, the mass loss was measured while the specimens were gradually exposed to increasing temperatures. Powdered specimens were used in TGA to ensure the achievement of thermal equilibrium during transient heating [7]. Fig.4 shows the results of the TGA/DTA analysis of the red soil performed at a heating rate of 20^0C per minute upto 1000^0 C.

From results it has shown DTA of triaxial composition reveals endotherms with maxima at approximately 30.71°C for dehydration. The peak at 375.04 °C corresponds to dehydroxylation of kaolin ($Al_2Si_2O_5(OH)_4$) to form meta-kaolin. The exotherm

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at 618.05°C corresponds to the onset of crystallization of mullite.

TGA profile (Fig.4), shows mass loss of 10.52 wt%.

It can be seen that TGA curves for these pastes consist of four zones:

- ~ 25-85.67 °C: Dehydration of pore water;
- ~ 85.673-287.78 °C: Dehydration of silicate hydrates;
- ~ 287.78-420.39 °C: Dehydroxylation of calcium hydroxide; and
- ~ 420.39-613.95 °C: Decarbonation of $CaCO_3$.

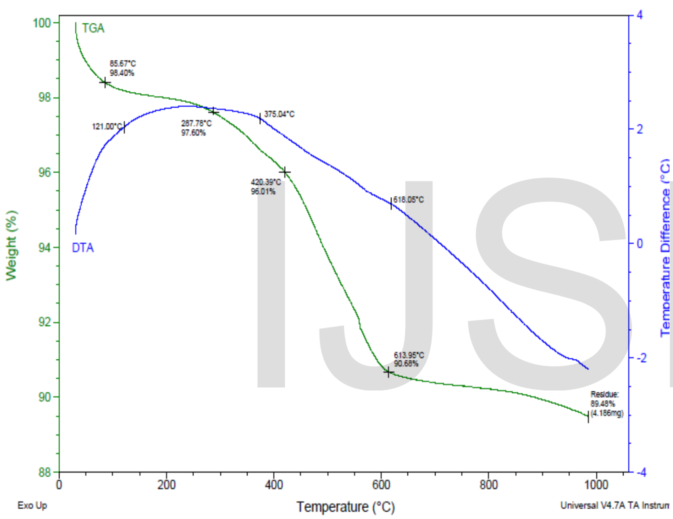

Fig.4 TGA/DTA of red soil particles

Differential Scanning Calorimetry (DSC):

DSC was used to measure a number of characteristic properties of the geopolymer pastes. Using this technique, it is possible to observe exothermic and endothermic reactions as well as glass transition temperatures (Tg) [7]. DSC diagram for red soil is shown in Fig.5 and this figure indicates that the DSC thermogram of red soil is smooth.

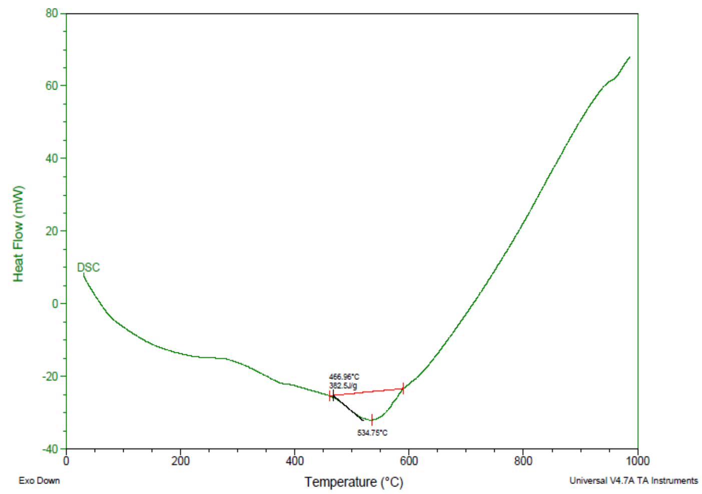

Fig5. DSC Thermogram for red soil.

