STUDIES ON GEO-POLYMER BASED LIGHT WEIGHT BRICKS

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ABSTRACT: Light weight geopolymer bricks were developed. The mix was made with lime sodium silicate and fly ash as ingredients. Bulk densities were also lower at the ratio of 1.0. The maximum Cold Crushing Strength (CCS) value is found at the ratio of 0.745. Increase in mix water decreases the strength. Water content of 24% is recommended for better workability with reasonably good properties. The free energy is positive hence reaction favors high temperature curing. The strength is increased with time. The strength reported at 27 days is 21.40 MPa. From the present study 5% CaO & 7% Na_2SiO_3 composition is recommended for practical purposes as it has porosity 22%, low bulk density 0.983g/cc and a good strength of 23.5 MPa.

Key Words: **Fly Ash, Geo Polymer, Brick, Lime, Bulk Density, Silica**

1. INTRODUCTION

Industrialization warrants construction and operation of thermal power plants of very high capacity. Coal based power generation is very significant in India as it is bestowed with waste coal reserves. Thermal power plants generate huge quantities of bottom ash, flue gas desulphurization cinder and fly ash. All these ashes are to be disposed safely to prevent environmental damage and sustained development. But the fixation of fly ash is of prime importance as it propagates through air as aerosol and reach flora and fauna and causes damage. Fly ash dispersed and deposited all around the neighboring areas followed by leaching of heavy metals which are associated with fly ash causing contamination of water bodies and other resources of water. Finally these heavy metals finds route into the system of both flora and fauna leading to the ecological damage. A number of health hazards are associated to neighboring communities. Hence it is necessary to immobilize (or fix) fly ash with the development of suitable product.

The research on fly ash fixation has been found on the top agenda for many power plants, R&D organizations and governing organizations. Considerable development has been done and several applications are identified. Among them, falgy brick [Sunil Kumar, Department of Civil Engineering, Harcourt Butler Technological Institute, Kanpur 208002, India] is a big success and finds place in every building. Other products of high interest are in the manufacture of pozzolona cement and as cement admixture. All these applications are possible only because of high activity associated with the fly ash. Other fly ash disposal techniques include ceramic tiles, brick with fly ah catalyst, CRF's (controlled release fertilizers) are forerunners [Murali Mohan.V., Rajendra Prasad. P and Sujatha. V; Indian Chemical Engineer Journal, Vol. 47 / No.3, July-Sep. 2005, pp 161-166]. All these techniques warrant energy for their processing except CRF and falgy. The entrepreneurs who are involved in the manufacture of falgy bricks thrive for technology with no heat energy and low density. In pursuit of this task, geo-polymer is found to be a promising option. Several works were carried out in various countries and are reported here under.

Fly ash is fine and its size range varies from 10 microns to sub microns. It contains mainly alumina, silica and light minerals like lime, magnesia, silica. It contains several other minerals in varying quantities based on source of coal and type of environment it has passed through. The chemical composition of fly ash has been presented in Table: 1Chemical composition of fly ash.

Geo-polymer is an inorganic polymer derived from silicon and aluminum atoms in the alumino-silicate materials such as fly ash, which is one of the most abundant sources of combined aluminum silicate. The chemistry and terminology of inorganic polymer was first discussed in detail by Davidovits 1991, 1999. Extensive research in this field has been carried out by several authors throughout the world with different names or wordings such as low temperature alumino-silicate, glass (Rahier et al., 1996), alkali-activated cement (Palono and Lopez Dela Fuente, 2003), alkali-bonded ceramic (Mallicoat et al., 2005) are some of the works seen frequently occurring designations.

