Development and optimisation of curing temperature of energy-efficient geopolymer bricks

Present study focuses on development of a sustainable geopolymer brick in which the use of fly ash is maximised and the concentration of an alkaline solution without cement is reduced, in order to reduce environmental burden and carbon emissions. The geopolymer bricks were developed using the Class F fly ash with 2 to 4 Molar alkaline solution. The bricks were cured for one hour at temperatures ranging from 100 to 600 °C. The compressive strength of such bricks amounts to 10.2 MPa and was achieved in the 4-Molar alkaline solution at the curing temperature of 400 °C. The geopolymer bricks exhibit crystalline structure and good thermal insulation properties.

Key words: geopolymer brick, fly ash, alkaline solution, curing temperature, physicomechanical properties

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U radu prikazan je razvoj održive geopolimerne opeke primjenom maksimalno dopuštenog udjela letećeg pepela i manjom koncentracijom alkalne otopine bez cementa u mješavini, kako bi se u konačnici smanjio negativan utjecaj na okoliš i emisija stakleničkih plinova. Geopolimerne opeke razvijene su primjenom letećeg pepela klase F s korištenjem 2 do 4 M alkalne otopine. Opeka se suši tijekom jednog sata na temperaturi koja varira od 100 do 600 °C. Tlačna čvrstoća takve opeke iznosi 10,2 MPa, a postignuta je u 4 M alkalnoj otopini na temperaturi od 400 °C. Geopolimerna opeka ima kristalnu strukturu i sposobnost toplinske izolacije.

Ključne riječi: geopolimerna opeka, leteći pepeo, alkalna otopina, temperatura sušenja, fizikalnomehanička svojstva

Prethodno priopćenje

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Razvoj i optimalizacija temperature njegovanja energetski učinkovite geopolimerne opeke

In der Abhandlung wird die Entwicklung nachhaltiger geopolymorer Ziegel durch Anwendung des maximal zulässigen Anteils an Flugasche und einer geringeren Konzentration an alkalischer Lösung ohne Zement in der Mischung vorgestellt, um letztendlich die negativen Auswirkungen auf die Umwelt und die Treibhausgasemissionen zu verringern. Geopolymer Ziegel wurden durch Anwendung von Flugasche der Klasse F mit Verwendung von 2 bis 4 M alkalischer Lösung entwickelt. Die Ziegel werden innerhalb einer Stunde bei einer Temperatur im Bereich von 100 bis 600 °C getrocknet. Die Druckfestigkeit solcher Ziegel beträgt 10,2 Mpa, und wird bei einer 4 M alkalischen Lösung bei einer Temperatur von 400 °C erreicht. Geopolymer Ziegel haben eine Kristallstruktur und die Fähigkeit zur Wärmédämmung.

Schlüsselwörter: geopolymer Ziegel, Flugasche, alkalische Lösung, Trocknungstemperatur, physikalisch-mechanische Eigenschaften
1. Introduction