2.3 Mixing and Specimen Preparation:

Red soil and mixture of alkaline activators namely sodium silicate and sodium hydroxide (NaOH) solution with a different alkaline activator/soil ratio (0.2, 0.3 and 0.4) were used to prepare a red soil-based geopolymer with a various sodium hydroxide concentrations such as 8M, 10M, 12M and 14M. Red soil to fine aggregates ratio was maintained at 1:3. Additional water used approximately 6 % of total sample weight. A ratio of sodium silicate solution-to-sodium hydroxide solution, by mass, of 2.5 was used **[8].** The sodium silicate solution and the sodium hydroxide solution were mixed together at least 24 hours prior to casting of the specimens.

The fresh Geopolymer mortar was used to cast cubes of size 50mm x 50mm x 50mm to determine its compressive strength, chemical durability, elevated temperature study, ultrasonic pulse velocity, sorptivity, water absorption as well as co-efficient of water absorption. Each cube specimen was cast in three layers by manual compaction as well as by using vibrating table. Each layer received 25-30 strokes of compaction by rod, followed by further compaction using vibrating table.

2.4 Curing of Test Specimens:

After casting, the specimens were allowed to set for 24 hours in moulds. Then, the specimens were removed from the moulds and heat cured in oven at 60-70^0 C for 24 hours. After that, the specimens were cured in room temperature until they reached the 7th and 28th days of age.

3.0 Tests Carried Out

The following tests were conducted to investigate the performance of red soil based mortar.

(a) Compression Test
(b) Chemical Durability
(c) Water absorption and Porosity
(d) Sorptivity
(e) Coefficient of Water Absorption
(f) Elevated Temperature Study
(g) Ultrasonic Pulse Velocity

3.1 Compressive Strength:

The compressive strength test of concrete is one of the most important and useful properties of concrete. In most structural applications concrete is employed primarily to resist the compressive stresses. The compressive strength is frequently used as a measure of these properties. Mortar cube specimens of 50mmx50mmx50mm were cast with a different percentage and concentration of binder. After a specified period, specimens are subjected to compression test by using universal testing machine of 100T capacity at a loading rate of 140kN/min.

3.2 Chemical durability:

The Chemical durability of mortar is an important requirement for the performance of the structure in aggressive environments throughout its design life period. The durability of mortar primarily depends upon its permeability characteristics. Impermeable blocks can resist the ingress of aggressive ions into the blocks, thereby reducing the damage occurring due to the deterioration of concrete or blocks and the corrosion of steel in concrete [9].Geopolymer mortars were subjected to acid and sulfate resistance test by immersing the specimens in 5% HCl, 5% H_2SO_4 and 5% Na_2SO_4, over a period of 28 days and the weight loss was calculated. 5% is chosen on the basis of the aggressiveness when compared to 10% concentration [10].

3.3 Water absorption and Effective porosity:

This test was done as per the procedure given in ASTM C 642-90 by oven drying method for this test 50mm x 50mm x 50mm cube was cast. After 48hours of de-molding, the specimens were kept in air and thermal curing. At the end of curing periods, the specimens were kept in open atmosphere for surface drying. Then the specimens were dried in an oven at a temperature of 100+5°C for 48hours and weighed by using a standard weighing balance to an accuracy of 1gm. Record the weight of dried specimens as Wd**.**

The specimens are immersed in water at room temperature (28°C) for 48 hours. Then the surface moisture was removed with a towel or cotton and weighed. This weight is designated as Ws. After that the specimens were immersed in tap water in a container and boiled for 5 hr at 60 ^{0} C. Allowed to cool for natural loss of heat for 14 hours and then surface moisture was removed and reweighed. This is designated as W_b. The suspended weight of the specimens kept in water is taken as Wi. Then the following parameters were calculated as follows:

% Water Absorption= (Ws-Wd)/Wd

Bulk Sp.Gravity(Dry)=Wd/(Wb-Wi)

Apparent Sp.Gravity=Wd/ (Wd-Wi)

Permeable voids (%) = (Wb-Wd)/(Wb-Wi)

3.4 Sorptivity:

Sorptivity measures the rate of penetration of water into the pores of mortar by capillary suction. The test was conducted by ASTM C1585 [11]. Sorptivity of the mortar is given by

$$S = Q/A\sqrt{t}$$

Where
S – Sorptivity in $kg/mm^2/\sqrt{min}$
Q – Quantity of water penetrated in kg
A – Surface area of specimen through which water penetrated
t – Soaking time (60 minutes)

3.5 Coefficient of water absorption:

The Coefficient of water absorption test was carried out as per ASTM C642-97. The same specimens used for effective porosity was used for this study also. Coefficient of water absorption is a measure of water permeability and is calculated as follows: [12].

