Preparation and Characterization of Lime Activated Unfired Bricks Made with Industrial Wastes

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Abstract — Utilization of fly ash can reduces the solid waste disposal problem and minimization of pollution also. Conventional production of bricks usually utilizes clay and shale as the source material and requires high temperature (900–1000 °C) kiln firing which is energy intensive, adversely affect the landscape, and can release high level of waste materials. Utilization of fly ash for making lime activated bricks not only give the waste utilization but also save earth shale as well as energy consumption and CO2 emission. The present study deals with the utilization of fly ashes for making lime activated fly ash bricks and their different property characterization after a normal water curing method up to 21 days. The crushing strength was observed 2.02, 3.17 and 5.32 MPa for 7, 14 and 21 days curing respectively. On the other hand apparent porosity and water absorption was observed 52, 52, 50 and 46, 47 and 43% for 7, 14 and 21 days curing respectively. The SEM-EDAX analysis was observed the initial formation of CASH phase along with free silica particle. But increasing curing time many new phases were observed such as CSH, CAH etc which reduce free silica particles present inside the bricks, responsible for further increase in strength.

Keywords — CASH, Crushing strength, Fly Ash, Lime activated bricks, Unfired bricks

1. INTRODUCTION

Fossil fuels are used in modern power plants throughout the world to produce electrical energy. The inorganic residue that remains after pulverized coal is burned is known as "coal combustion byproducts" (CCBs) [1]. Fly ash (FA) and, to a lesser extent, bottom ash (BA) are the combustion residues produced and collected during coal burning in a Thermal Power Stations (TPS). In general, coal ash in a power plant consists of up to 25% bottom-ash and 75% fly ash [2]. Chinese power plants consumed 1.09 billion tons of coal and generated 81.8% of the total electricity in 2005. Meanwhile, 293 million tons of coal combustion residues were generated by Chinese power plants in 2005 and the amount of that will increase every year [3]. Indian coal used in power plants generally has high ash yield (35–45%) and is of low quality [4]. Presently about 110 million tons (MT) of coal ash is generated in India from more than 70 thermal power plants per year [5]. Environmental problems can be caused by coal mining, transportation, storage and utilization, among which coal combustion is most significant [6].

In India, as per report on Fly Ash Generation 2010-11 [7], out of 31 power utilities, six utilities have achieved ash utilization level of 100% or more and eleven power utilities have achieved ash utilization level in the range of 100 to 75%.

In coming year (2016-17) it is expected that India will produce 300-400 Million Tons Fly Ash per year which is approximately double the quantity it is produced now so the consumption should be increased subsequently [8].

bricks with the amount of replacement above 50%. From the above studies it was concluded that constructional bricks can be made by the effective utilization of fly ash.

Thus, utilization of fly ash can also reduce the solid waste disposal problem and minimization of pollution. Conventional production of bricks usually utilizes clay and shale as the source material and requires high temperature (900–1000 °C) kiln firing. Quarrying operations for producing the clay and shale are energy intensive, adversely affect the landscape, and can release high level of waste materials. The high temperature kiln firing not only consumes significant amount of energy, but also releases substantial quantity of greenhouse gases. It is also noted that there is a shortage of clay and shale in many parts of the world. To protect the clay, shale resource and the environment, some countries such as China have started to limit the use of bricks made from clay and shale [17],[18].

On the other hand, the production processes of a construction material have a considerable impact on the environment. Edwards and Bennett, [19] reviewed the lifecycle concepts and considered recent developments. Works on the use of some secondary materials and waste types, as partial substitute for primary clay in the manufacture of fired bricks, in order to reduce the energy and firing cost of the clay brick production process was conducted by many authors [20]-[23]. Previous researchers [24]-[26] studied the possibility of producing low-cost unfired clay building bricks utilizing PFA/lime clay mixture. Jayasinghe and Kamaladasa [27] among others reported on compressive strength and erosion characteristics of unfired clay bricks. Abali,Y.et al [28] produced unfired phosphorgypsum bricks. Venkatarama Reddy et al. [29] reported on enhancing bond strength and characteristics of unfired clay bricks made from PC-clay mixture. Coal fly ash consist mainly of Fe, ca, K, Si, and Al in the form of quartz and amorphous. [30]. For that reason fly ash is being gaining popularity for making of lime activated bricks.