Manufacturing of new building materials that promote sustainable development is highly needed in construction industry in developing countries. The maximum use of industrial waste like fly ash is necessary to reduce environmental burden. Manufacturing of one ton of Portland cement generates 0.95 tons of CO₂. On the other hand, geopolymer material results in lower CO₂ emissions in the range of 40-80 % as it does not rely on calcium carbonate [1]. The geopolymer material is an inorganic polymer obtained by mixing the alkaline solution with aluminosilicate-rich materials such as fly ash. Polymerization products exhibit some advantages such as higher physical strength, higher thermal insulation properties, lesser degradation under chemical attack, and lower shrinkage, when compared to conventional concrete [2]. Compressive strength of geopolymer mixtures improves with the percentage of Na₂O present in the alkaline solution, which is required for bonding with siliceous material present in fly ash for the polymerization process [3]. Currently, many brick manufacturers are producing bricks with cement and crushed sand based fly ash bricks, clay bricks, concrete blocks, autoclaved lightweight blocks, etc. using various curing methods such as steam curing, heat curing, and pressurized autoclaved steam curing. However, commercial production of geopolymer bricks has not been started due to higher molarity of alkaline solution, heat curing temperature, and duration of curing process. Jeyasehar et al. [4] produced bricks with granulated blast furnace slag (GGBS) and 5 M and 12 M solution with steam curing at 60 ºC for 24 hours, and the observed strength amounted to 16.11 MPa and 12 MPa, respectively. Sivasakthi M. et al. [5] studied how silica fume enhances mechanical strength of the geopolymer paste, mortar and concrete for up to 45 MPa during temperature curing at 800 ºC for 2 hours. Ibrahim et al. [6] stated that the alkaline activator concentration improved properties of the geopolymer concrete mix and that the observed compressive strength amounted to 20.3 MPa in 12 Molar alkaline solution during temperature curing at 70 ºC for 24 hours. Okoye et al. [7] achieved the compressive strength of 58 MPa with the addition of silica fumes and 14 molar NaOH concentration at the curing temperature of 100 ºC. Silva et al. [8] established that the CEB masonry stabilized blocks consist of 10 % fly ash, 90 % soil and geopolymer solution at 20 ºC curing temperature. Sukmak et al. [9] developed geopolymer bricks with fly ash to clay ratio of 0.3, involving a 10 molar alkaline solution that was cured at 65 ºC for 48 hours. The compressive strength increased with the heat energy up to a certain limit and, beyond that limit, the formation of micro-cracks was observed. According to previous studies, it can be stated that many authors work on a higher concentration of alkaline solution, from 5 molars to 14 molars, higher curing heat of up to 800 ºC, and longer heat curing time, usually ranging from 24 to 72 hours. The present study aims to minimize the use of concentration of an alkaline solution with maximum fly ash to develop the geopolymer brick. The study evaluates the effect of curing temperature on geopolymer bricks with 80 % of fly ash, 10 % of micro silica, and 10 % of clay mineral, without cement and fine aggregate. The effects of alkali solution and curing condition on mechanical properties of geopolymer bricks were studied. The brick was cured at different temperatures varying from 100 to 600ºC for one hour. Hence, this research is aimed at reducing environmental burden by creating building bricks that are both energy efficient and cost-effective.

2. Materials and manufacturing process

2.1. Materials

In this research, Geopolymer fly ash bricks were manufactured with the class F fly ash, clay, micro silica, and alkaline solution (sodium silicate + sodium hydroxide). Physical and chemical

| Physical analysis | Fly ash | Micro silica | Clay |
|-------------------|---------|--------------|------|
| Fineness [m²/kg]  | 329     | 19500        | 416  |
| Soundness        | 0.08    | 0.095        | -    |
| Specific gravity | 2.23    | 2.15         | 2.66 |
| Chemical analysis|         |              |      |
| Silica content [SiO₂] by mass [%] | 52.32 | 95.12 | 59.2 |
| Ferric oxide + Aluminium Oxide by mass [%] | 32.29 | 0.73 | 21.1 |
| Calcium Oxide [CaO] by mass [%] | 5.83 | 0.24 | 4.37 |
| Sulfate [SO₃] by mass [%] | 0.15 | 0.13 | 0.3 |
| Magnesia [MgO] by mass [%] | 1.57 | 0.53 | 0.83 |
| (Na₂O + K₂O) by mass [%] | 0.04 | 0.41 | 0.89 |
| Loss on ignition by mass [%] | 4.48 | 1.9 | 8.4 |
Development and optimisation of curing temperature of energy-efficient geopolymer bricks

The properties of materials used were analysed as per Indian Standard IS 1721: 1967 [10]. The fly ash was procured from the National Thermal Power Corporation, Sipat, India (NTPC Sipat). Physical and chemical properties of fly ash, micro silica, and clay, are shown in Table 1. Properties of sodium hydroxide and sodium silicate used in this study are given in Table 2 and Table 3, respectively. The alkaline solution was formed by mixing NaOH and Na₂SiO₃ at the ratio of 1:1.5. This ratio of Na₂SiO₃/NaOH was taken from the past research work conducted by P. Sukmak et al. [9], Unnati et al. [11], etc. The molarity of solution was determined via concentration of NaOH per litre of water.