Falgy brick has been success for the last two decade in India. It has very good strength, texture but with very high density. The bricks have very high coefficient of expansion unmatched with the concrete structures resulting in the cracks. It has a high density and hence imparts high loads to high raise buildings. On the other hand they possess higher thermal conductivity which is undesirable. Buildings constructed with Falgy bricks necessitate higher reinforcement costs. Hence consume large quantities of steel making the building costlier. All these factors warrant the development of fly ash based light weight bricks/building materials with low density and higher strength. Geo-polymer based materials provide reasonable solution to all the problems raised by Falgy bricks. Therefore the present study is directed towards the development of geo-polymer based light weight geo-polymer brick suitable as building material. Light weight materials offer the following advantages

Table:

Chemical composition of fly ash	
Constituents	Values in weight %
Silica	49-67
Alumina	16-29
Iron oxide	4-10
Calcium oxide	1-4
Magnesium oxide	0.2-2
Sulphur	2
Loss of ignition	0.4-0.6
Surface area(m^2/g)	2300-5700

Lower load hence lower cost of reinforcement. Thermal insulation could be achieved with high porosity and is particularly suitable for the construction of cold storage. It offers smooth surface hence plastering is not required. Present method fixed the highly mobile fly ash into safe aggregate. Present method does not use any energy. Portland cement is not a part in the geo-polymer and hence reduces the cost of the brick.

The methodology adopted in the present study is the formulation of geo-polymer with ingredients like sodium silicate (2-7%), lime (2-7%) and fly ash (86-96%) in varying quantities. All these constituents are mixed thoroughly and molded manually. The bricks were air cured at room temperature. Subsequently the fly ash based geo-polymer bricks were tested for their properties like:

1. Bulk density
2. Crushing strength
3. Water absorption
4. Apparent porosity
5. True porosity
6. Linear shrinkage
7. Temperature effect

All these properties are evaluated in terms of constituents. It is proposed to find out the best composition with reasonable properties.

2. MATERIALS

Materials used in the study encompass fly ash, lime and sodium silicate. Properties of each of the material are presented here under.

2.1. Fly ash

Fly ash used in the present study is obtained from the National Thermal Power Plant, Visakhapatnam. It has a specific gravity of 3.16 and a particle range of 100-0.5 micrometers. The composition of the fly ash used in the present study is given below.

Table: 2

Composition of Fly ash	
Compound	Composition (%)
SiO_2	59.96
Al_2O_3	26.15
Fe_2O_3	5.85

2.2. Sodium Silicate

Sodium silicate, also called water glass or Soluble Glass, is any one of several compounds containing sodium oxide, Na_2O, and silica, Si_2O, or a mixture of sodium silicates varying ratios of SiO_2 to Na_2O, solids contents, and viscosity. Traditionally, sodium silicates are classified according to the acid from which they are derived as Orthosilicate Na_4SiO_4; Metasilicate Na_2SiO_3; Disilicate $Na_2Si_2O_5$; Tetrasilicate $Na_2Si_4O_9$. They are also classified by an X-ray diffraction method according to their crystalline structure. All these compounds are colorless, transparent, glasslike substance available commercially as a powder or as a transparent, viscous solution in water.

Sodium silicate is a binder to provide the required green strength to the samples. It is available in aqueous solution and in solid form and is used in cements, passive fire protection, refractories, textile and lumber processing, and automobiles. Sodium carbonate and silicon dioxide react when molten to form sodium silicate and carbon dioxide.

Sodium Silicate is a liquid material that can be mixed with sand to form a core. It is an easy binder to use because it is a "no bake" product that doesn't require a drying oven.

2.3. Lime

Calcium oxide (CaO), commonly known as quicklime or burnt lime is a widely used chemical compound. It is a white, caustic, alkaline crystalline solid at room temperature. Calcium oxide is usually made by the thermal decomposition of materials such as limestone that contain calcium carbonate ($CaCO_3$; mineral calcite) in a lime kiln. This is accomplished by heating the material to above 825°C, a process called calcination or lime-burning, to liberate a molecule of carbon dioxide (CO_2) leaving quicklime. The quicklime is not stable and, when cooled, will spontaneously react with CO_2 from the air until, after enough time; it is completely converted back to calcium carbonate.