In respect of the environmental and sustainability, scoring method of BREEM, [31] has been used. The criteria for BREEM are transportation, carbon dioxide emissions, embodied energy, depletion of resources; (use of waste materials, landfill), occupants’ health (regarding end-products), product reuse and overall perception in terms of care for the environment were considered. Such analyses can lead to improvements in the life cycle of products and provide criteria for design decisions, when choosing materials offering similar performances for a given application. For common fired bricks, the total energy usage (input) is estimated at 4186.8 MJ/tone with equivalent output emissions of 202 kgCO2/t [32]. The total energy usage for the unfired brick types (experimental unfired brick) was estimated at around 657.1 MJ/tone and the carbon dioxide emissions for the experimental unfired bricks were estimated at 40.9 kgCO2/tone [33].

There are a number of patents on the use of fly ash–lime mixtures for making unfired bricks [34]-[36]. In addition to lime, other solidifying agents such as slag, calcined gypsum and dextrin are added in the production. The studies usually focus on the manufacturing process and less attention is paid to the chemical reaction and the microstructure. It is well known that chemically pozzolanic reaction of fly ash and lime occurs readily under thermal treatment creating strong structures with an increase of mechanical strength [37]. This reaction involves the formation of calcium silicate hydrate (CSH) and calcium alumino-silicate hydrate (CASH) and enhances the strength of the materials [38]. The type of CSH phases plays a crucial role in the strength development of the final product owing the variation of Ca/Si ratio directly affects the types of hydrate products. For CASH, Al/(Si+Al) ratio also affects the types of products. For example, the formation of hydro garnet (C3ASzH6–2z) requires the ratios in the range of 0.12–0.50 when kaolinite is used as the source of aluminium [39]. CASH can be variable in compositions, especially in hydroxyl sites where hydrating water is localized. The amount of water available for the reaction, therefore, must be taken into consideration as it is an important factor affecting the type of hydrate products for both CSH and CASH systems. The hydro garnet is formed directly from pozzolanic reaction and not as result of a conversion reaction. Several researches study the effect of clayey materials such as kaolinite, sercite and montmorillonite, incorporating with quartz–limestone (CaO–SiO2–Al2O3–H2O) on the structure and composition of reaction products such as CSH, tobermorite and hydrogarnet [40]. The types of starting materials thus affect the formation of hydro garnet due to the differences of alumino-silicate structures. The different starting materials result in the different rates of dissolution and thus the different nature of precipitation of new products. These new products such as ordinary tobermorite and Al-substituted tobermorite have potential in fixing radioactive species and several kinds of heavy metals [41]. National thermal power corporation limited [42] made the fly ash bricks with the composition fly ash-55-65%, sand dust-18-32%, hydrated lime-8-12%, gypsum-5%. Curing was carried out by sprinkling water manually. After curing period of 28 days, Compressive strength and Water absorption were got 7.5-10 N/mm2 and 17-20% respectively.

Usually curing is carried out by sprinkling water manually or by covering the bricks in gunny bag to maintain the appropriate humidity or kept inside the predetermined humid chamber etc. [42]-[45] which is very difficult; to maintain for a low scale bricks producer due to unavailability of infrastructure and lower capital investment.

For avoiding this, the present work deals with the making of Fly Ash Lime activated (FaL-G) bricks with a typical NTPC fly ash and cured with dipping in water method on the effect of different physical and mechanical properties.
II. EXPERIMENTAL

A. Raw Materials
Fly ash, from Renusagar Thermal Power Plant Uttar Pradesh (India), River sand from the Ganges River (India), and Lime and Gypsum from local dealer were used to prepare Sand–Lime-Gypsum brick. The lime and part of the sand were ground to powers by a lab ball mill to get the required fineness.

B. Characterization of Raw Materials
Chemical analysis and physical properties of raw material were done by using Energy Dispersive X-ray Fluorescence Spectrometer (XRF, Philips, PW 1840). Proximate and ultimate analysis of Fly ash was carried out using gravimetric methods. Particle size analysis was done by sieving the raw materials in different sieves (shown in Fig. 1).