This is calculated from the formula:

Coefficient of water absorption $Ka = (Q/A)^2 \times (1/t)$

Where Q = Quantity of water absorbed by the oven dried specimen.

t = 48 hours (172800 second).

A = Total surface area of concrete specimen through which water penetrates.

A lower value of Ka indicates a higher degree of imperviousness of concrete for water penetration.

3.6 Elevated temperature study:

Geopolymer Specimens with different concentration were prepared to study the thermal stability of geopolymer mortar, especially on the compressive strength. Muffle furnace was used for this study. Specimens were subjected to heat at temperatures of 200°C and 400°C at an incremental rate of 20°C per minute from room temperature. The temperature was sustained for 24 hours. Then the specimens were allowed to cool down for 24 hours at room temperature inside the furnace and tested for their compressive strengths.

3.7 Ultrasonic pulse velocity:

UPV is a recognized non-destructive evaluation test to qualitatively asses the homogeneity and integrity of concrete and mortar. This test essentially consists of measuring travel time, T of ultrasonic pulse of 50 to 54 kHz, produced by an electro-acoustical transducer, held in contact with one surface of the concrete member under test and receiving the same by a similar transducer in contact with the surface at the other end. With the path length L, (i.e. the distance between the two probes) and time of travel T, the pulse velocity (V=L/T) is calculated [13].

4.0 Result and discussion

4.1 Compressive Strength:

The result of Compressive strength at 7 and 28 days obtained for mortar cubes are reported in Table 1. From the table, it is found that the compressive strength increases with increase in curing period as well as increasing alkaline liquid to red soil ratio.

Table 1: Compressive strength value for red soil

Alkaline liquid to red soil ratio	8M		10M		12M		14M	
	7 Days	28 Days	7 Days	28 Days	7 Days	28 Days	7 Days	28 Days
0.2	1.4	1.91	1.56	2.01	1.72	2.10	1.92	2.36
0.3	3.5	3.86	3.61	4.01	3.7	4.10	4.84	5.05
0.4	3.78	4.83	3.87	5.41	4.01	6.87	5.13	7.73

4.2 Chemical Durability:

The exposure of the geopolymer in the acid solution shows that the weight loss due to the exposure is only 0.8 % when immersed in 5% sulfuric acid and 0.9% when immersed in 5% hydrochloric acid solution. Moreover exposure in sulphate solution has shown 0.6 % weight loss when immersed in 5% sodium sulphate solution. Bar chart showing reduction of compressive strength for 40% binder with different concentration when exposed in acid and sulphate solution for a period of 28 days is shown in Fig 6.

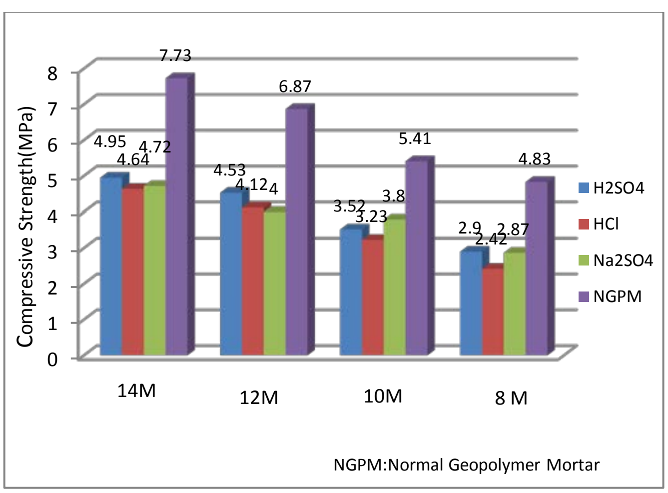

Fig 6.Comparison of compressive strength after immersion in H_2SO_4, HCl & Na_2SO_4

4.3 Test for Water absorption, Specific gravity and Permeable voids (ASTM C642-90):

The results for Water absorption, Specific gravity and Permeable voids are tabulated in Table 2, Table 3 and Table 4.From these result it has shown that better performance is showing when alkaline liquid to red soil ratio=0.4