Several binding agents were visualized for the study. Lime and sodium silicate are good binding agents to fly ash. Lime has natural affinity to fly ash to form aggregate and also a nutrient for the plant uptake. Lime is selected for the study as it does not pollute the environment. The application of lime for agricultural or engineering purposes is a well established practice. Addition of lime to soil is intended to maintain a neutral soil pH. Different formulations of lime are available for agricultural and engineering use; both have different chemical and physical properties.

3: EXPERIMENTAL PROCEDURE

3.1. Preparation of the samples

Fly ash used in the present study is obtained from the National Thermal Power Plant Visakhapatnam. Known quantities of chemicals (fly ash, lime and sodium silicate) were exactly weighed and mixed with minimum water necessary for thorough mixing is used in the present study. The minimum water content of 0.24ml/g of mix was maintained constant throughout the study. The Charge was thoroughly mixed. The mix was placed in to mould and allowed for 12 hr and then released. These bricks were dried in the open atmosphere for 1 to several days. The bricks gained sufficient strength. The samples were evaluated for their useful characteristic parameters such as cold crushing strength, bulk density, water absorption, true density, apparent porosity, true porosity and shrinkage.

3.2. Characterization of bricks samples

Linear shrinkage, water absorption, bulk density, true density and apparent porosity of the samples were determined by the following procedure. The apparent porosity, true porosity, bulk density and true density were determined using suspended weight, which is based on Archimedes principles. The step-by-step procedure followed involves the following procedure. Weights of all dry samples were noted down with a balance of accuracy 10mg. Samples were then taken in a vessel and immersed in water and brought to boiling condition and allowed to boil for one hour, brought to room temperature and then soaked for 24hrs. Weights of all samples saturated with water were determinate after wiping off the surface water using a clean cloth. Using the following formulae, the properties of samples were evaluated.

3.3. Water absorption

It is defined as the ratio of weight of foam sample saturated with water to the weight of dry sample.

Apparent water absorption = {(W-D)/D}*100(%)

3.4. Bulk density

It is defined as the ratio of the mass of the material to this bulk volume and it is given by

Bulk density = D/ (W-S) g/cc

3.5. True density

It is given by the following formula

True density = D/ (D-S) g/cc

3.6. Apparent porosity

It is defined as a ratio of volume of open pores to the bulk volume of material and it is given by

Apparent porosity = {(W-D) / (W-S)}*100(%)

3.7. True porosity

It is given by the following formula

True porosity = {1- (bulk density/true density)}*100 (%)

Where D = weight of dry sample,

S = weight of sample when suspended in cold water after boiling in water for 1 hr,

W= weight of sample saturated with water.

3.8. Shrinkage test

The percentage shrinkage was calculated from the measured dimensions of the green body and fired body namely length, breadth and width of the sample and was measured by a vernier calipers which has an accuracy of 0.01 mm. Percentage shrinkage in length of sample = (length of the green body - length of fired body)/length of the green body. Similarly shrinkage in height and breadth of the sample were computed.

3.9. Crushing strength

The samples are tested for their strength using compression testing machine. The compression testing machine consists of a loading unit and a motorized pumping unit. The loading unit consists of one steel base four support pillars, one cross head, one hydraulic jack, one dust cover, one lower platen and one upper platen with self alignment action attached to the lead screw with handle which is screwed to upper cross head. One block fitted to upper cross head with four adaptors for pressure gauges and for hydraulic jack with rubber hose pipe with end connections. One detachable pressure gauge mould mounted on the upper cross head with the help of Allen bolts with one/two, or three pressure gauges and isolating valves. The pumping unit consists of oil reservoir, fitted with an electrically driven plunger type pumping system with slow and fast lever and one knob fixed on this lever for fine control, one ON/OFF push button switch, one handle for hand operation and one release valve. Insert spacers in between the hydraulic jack ram and the lower platen so that when the specimen to be tested is placed on the lower platen, it is about 10 mm from the upper plate. Close the pressure release valve. Bring back the maximum red pointer of the gauge in contact with the black needle and operate the pump to apply load at the specified rate till the material is crushed. The crushing strength was computed and used in the further analysis.