Scanning electron micrograph photographs have been recorded using (FEI Quanta-200FEG) at 20kv on scan rate 10µs with ETD detector. Physical and chemical properties of raw materials were given in table (I-III). The main mineral constituents of Fly ash were silica as detected by EDAX (not shown). The SiO$_2$ content of the Fly Ash was only about 45.6%, much lower than the Ganges river sand (90.6) usually used for sand–lime brick, [46] that is why sand powder was used to increase the content of active SiO$_2$. The lime powder used was of high quality because of its high CaO content (88.26%). CaO and Silica content in Gypsum were 31.3% and 4.6% respectively. This preliminary result indicated that the lime and gypsum are qualified for preparing autoclaved sand–lime-gypsum brick.

C. Procedure
First Lime milk was prepared with known quantity of water in a container. Then Fly Ash, sand powder and gypsum were mixed in dry condition. Then lime milk was mixed with the dry mixture for 5 minute manually to get homogenize mixture as per the designed proportions shown in Table-IV.

Wet mixture then placed in a wooden pattern (dimension 30 mm × 30 mm ×30 mm) and pressed manually for better compaction. Then the green bricks were taken out from the mould to get the bricks.

D. Influence of water addition on green crushing strength
At low moisture-15%, it was found that the bricks were low strength and also difficult to remove from the mould. At

<table>
<thead>
<tr>
<th>Mesh Size of particles</th>
<th>Cumulative passing F.A.(%)</th>
<th>Cumulative e passing Sand(%)</th>
<th>Cumulative e passing Lime(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-5 µm</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>5-8 µm</td>
<td>30</td>
<td>40</td>
<td>50</td>
</tr>
<tr>
<td>8-16 µm</td>
<td>60</td>
<td>70</td>
<td>80</td>
</tr>
<tr>
<td>16-30 µm</td>
<td>90</td>
<td>100</td>
<td>100</td>
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</table>

TABLE II. PROPERTIES OF SAND

<table>
<thead>
<tr>
<th>Properties</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific Gravity</td>
<td>2.66</td>
</tr>
<tr>
<td>Bulk Density(Kg/m3)</td>
<td>1250</td>
</tr>
<tr>
<td>Fineness Modulus</td>
<td>2.16</td>
</tr>
<tr>
<td>Water Absorption(%)</td>
<td>2.0</td>
</tr>
</tbody>
</table>

TABLE III. PROPERTIES OF FLY ASH

<table>
<thead>
<tr>
<th>Properties</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific Gravity</td>
<td>2.18</td>
</tr>
<tr>
<td>Specific Surface Area (m2/gm)</td>
<td>2.05</td>
</tr>
<tr>
<td>pH</td>
<td>11.9</td>
</tr>
</tbody>
</table>

TABLE I. CHEMICAL ANALYSIS OF RAW MATERIALS

<table>
<thead>
<tr>
<th>Constituents</th>
<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>CaO</th>
<th>Fe$_2$O$_3$</th>
<th>Na$_2$O</th>
<th>K$_2$O</th>
<th>P$_2$O$_5$</th>
<th>MgO</th>
<th>TiO$_2$</th>
<th>MnO</th>
<th>SO$_3$</th>
<th>L.O.I</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fly Ash</td>
<td>45.60</td>
<td>4.75</td>
<td>5.35</td>
<td>37</td>
<td></td>
<td>0.25</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1.98</td>
</tr>
<tr>
<td>Lime</td>
<td>4.72</td>
<td>0.95</td>
<td></td>
<td>88.26</td>
<td>0.46</td>
<td>0.15</td>
<td>0.017</td>
<td></td>
<td>0.86</td>
<td>0.072</td>
<td>0.15</td>
<td>3.94</td>
</tr>
<tr>
<td>Sand</td>
<td>90.6</td>
<td>4.36</td>
<td>0.34</td>
<td>0.83</td>
<td>0.36</td>
<td>2.27</td>
<td>0.067</td>
<td>0.36</td>
<td>0.081</td>
<td>0.024</td>
<td>0.068</td>
<td>0.66</td>
</tr>
<tr>
<td>Gypsum</td>
<td>4.6</td>
<td>1.75</td>
<td>31.3</td>
<td>1.15</td>
<td>0.13</td>
<td>0.55</td>
<td>0.067</td>
<td>1.35</td>
<td>0.096</td>
<td>0.042</td>
<td>14.1</td>
<td>17.2</td>
</tr>
</tbody>
</table>