Table 2: When Alkaline liquid to red soil ratio=0.2

Name of the Test	8M	10M	12M	14M
Absorption of water (%)	11.9	9.7	10.9	11.7
Bulk sp. Gravity (dry)	1.7	1.8	1.62	1.80
Apparent sp. gravity	2.28	2.29	2.0	2.44
Permeable voids (%)	25.43	21.39	19.0	26.23

Table 3: When Alkaline liquid to red soil ratio=0.3

Name of the Test	8M	10M	12M	14M
Absorption of water (%)	8.5	7.68	8.68	7.1

Alkaline liquid to red soil ratio	Concentration	Sorptivity (kg/mm $^2/\sqrt{min}$)
0.3	8M	1.24×10^{-6}
	10M	1.03×10^{-6}
	12M	1.20×10^{-6}
	14M	1.06×10^{-6}
0.4	8M	1.14×10^{-6}
	10M	1.04×10^{-6}
	12M	1.17×10^{-6}
	14M	1.10×10^{-6}

Bulk sp. Gravity (dry)	1.58	1.54	1.55	1.56
Apparent sp. gravity	1.92	1.82	1.86	1.87
Permeable voids (%)	17.7	15.38	16.67	16.60

Table 4: When Alkaline liquid to red soil ratio=0.4

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4.4 Sorptivity:

The results of the sorptivity test are presented in Table 5. The result has shown that the reduction of sorptivity was proportional to the increase level concentration as well as alkaline liquid to red soil ratio.

Table 5: Sorptivity value for red soil based Geopolymer mortar.

4.5 Coefficient of Water Absorption:

The average value of Coefficient of water absorption for red soil is presented in Table 6.From this table it was observed that, the coefficient of water absorption values are decreasing with the increase of molarity.

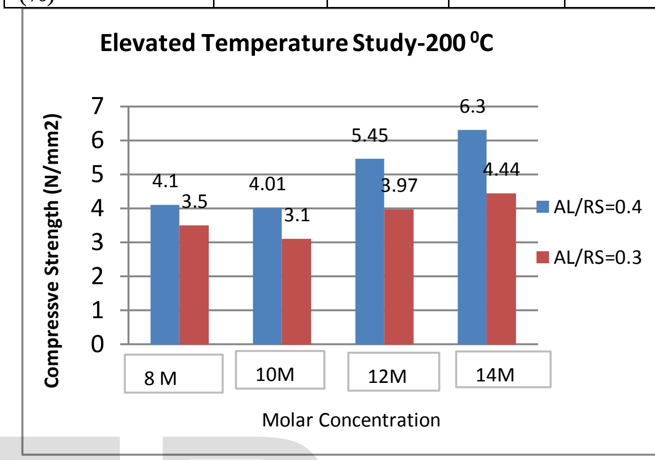
Table 6: Co-efficient of absorption for red soil

Alkaline liquid to red soil ratio	**Concentration**	**Water Permeability (m^2/s)**
0.2	8M	2.61 x10^{-8}
	10M	2.11 x10^{-8}
	12M	1.65 x10^{-8}
	14M	1.88 x10^{-8}
0.3	8M	2.6 x10^{-8}
	10M	1.79 x10^{-8}
	12M	2.41 x10^{-8}
	14M	1.88 x10^{-8}
0.4	8M	2.51 x10^{-8}
	10M	1.98 x10^{-8}
	12M	1.90 x10^{-8}
	14M	2.03 x10^{-8}

4.6 Elevated Temperature Study:

Elevated temperature studies were carried out at 200°C and 400°C and for all the systems and represented in Fig7.

Name of the Test	**8M**	**10M**	**12M**	**14M**
Absorption of water (%)	8.34	8.14	7.98	8.0
Bulk sp. Gravity (dry)	2.01	2.0	2.02	2.02
Apparent sp. gravity	2.43	2.55	2.55	2.57
Permeable voids (%)	21.4	20.14	19.34	21.4