4. RESULTS AND DISCUSSION

The chapter deals with the properties and durability of brick samples made with varied composition of lime (CaO), sodium silicate (Na_2SiO_3) and fly ash. Cold crushing strength, bulk density, water absorption, linear shrinkage, apparent and true porosity values were evaluated and the discussion was continued in terms of composition of geo-polymer brick.

Proportioning of lime, sodium silicate and fly ash is a key factor in the present investigation. Fly ash mainly contains silica, alumina, iron and calcium oxides in different proportions. When lime and sodium silicate are treated together with fly ah they form a polymeric gel to form geo-polymer of highly heterogeneous and bind fly ash particles by producing micro fluid binds particulate matter. As the time proceeds extent of gel formation or polymerization enhances and the character/properties of aggregate enhanced. These properties depend on the composition of the brick, treatment it has under gone and the length of time.

Water content was varied as 52-65% both the limits are arrived by depending on the fabrication method. CaO: Na_2SiO_3 ratio was varied as 0.28 to 3.5. The parameters of the study are presented in table 4.

Geo-polymer based light weight bricks are principally based on the binding capability of calcium-silicate. Lime and sodium silicate are the parameters of high influence on physical properties of bricks. Therefore, bricks were analyzed in terms of composition of the brick namely percent lime, percent sodium silicate and fly ash proportions. Effect of each parameter on each property is discussed separately in the following paragraphs.

Table: 3

The range of variables	
Property	Range
Fly ash	86-96%
Lime	2-7%
Sodium silicate	2-7%
Water content	52-65%
Ratio(CaO/ Na_2SiO_3)	0.28-3.57

4.1: Effect of percentage Sodium Silicate on Crushing Strength

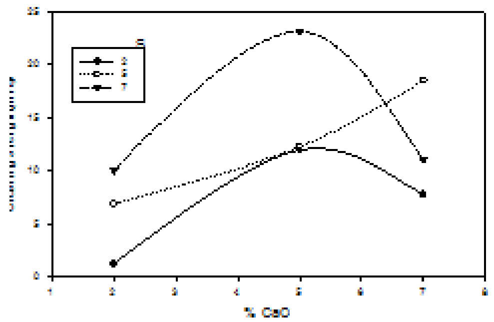

Figure 1: Effect of percentage sodium silicate on crushing strength

Crushing strength is an important property of brick which depends on the extent of sodium silicate present in the brick. The sodium silicate (Na_2SiO_3) reacts with lime to form $CaSiO_3$ gel that binds particulate matter. Some amount of Na_2SiO_3 reacts with free lime and fixes lime present in the fly ash but the exact quantity could not be revealed so easily. Figure 1 is a graph drawn as Cold Crushing Strength (CCS) Vs percent CaO in the brick with Na_2SiO_3 as parameter. The graph reveals as Na_2SiO_3 increases CCS increases. As Na_2SiO_3 increases CCS reaches to a maximum value followed by a decrease. The peak CCS is observed with 5% CaO & 2% Na_2SiO_3 and also with 5% CaO & 7% Na_2SiO_3. The most probable reason is the formation of CaO.SiO_2 ratio in between 0.75 to 2.5 indicating the formation of Ca-silicates. A maximum CCS of 23.54MPa is obtained at 5% CaO & 7% Na_2SiO_3 and hence suggested.

4.2: Effect of percentage Lime on Crushing Strength

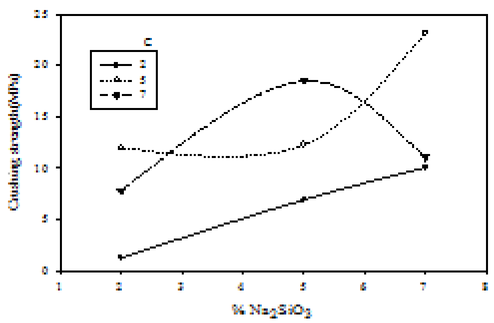

Figure 2: Effect of percentage lime on crushing strength

Figure 2 is a graph drawn as Cold Crushing Strength (CCS) Vs Percentage Na_2SiO_3 with lime as parameter. The graph reveals the following information. At 2% CaO the bricks exhibiting an increase in CCS with Na_2SiO_3 where as for those with 7% CaO, CCS values are increasing to a peak value of 18.5MPa followed by a decrease. The geopolymer with 5% CaO exhibited a marginal decrease followed by an increase.