Table 2. Properties of sodium hydroxide

| Property   | NaOH  |
|------------|-------|
| Specific gravity | 2.18  |
| Purity [%]  | 96.1  |

Table 3. The properties of Sodium silicate

| Property   | Na₂SiO₃ |
|------------|---------|
| Na₂O       | 13.53   |
| SiO₂       | 35.24   |
| Voda       | 44.76   |
| SiO₂ / Na₂O | 2.60   |
| Specific gravity | 1.68  |

3. Manufacturing of geopolymer bricks

3.1. Geopolymer brick manufacturing procedure

The fly ash and clay were fired at 100 °C for 24-hours to remove moisture. The alkaline solution with different molarity was prepared. The alkaline solution was preheated at 60–70 °C before mixing in dry material to accelerate the geo-polymerization reaction. Subsequently, the dry material was mixed with alkaline solution for complete homogeneity. Materials needed to produce 1 m³ of geopolymer bricks with the desired ratio are given in Table 4. The alkaline solution to dry material ratio (L/M) and molarity are significant parameters for the enhancement of strength parameters. The NaOH to Na₂SiO₃ ratio was fixed to 1:1.5 based on a previous study, and the alkaline solution to dry material ratio was fixed to 1:4 [6]. The geopolymer brick mix was transferred to the hydraulic press brick making machine. Geopolymer bricks were produced in the size of 200 mm ×100 mm ×75 mm, and they were kept in moulds for 24 hours to set before demoulding. Then, the geopolymer bricks were heat-cured in an electric oven at 100 to 600 °C for one hour. The flow diagram of the geopolymer brick manufacturing process is shown in Figure 1. Manufactured geopolymer sample bricks are shown in Figure 2.

Table 4. The materials needed to produce 1 m³ of geopolymer bricks

| Material     | Quantity |
|--------------|----------|
| Fly ash      | 0.959 kg |
| Clay         | 0.419 kg |
| NaOH (1:1.5) | 0.240 kg |
| Na₂SiO₃      | 0.360 kg |
| Total        | 1.975 kg |

Figure 1. Geopolymer bricks manufacturing process

Figure 2. Manufacturing of geopolymer bricks
4. Results and discussion

A total of 64 brick samples measuring 200 x 100 x 75 were prepared for each combination in order to test compressive strength, flexural strength, shear bond strength, water absorption, and efflorescence, and nine prepared samples were tested in the scope of each test. Nine samples 100 mm in diameter and 10 mm in thickness were cast for each combination to determine thermal conductivity. One extra brick sample was cast for the SEM and XRD analysis of geopolymer bricks. These specimens were cured at temperatures varying from 100 to 600 ºC for one hour, and were then kept for 28 days at ambient temperature. The following physico-mechanical tests were performed after 28 days of resting time on geopolymer bricks, as discussed in the following sections.

4.1. Density of geopolymer brick

The density of geopolymer bricks heat-cured at different temperatures, and at different molarities of alkaline solution, was calculated. The higher the molarity and temperature the greater the degree of geopolymerisation process and, hence, the lighter the geopolymer material produced.

Figure 3 shows that density decreases with an increase in molarity and temperature. The density of conventional fired clay bricks was 1650 Kg/m^3. The density of geopolymer bricks is smaller compared to conventional clay bricks.