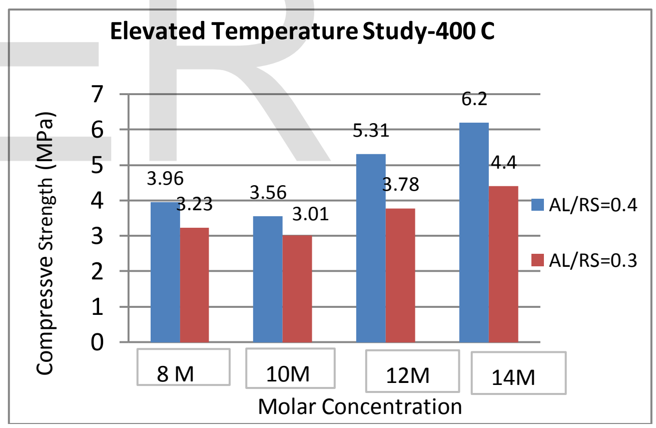


Fig7. Compressive strength vs. mortar concentration at 200^0 C & 400^0C

From the studies, compressive strength was found to decrease with the increase in temperatures for all type of mortar. But among all, 14M and 40% Geopolymer mortar has shown very less strength reduction compared to others.

4.7 Ultrasonic Pulse Velocity (UPV):

A column chart (Fig.8) showing the comparison of ultrasonic pulse velocity for red soil Geopolymer mortar with different concentration and percentage of binder for 28 days .It has shown that red soil based mortar containing 40 % binder has shown higher UPV value compared to others.

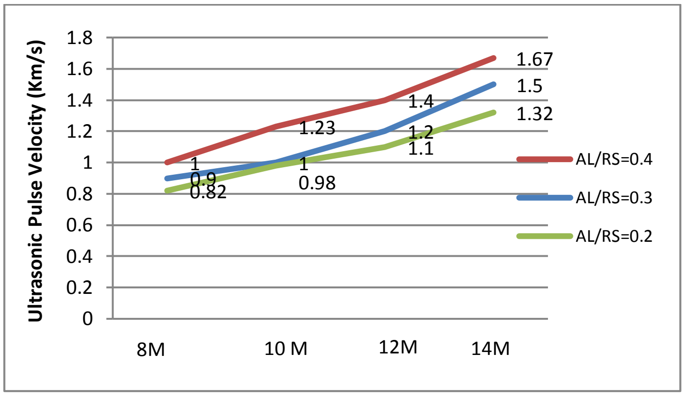


Fig8.Ultrasonic Pulse Velocity for red soil mortar

5.0 Sustainable Application of Red Soil

Production of a wide range product, red soil acts as a raw material and current research has been shifted towards its utilization for sustainable construction. It can be used as a construction material in bricks, blocks, light weight aggregates, in cement industry as cements and special cements and in concrete industry [14]. It is considered as a raw product for production of building materials such as cement, roofing tiles and glass-ceramics.

An efforts have been made at Central Building Research Institute, CBRI, India [15] to produce burnt clay bricks as a fired building materials by partially replacing the clay with red soil (from the Indian Aluminium Company), lime and fly-ash. Moreover an investigation has been done with a small amount of lime in red soil and compresses the mix at minimum moisture content in the form of bricks. The main purpose of this investigation was to check the strength and durability to the erosive action of water. It was found that a maximum wet compressive strength of 3.75 MPa with 5% lime and 4.22 MPa with 8% lime after 28 days of casting and humid curing of these bricks [16].

Red soil reacted with fly ash, sodium silicate via geopolymerization process to get Geopolymer which are a viable cementitious material that can be used in roadway constructions and structural elements such as massive bricks [17, 18]. It can be utilized as an additive material to produce cements, mortars and concretes, construction of dykes, stabilization material and ceramic/refractory product [19].

6.0 Conclusion

The following conclusions were drawn from the above investigation:

- Compared to ordinary Portland cement mortar red soil mortar has shown lower performance but in future these are potential material for replacing the use of OPC in infrastructure development.
- By changing or modifying the various parameters it is possible to improve the performance of these materials which plays an important role for sustainable construction.
- As this materials contain zero percent cement, this is one of the potential materials for reducing global CO_2.Also as an energy efficient materials these can be considered as

a less energy intensive, since Portland cement is highly energy intensive material next to Steel and Aluminum.

- Moreover this Geopolymer mortars have been utilized the locally available materials for producing the binding material in mortar, so it can be considered as a sustainable material for eco-friendly construction. This report could be useful as guidance and as a reference to the related organization and future research on red soil based products for engineering application.

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