4.3: Effect of CaO/Na_2SiO_3 ratio on crushing strength:

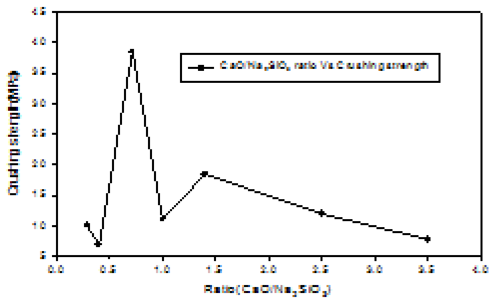

Figure 3: Effect of CaO/Na_2SiO_3 ratio on crushing strength

A graph is drawn as Cold Crushing Strength (CCS) Vs Ratio of CaO and Na_2SiO_3 and shown in figure 3. The figure indicates that the cold crushing strength is increasing with increase in CaO/Na_2SiO_3 ratio followed by decrease to a minimum. The maximum value is found at the ratio of 0.745.

4.4: Effect of CaO/Na_2SiO_3 ratio on linear shrinkage:

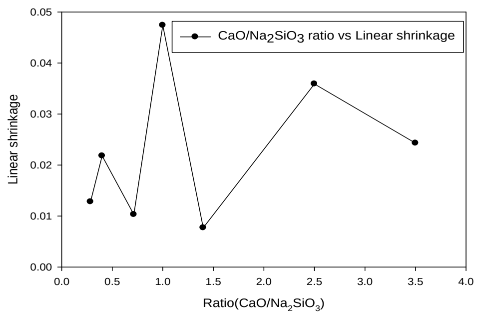

Figure 4: Effect of CaO/Na_2SiO_3 ratio on linear shrinkage

Linear shrinkage is an indication for the interaction among various particles present in the mixture. The interactions are complex and were interpreted by various authors by X-ray diffraction and through microscopy. The interaction is the polymerization of silica with the inclusion of other minor minerals. The network or the formation of gel is the key to the success of material and can be indirectly observed by the linear shrinkage. The linear shrinkage has other advantage like the release from mold. The linear shrinkage is a function of composition of brick and depends on %CaO, %Na_2SiO_3. Figure 4 is a graph drawn as linear shrinkage against the ratio of CaO/Na_2SiO_3. The linear shrinkage has no specific trend with the ratio of CaO/Na_2SiO_3. It may be due to reaction and growth of crystals of CaO.SiO_2 at different ratios like 0.25, 0.5, 0.75, 1.0 and 1.5 etc. A maximum shrinkage is observed at the ratio of 1.0. Incidentally the crushing strengths were found at a considerably lower value indicating the dominance of a specific phase at this molar ratio and can be viewed through electron microscopic studies. The bulk densities were also lower at the ratio of 1.0.

4.5: Effect of Lime on Water Absorption

Lime and sodium silicate have strong influence on the gelation characteristic of geopolymer. As Na_2SiO_3 interact with CaO and fly ash particles resulting in the formation of a range of chemical compounds such as CaO.SiO_2, 2CaO.SiO_2, 3CaO.SiO_2 and $(CaO)_x(Al_2O_3)_y(SiO_2)_z(Fe_2O_3)_v$, where v, x, y, z are numerical values largely depends on origin and constituents of fly ash. Relative quantities and phases of all the above compounds present in the sample dictates the porosity of the polymer and hence water absorption.