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moisture-20%, it was slight easy to remove the brick from the mould but its handling was difficult. The bricks were kept for two days to get surface moisture free but because of low strength it was broken. At moisture 25% the bricks were easily removable from the mould and having good strength, easy to handle. At moisture 30% water bleeding was take place and water comes on the surface and hence due to increasing plasticity own shape and size got disturbed. So after increasing green crushing strength also, it was difficult to remove brick from the mould. For that reason optimum moisture content of the bricks were kept 25%. Green crushing strength at optimum water content (25%), of bricks sample was 0.40 kg/cm² (Fig. 2)

\[
\text{Absorption} \times 100 = \left( \frac{W_2 - W_1}{W_1} \right) \times 100
\]

Where \( W_1 \) is the weight of specimen after complete drying at 105°C and \( W_2 \) is the weight of specimen after soaking. Data were given in Fig. 4.

F. Test method

1) Compressive strength: Compressive strength of samples of different curing time were done in low range UTM (Company: SHIMADZU, Type: SBL-5Kn, P/N 340-43120-01, Capacity: 5kN/500kgf Cell code: 8). Data were given in Fig. 4.

![Graph of green crushing strength with moisture content](image)

\[ \text{Green Crushing Strength (Kg/cm2)} \]

\[ \text{Moisture addition (\%)} \]

![Graph of properties at different curing period](image)

2) Water Absorption: Water absorption tests were conducted according to 'ASTM C67-07,' [47] to study the capability of specimens in absorbing water, which depends on the microstructure and porosity of the specimens. Besides that, water absorption can be an indicator of the degree of geo-polymeric reaction. The geo-polymer brick specimens prepared at 25% initial water content and cured in different way for 7 days to 21 days were soaked in water and weighed. Before weighing the soaked specimens, the wet surface was dried with a damp cloth. The percentage absorption was calculated as follows

\[ \text{Absorption} (\%) = \left( \frac{W_2 - W_1}{W_1} \right) \times 100 \]

Where \( W_1 \) is the weight of specimen after complete drying at 105°C and \( W_2 \) is the weight of specimen after soaking. Data were given in Fig. 4.

3) Porosity: Porosity determined with the help of HTBW method [48]. Values were given in Fig. 4.

4) Microstructure and Phase: To investigate the effect of curing time on the microstructure and phase composition of the geopolymer bricks, SEM imaging were performed. The SEM imaging of geopolymer specimens was performed in the conventional mode using the (FEI Quanta-200FEG) at 20kv on scan rate 10μs with ETD detector microscope. The freshly failed surfaces from the unconfined compression tests, without polishing to keep the fractured surface “un-contaminated”, were used for the SEM imaging. (Fig.5 and 7) The EDAX analysis was performed with for the samples having minimum and maximum strength (i.e. 7 days and 21 days.). Different phases are shown in figure No. 6 and 8.
Fig. 5 SEM Micrograph of 7 days cured sample

Fig. 6 Silica in free phase (A) and less amount of only CASH phase (B and C) formed in the bricks at initial stage (determined by EDAX analysis)

Fig. 7 SEM Micrograph of 21 days cured sample

Fig. 8 Formation of CASH (A and D), CSH (B), Less amount of Silica phase (C) and phase formed in the 21 days cured bricks
The main agents of deterioration require the presence and movement of water within the material itself. The presence of water can cause freeze-thaw damage to the product. Furthermore, water can carry chlorides and sulfates as well as other harmful ions. Hence, the absorption of the product has a great effect on its durability. Fig. 4 shows the water absorption values (%) for the manufactured bricks at different curing period. It can be observed that the water absorption of the manufactured bricks decreased with increasing curing time. Bricks were made without applying adequate external pressure so the water absorption and porosity was more but the strength was more than bricks produced with same composition by NTPC [42].

IV. CONCLUSION

From the results of the investigation on the preparation of high strength lime activated fly ash bricks with addition of sand powder, the following conclusions can be drawn. Lime activated unfired fly ash bricks can be made by utilizing industrial wastes such as fly ash to prevent earth shell. Unfired bricks are more environments friendly and less CO₂ emission as well as less power consumption. For curing unfired bricks only dip method in water can also give the sufficient strength as much as other complicated curing methods which can be beneficial for the lower capital investment bricks producer. The formation of CASH in the initial stage is responsible for the initial strength formation. After increasing curing time more new phases formed such as CSH, CAH etc, which decreased the free silica present inside the bricks resulting more strength. The crushing strength can be further increased by increasing molding pressure applied by machine molding.

REFERENCES

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