4.2. Compressive strength of geopolymer bricks

The compressive strength of all developed geopolymer bricks was tested as per Indian Standard BIS 3495: 1992 Part 1 [12]. The compressive strength of geopolymer bricks enhances with geopolymerisation reaction [13] and determines the strength and quality of construction materials [14]. Hence, significant reduction of heat curing time is possible by increasing the curing temperature up to 400 ºC [4, 8]. Various studies have shown that the analysis focuses on high concentration of alkaline solution to achieve the desired strength parameter. No research has been done for low molarity i.e.1–4M. Jeyasehar et.al. [5] achieved the compressive strength of 16.11 MPa for 12 M solution for heat curing at 60 ºC for 24 hours, and Sivashakthi et al. achieved the compressive strength of 28 MPa strength for 12.5 M liquid solution for heat curing at 800 ºC for two hours. Thus, in both studies the concentration of alkaline solution was not varied but the curing temperature was increased from 60 ºC to 800 ºC and the curing time was reduced from 24 hours to 2 hours, and the compressive strength increased by 73 %. In the present study, the research concentrated on the development of geopolymer bricks with minimum concentration of alkaline solution, minimum time of heat curing, and reduced heat curing temperature, without compromising the strength criteria required as per Indian Standard code. The FB11 composition provided 10.2 MPa compressive strength at 4 Molar concentration with 400 ºC curing for one hour.
4.2.1. Compressive strength variation with temperature

The molarity of alkaline solution plays a decisive role in compressive strength assessment. The combined effect of molarity and temperature on compressive strength due to increased geopolymerisation process is studied. Figure 4 shows that compressive strength increases with an increase in molarity. The rate of strength gain was lower at low molarity, whereas the rate of strength gain increased at higher molarity. The rate of compressive strength gain with respect to temperature is given in equations (1) and (2) for 4 Molar alkaline solution and 3 Molar solution, respectively.

\[
\begin{align*}
CS_3 &= 0.77 \cdot T + 1.77 \\
CS_4 &= 2.29 \cdot T + 1.55
\end{align*}
\]

where:
CS - compressive strength
T - temperature.

Figure 4 shows that the compressive strength increases with an increase in heat curing temperatures from 100°C to 400°C for the duration of one hour, and molarity from 2 to 4 Molar. Further increase in curing temperature beyond 400 °C resulted in the occurrence of micro-cracks on the surface of the brick. A reduction in compressive strength of geopolymer bricks was observed at 600 °C curing temperature due to development of micro-cracks, as shown in Figure 5. The cracked bricks cannot be used in construction industry for masonry wall structures as per IS1077:1992 [14].

4.3. Flexural strength test of geopolymer bricks

Geopolymer bricks were subjected to flexural strength testing with one-point load, as shown in Figure 7. It can be seen in Figure 6 that flexural strength increases with an increase in molarity and curing temperature up to 400 °C.

On the other hand, an increase in curing temperature above 400 °C decreases the strength due to crack formation in the geopolymer brick material due to overheating. The FB7 composition exhibited 1.87 MPa and maximum for FB11 composition was 1.97 MPa. The geopolymer brick met the minimum flexural strength requirement criteria for Class-2 bricks (70 Kg/cm²) as per IS 4860:1996 [16]. The relationship between the compressive strength and flexural...
strength is plotted as shown in Figure 8. For developed geopolymer bricks, the compressive strength was marked as X and flexural strength as Y. The intercept was 1.3831 and slope of the equation was 0.1057. The flexural strength of geopolymer bricks increased with an increase in compressive strength. The flexural strength of conventional fired clay brick amounted to 0.86 MPa. The developed geopolymer bricks showed higher flexural strength compared to conventional clay bricks.

Figure 8. Relationship between compressive strength and flexural strength

4.4. Shear bond strength

The masonry structure undergoes different types of loading such as wind load and seismic load at different angles, which causes shear of brick wall masonry. The strength of masonry bond is influenced by parameters such as characteristics of masonry units and mortar, surface texture of brick, porosity etc. [17]. The shear bond strength testing on a geopolymer triplet brick prism was conducted as per ASTM C1314-16 standard [18]. The failure pattern of a geopolymer brick subjected to the shear bond strength testing is shown in Figure 9. The failure pattern involved partial block and interface mortar failure. It can be seen in Figure 10 that the bond strength of geopolymer brick amounted to 0.44 MPa and 0.38 MPa for FB11 (4 M, 400 ºC) and FB7 (3 M, 400 ºC), respectively. In the scope of previous studies C. Christy et al. achieved the bond strength of 0.3 MPa on fly ash brick [19], while B. V. Reddy achieved the shear bond strength of 0.12 MPa on soil-cement masonry with 5 % cement [17]. It was observed that developed geopolymer bricks have higher shear bond strength compared to conventional fly ash bricks and clay bricks.