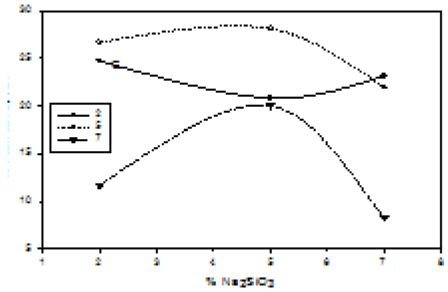

Figure 5: Effect of lime on water absorption

Figure 5 is a graph drawn as Percent Water Absorption Vs Percent Na_2SiO_3 with lime as parameter. Effect of lime and sodium silicate could clearly be visualized from the graph. As Na_2SiO_3 increases percent water absorption raises to peak value followed by a decrease except for the sample with 2% CaO, for which at 5% Na_2SiO_3 showed a slight decrease followed by an increase. Increase in lime content led to an increase followed by a decrease in water absorption. In the present study the brick with the composition of 5%CaO, 7% Na_2SiO_3 showed relatively good strength of 23.54MPa and moderate water absorption of 23% and is suitable for commercial purpose.

4.6: Effect of lime on bulk density

Bulk density is a measure for compactness. Higher bulk densities are necessary for greater compactness which in turn leads to good crushing strengths. Figure 6 is a graph of Bulk density Vs Percent Na_2SiO_3. The figure reveals bulk density is increasing with %Na_2SiO_3 with marginal decrease at high value except for samples with 7% lime for which bulk density is decreased to a minimum followed by an increase. The sample with 5% CaO, 7% Na_2SiO_3 exhibited strength of 23.5MPa and lower bulk strength of 0.983 is recommended.

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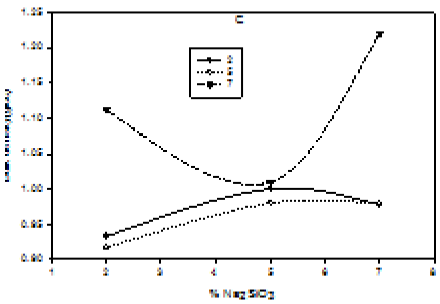

Figure 6: Effect of lime on bulk density

4.7: Effect of Sodium Silicate on Porosity

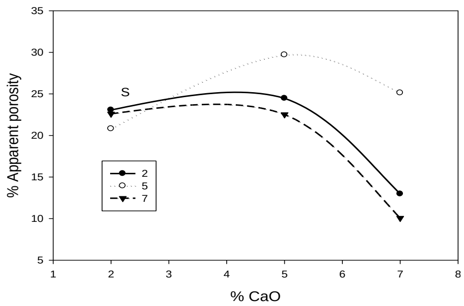

Figure 7A: Effect of sodium silicate on apparent porosity

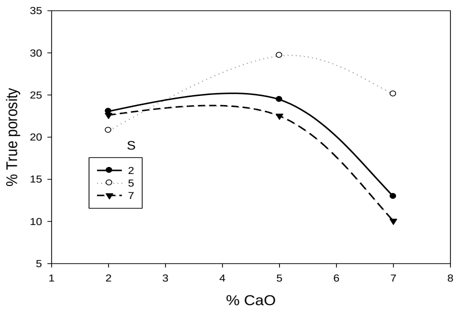

Figure 7 B: Effect of sodium silicate on true porosity

Figure 7A and 7B are graphs drawn as apparent porosity Vs percentage CaO and True Porosity Vs Percentage CaO respectively. These graphs reveal that the porosity is increasing up to 5% CaO beyond which a decline is observed. At 5% lime porosity increased to a peak value followed by a decrease was recorded. As the porosity increases, the strength decreases but bulk density decreases. It is also observed that the variation between apparent porosity and true porosity are marginal. As the silicate composition increases, the porosities are also increased. In the present study 5% CaO & 7% Na_2SiO_3 is recommended as it has a porosity of 22% and strength of 23.5MPa, hence it is recommended for practical purposes.

4.8: Effect of Curing Temperature on Crushing Strength

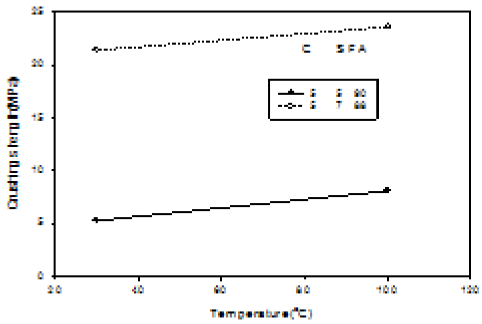

Figure 8: Effect of temperature on crushing strength

Figure 8 is a graph drawn as Cold Crushing Strength Vs maximum Temperature to which the brick exposed. Exposure to higher temperatures recorded approximately 10% increase. The increase is obvious as higher temperatures offer greater extent polymerization silicates leading to higher strengths.

4.9: Effect of water variation on crushing strength

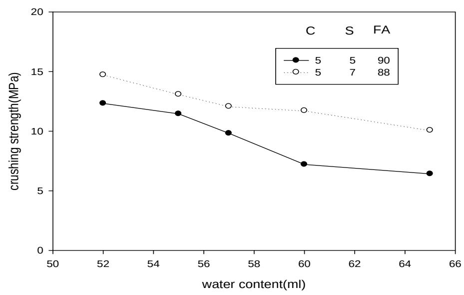

Figure 9: Variation of crushing strength with water content

Figure 9 is a graph drawn as Cold Crushing Strength Vs percent water in the mixture. Present of water in the brick increases the porosity of the brick, which in turn reflects on the strength. The figure reveals, increase in mix water decreases the strength. However, because of workability the water content in the rest of brick were made at 60ml i.e., 24% of water by weight.

4.10: Effect of Curing Time on Crushing Strength

Time of curing is an important parameter for any cementious material. As the time progresses the strength increases but the increase in strength is large up to 27 days beyond which the increase is marginal. The strength reported at 27 days is 21.40 MPa for which composition was

5% lime, 7% silicate. The brick has bulk density 0.983 was recorded.

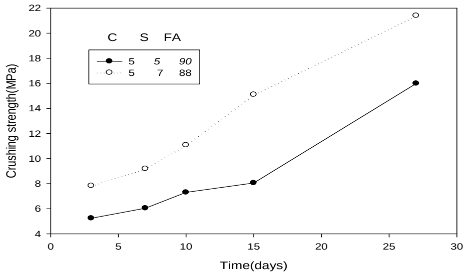

Figure 10: Effect of curing time on crushing strength

4.11: Thermodynamic analysis of Bricks

Geo-polymer settling is a complex reaction which involves the formation of compounds of mixed silicates like $CaOSiO_3$, $2CaOSiO_2$, $3CaOSiO_2$, $4CaOSiO_2Fe_2O_3$ and many other compounds. Silicate hydrates of Calcium (Ca), Magnesium (Mg), Fe_2O_3, TiO_2 are also present. The products also contain Ca, Mg, aluminates. The complexity increases with the formation of different phases like Mullite, Belite, Celite, Felite etc. exact nature of the material may be elucidated by the microscopic studies together with thermo-gravimetric analysis. But some information could be retrieved from thermodynamic analysis presented as follows

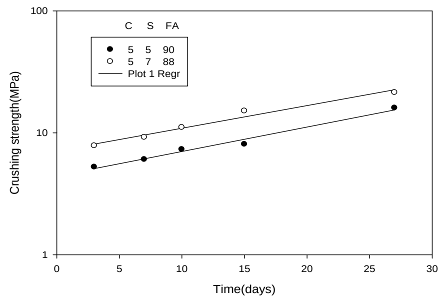

Figure 11: log crushing strength Vs time

Fig. 11 is a graph drawn as log CS Vs Time for different compositions. CCS is the property which depends on the bonding reactions involved in the study. More the complex bonding, greater is the strength. Strength has direct bearing on the extent of polymerization of silicate structure. Greater the extent of Na_2SiO_3 reaction higher is the polymerization extent. Slope of the curve directly relates the Na_2SiO_3 present in compound form. Hence, Na_2SiO_3 compound formation is computed by (δ/C_S); where δ is CCS at t=0 and $C_S=C_{So.}$ The values of δ have been computed and combined silicate concentrations were computed. Further a Figure 12 is drawn as $\ln(C_t/C_0)$ Vs time. It is found to follow first order kinetics. The rate constants are reported as follows.