Figure 9. Testing arrangement for shear bond strength

4.5. Capillary absorption

The water absorption test was conducted on geopolymer bricks as per IS 3495:1992 part 2 [20]. It can be seen in Figure 11 that capillary absorption of the developed geopolymer brick varied between 14 and 16 % by weight and that it satisfied limiting criteria as per Indian Standard 1077:1992 (less than 20 %) [14]. It was observed that absorption was constant at varying temperatures. With an increase in molarity, the absorption also increased, which is due to formation of a more amorphous NaSH matrix at higher molarity.
4.6. XRD pattern analysis of geopolymer bricks

The X-ray diffractometer analysis was conducted on geopolymer brick samples at the curing temperature of 400 °C. Geopolymer bricks with 3 Molar alkaline solution (i.e. FB7) were analysed, as shown in Figure 10. The peak 2θ degree position is 26.45 and the height counts are 55.60 for 100 % relative intensity, respectively. The calculation of crystallinity by XRD is based on the presumption that the broad peak comes from amorphous phases and that the sharp peak comes from crystal phases [21, 22]. The analysis of XRD-patterns reveals the presence of quartz, hematite, calcite, and various clay minerals, mica minerals in particular. The XRD sharp peak pattern indicated that the geopolymer brick was crystalline in nature.

4.7. SEM analysis of geopolymer bricks

Figure 13 shows the SEM micrographs analysis and the EDS (energy-dispersive X-ray spectroscopy) for the developed geopolymer bricks. The SEM images depict characteristic morphologic features [5]. The SEM images do not show any cracks in the brick sample at 400 °C, which is probably due to the filling with fine fly ash and micro silica particles. The micro silica plays a major role in enhancing the alumina-silicate gel formation by increasing the Si/Al ratios.

It is not recommended to cure the sample above 400 °C, and so the SEM and XRD analysis was not performed beyond 400 °C. The formation of NaSH matrix, dense alumina-silica gel formation, and some unreacted partials of fly ash, were observed at 28 days. The geopolymer matrix formed with Si and Al present in fly ash and micro silica by alkali activation. The unreacted fly ash may improve the compressive strength with time, and can also act as filler to reduce porosity. The EDS spectrum was obtained from the area related to SEM images, as shown in Figure 14. The geopolymer material comprises raw materials containing Si, Al, Na, and O, as major elements with little amount of Ca. The elemental composition and Si/Al ratios were 3.1, which was calculated using the EDS spectral energy data.
4.8. Efflorescence of geopolymer bricks

The efflorescence of geopolymer bricks was tested as per IS 3495: 1992 (part 3) [23]. In the present study, when geopolymers were subjected to efflorescence analysis, a very thin deposit of salt was observed on the surface of bricks. The salt-affected area amounted to less than 10% of the total exposed area of the brick. The efflorescence of geopolymer bricks was reported as SLIGHT efflorescence as per Indian Standard 1077: 1992 [15].

4.9. Thermal conductivity analysis of geopolymer brick

The thermal conductivity test was performed on geopolymer material by using the two hot plate method as per IS 3346: 1980 [24]. The heat flow through the 10 mm thick and 100 mm diameter material sandwiched between 2 slabs/guarded reference plates was analysed as shown in Figure 15.

According to Figure 16, the thermal conductivity of geopolymer bricks varied from 0.42 to 0.46 W/m°K. It was observed in previous studies that the thermal conductivity amounts to 0.43-0.45 W/m K for fly ash geopolymer concrete [25], 1-1.7 W/m K for concrete blocks [26] and 0.4-0.5 W/m K for conventional clay bricks [27]. It was established that the developed geopolymer brick have good thermal resistance properties compared to conventional clay bricks.