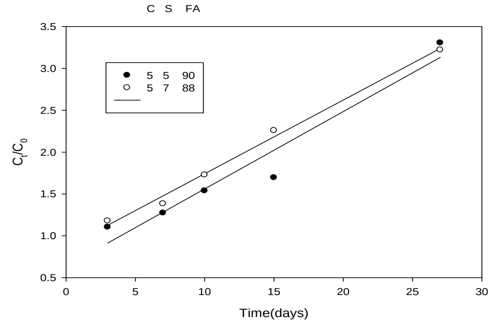

Figure 12: Combined silicate concentrations Vs time

Figure 13 is an Arrhenius plot drawn as ln K Vs 1/T. the graph yielded ΔH= ΔE/R = 17.3871. It is positive hence reaction favors high temperature curing.

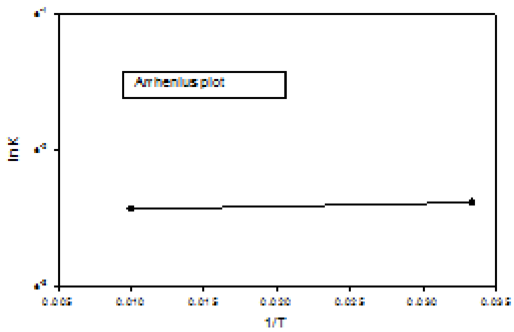

Figure 13: ln K Vs 1/T

5. CONCLUSIONS

1. A maximum CCS of 23.54MPa is obtained at 5%CaO & 7% Na_2SiO_3 and hence suggested.
2. As Na_2SiO_3 increases CCS reaches to a maximum value followed by a decrease except for 5% Na2 SiO3.
3. The maximum CCS value is found at the ratio of 0.745.
4. A maximum shrinkage is observed at the ratio of 1.0. Bulk densities were also lower at the ratio of 1.0.
5. Increase in lime content led to an increase followed by a decrease in water absorption.
6. From the present study 5% CaO & 7% Na2SiO3 is recommended for practical purposes as it has a porosity of 22% and strength of 23.5MPa.
7. Exposure to higher temperatures recorded approximately 10% increase in CCS.

8. Increase in mix water decreases the strength.
9. Water content of 24% is recommended for better workability with reasonably good properties.
10. The strength is increased with time. The strength reported at 27 days is 21.40 MPa for which composition was 5% lime, 7% silicate.
11. Geo-polymer brick developed in the present study has a lower bulk density of 0.983g/cc.
12. The free energy is positive hence reaction favors high temperature curing.

6. NOMENCLATURE

CS	:	Crushing Strength, MPa
WA	:	Water absorption, %
BD	:	Bulk density, g/cc
AP	:	Apparent porosity, %
LS	:	Linear shrinkage
TD	:	True density, g/cc
TP	:	True porosity, %
L_1	:	Length of the green body, cm
L_2	:	Length of the fired body, cm
D	:	Weight of the dry sample, gm
W	:	Weight of the soaked sample, gm
S	:	Weight of the suspended sample, gm
C	:	Lime
Na_2SiO_3	:	Sodium silicate
MPa	:	Mega Pascal
SiO_2	:	Silica
Al_2O_3	:	Alumina
Fe_2O_3	:	Iron oxide
X.R.D.	:	X-Ray diffraction
S	:	Sodium silicate
C_t	:	Indicative concentration at time t, mol/cm^3
C_{St}	:	Crushing strength at any time t, MPa

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