Figure 15. Thermal conductivity of geopolymer brick

Figure 16. Thermal conductivity analysis of geopolymer bricks

4.10. Embodied Energy

To reduce environmental burden, the developed geopolymer brick was analysed to determine the embodied energy required for an optimum FB7 composition, as shown in Table 5. Past research shows that the embodied energy of cementitious material from fly ash is reduced to 50% compared to conventional concrete [28]. The embodied energy required for a material is 6.56 MJ/kg for NaOH and 5.37 MJ/kg for Na2SiO3 [29]. The amount of NaOH and Na2SiO3 per brick was 0.06 kg and 0.09 kg, respectively. The electric oven was used for 1-hour heat curing. The embodied energy associated with this electric oven is 3.6 MJ/MW-h [30]. The calculation of embodied energy per brick was 3.89 MJ/brick for FB7 composition, which is less than the embodied energy calculated for conventional clay brick (6.25 MJ/brick).

Table 5. Embodied energy of geopolymer brick and conventional clay brick

| Material          | Unit energy required | Material required | Embodied energy (MJ/brick) |
|-------------------|----------------------|-------------------|---------------------------|
|                   | Fly ash 0 [MJ/kg]    | Geopolymer unit brick | Geopolymer brick | Clay unit brick | Clay brick |
|                   | Clay 0.1 [MJ/kg]     | 1.6               | 0                         | 0              | 0.25       |
|                   | Micro silica 0.1 [MJ/kg] | 0.2            | 2.5                       | 0.02           | 0          |
|                   | NaOH 6.56 [MJ/kg] [29]| 0.06            | 0                         | 0.39           | 0          |
|                   | Na2SiO3 5.37 [MJ/kg] [29]| 0.09          | 0                         | 0.48           | 0          |
|                   | Heating 3.6 MJ/MW-h  | Sušenje sat vremena na 400 °C | Sušenje pet sati na 1100 °C | 3              | 6          |
| Total embodied energy (MJ/brick) | 3.89 | 6.25 |
According to previous results, the embodied energy required for conventional bricks is 7.02 MJ/brick [30]. The developed geopolymer brick is more energy efficient compared to a conventional clay brick.

### 4.11. Economic analysis of geopolymer bricks

A simple economic study performed on geopolymer brick proved that the cost required for one m$^3$ of geopolymer brick using the suggested mix was by about 16.4% lower than that of conventional clay building bricks. The costs associated with an optimized composition of geopolymer bricks, including transportation costs, were calculated as given in Table 6. The cost required per m$^3$ of geopolymer bricks amounted to 24.48 EUR and for conventional brick to 28.5 EUR.

### 5. Conclusions

It was observed that microcracks developed on the bottom surface of bricks during 1-hour heat curing at temperatures of more than 400 °C. As per IS1077:1993, such bricks cannot be used in construction work. It was established in this study that an optimum temperature for heat curing is 400 ºC and that the curing time should be one hour. The compressive strength of geopolymer bricks is proportional to the molarity of alkaline solution i.e. the strength increases with an increase in molarity. However, the FB11 composition exhibited the maximum compressive strength of 10.2 MPa with 4 M solution following the 400 °C heat curing that lasted one hour. The FB7 composition exhibited 4.9 MPa (required minimum compressive strength is 3.5 MPa) as per IS 1077:1992 for the 3-molar alkaline solution and the one-hour 400 °C heat curing, which is an optimum composition compared to other trial mixes. The FB7 composition of geopolymer bricks enhanced the flexural strength, and the X-Ray Diffractometer pattern of FB7 geopolymer brick showed that geopolymer bricks were crystalline in nature. The geopolymer bricks showed good thermal resistance compared to conventional fired-clay bricks, which is why geopolymer bricks can be used as a heat resistant material. The geopolymer bricks were developed by lowering the molarity of alkaline solution, and by maximising the use of fly ash (i.e. 80 %), without the use of cement and aggregate, so as to reduce environmental burden. Reduction of curing time to one hour is expected to speed up brick production in construction industry. The developed geopolymer bricks required less embodied energy than conventional fired-clay bricks. Finally, an economic study of geopolymer bricks revealed that the suggested mix costs about 16.42% less compared to conventional bricks. These geopolymer bricks are a sustainable, energy efficient, and economical solution for use in construction industry.

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