CONVERSION OF ALUMINO-SILICEOUS REJECTS FROM POWER AND STEEL INDUSTRIES INTO BINARY GEOPOLYMER BRICK: EXPLOITING-GBFS MATERIAL SYNERGY UNDER AMBIENT CONDITIONS FOR SUPPORTING CIRCULAR ECONOMY

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ABSTRACT

In recent year, advances in construction material have enabled the utilization of abundant reject material for value added purposes. The mining and metallurgical industries, particularly those with mineral rich rejects, have emerged as alternative sources of material for the construction application. The combination of granulated blast furnace slag (GBFS) from the steel industry and fly ash (FA) has been identified as one of the most suitable materials combinations for producing geopolymer blocks without the need for reinforcement or with the additives. Binary blended FA and GBFS based geopolymer bricks were cast with variable raw precursors and activator ratio using mini brick plant setup and cured at ambient temperature. The decisive parameters based on Indian standards for which the testing was done are compressive strength, water absorption, efflorescence and density. The compressive strength achieved for the test cubes casted for prism test was in the range of 20 - 30 MPa at ambient temperature. The bricks were found to meet the requirements established by Indian Standard 1077:1992 for producing energy-efficient masonry products. The cost and embodied energy (EE) of the developed geopolymer bricks were estimated. As being a binary blend of FA and GBFS cured at room temperature, the developed bricks could reduce the EE and carbon dioxide emission (CO_2) and energy required for heat curing. The comparative analysis for the conventionally available bricks with the geo-brick showed promising results. Utilization of steel and power industry waste is a way to achieve circular economy in the waste.

Keywords: industry reject, material synergy, geopolymer, physical properties, value addition, prism test, embodied energy.

INTRODUCTION

As per the United Nations Environment program, 30 % of the consumption of natural resources and 40 % of energy usage is by the built environment [1]. The growing population has created the need for more and more basic requirement of housing and other infrastructure. The factual demand continuously putting burden on the manufacture of building materials and consumption of natural raw materials. The increasing demand of building materials and quick resource of raw materials turned the attention towards use of industrial rejects and alternate construction materials. Conversely, due to increased urbanization and industrialization, the by-products from various industries have been a key concern considering recycling and waste management.

During the process of generating electricity in coal-based thermal power plants, fly ash (FA), a byproduct of combustion, is produced. It is currently used to make Portland Cement, bricks, blocks, tiles, and road embankments. It is a resource material that has been used successfully in numerous building industry applications. Coal/lignite-based fly ash generation in India in the 1st half of the Year 2021 - 2022 is 133 million tons and demanding large areas of land for disposal [2]. Fly ash contains appreciable amount of silica and alumina which could lead to acceleration of good geopolymerization reaction when it is mixed with an alkali activator.

The process of iron making is the oxidation-reduction process of iron ore in blast furnace to produce metallic iron. The easy alkali activation property of Granulated Blast Furnace Slag (GBFS) formed in cement making is considered as one of the most suitable raw materials for preparing alkali activated material, which consumes less energy when used to replace OPC as binder [3]. The primary components of Indian slags contain an equilibrium quantity of silica to calcium and are effective in producing calcium-silico compounds, sodium silicate, and alumina silicate.

According to earlier studies, materials high in silicon (such fly ash or slag) and aluminous minerals are necessary for geopolymerization to take place [4]. Geopolymer development with different types of industrial ash in combination with metallurgical slags which contain good amount of silica and alumina proved to be the better option for preparing alkali activated / geopolymer based building materials for voluminous utilization of the reject materials. The temperature range of 50 to 80°C was commonly accepted as the ideal range for successful geopolymer hydration. The curing temperature and curing time directly affect the specimen's final compressive strength values [4].

To produce geopolymer brick, blocks, mortar, concrete, previous research has demonstrated the utilization of fly ash as a precursor along with ground granulated blast furnace slag (GBFS), sand and red mud [5 - 8]. Class-C fly ash (FA) and powdered granulated blast-furnace slag based geopolymers activated in NaOH and NaOH + Na_2SiO_3 were examined regarding setting time, compressive strength, porosity, microstructure, and crystalline phase development [9]. The findings showed that NaOH had less of an impact on the development of strength and a denser microstructure than did NaOH + Na_2SiO_3, since Na_2SiO_3 adds the silica source needed to create a more compact structure. With the addition of more fly ash, the class-C FA and GBFS mixes had a longer setting time, less strength, and a loose matrix. The strength loss observed by the increased level of fly ash due to un-reactivity of calcium in the mix. According to the XRD patterns, the calcium in fly ash had no effect on how the C-Si-H bond formed.

Table 1 details out the geopolymer composition considering FA and GBFS as a precursor either at room temperature or at elevated temperature. FA and GBFS based geopolymer mainly used ternary combination of mix with either sand, aggregates, quarry dust, clay and admixtures like iron chips, nano silica [10 - 14]. These ternary blends showed promising results for strength, water absorption, thermal conductivity etc. FA-GBFS based geopolymer with 2 % amorphous nano silica resulted in 13 % improvement in compressive strength [12]. It was very well researched that geopolymer utilization will help to reduce the CO_2 emission by elimination of cement. When the ternary blended geopolymer were developed it mostly uses natural resources such as sand, aggregates or other costly additives and admixtures like nano silica. But it was established that those additives are contributing for embodied energy and CO_2 emission as well (Fig. 1).

The research activities discussed here largely focused on the development of high strength geopolymer brick at ambient curing condition primarily using binary blended with GBFS and fly ash only, to further reduce the Embodied Energy and CO_2. The geopolymer brick were cast using synergy of GBFS and FA as a source material at ambient temperature. Compressive strength, density, embodied energy, and cost are used to evaluate the viability of various geopolymer mixtures.

EXPERIMENTAL

The main ingredients for geopolymer brick development were: Granulated Blast Furnace Slag (GBFS), Fly ash, Sodium Hydroxide (NaOH in 6 M and 10 M); Sodium Metasilicate (Na_2SiO_3). For the study, GBFS were collected from Bhilai Steel Plant, Chhattisgarh in granulated form and prepared 100 % mesh fractions for better homogeneity and reactivity. Fly ash was collected from Nagpur, Maharashtra (Koradi and Khaperkheda power plants).

The activator media was made using Na_2SiO_3 (Qualigens, India) and NaOH (98 % purity, Molarity 40 g mol^{-1}), all of which are commercially available. Because it is readily available and inexpensive, sodium hydroxide was chosen as the primary alkaline solution for the manufacture of the activator medium. To create a solution with the desired concentration, the solids must be dissolved in water. Six M and 10 M of aqueous NaOH

Table 1. FA and GBFS based geopolymer composition.

Sr. No.	Raw material	NaOH	Fluid/ Binder	NaOH/ Na_2SiO_3	Curing conditions	Compressive strength	Ref.
1	Fly ash, GBFS	8M, 12M	0.3, 0.35 and 0.40	1:2	Room temp.	-	[6]
2	FA and GBFS	6, 8, 10, 12 M	0.2	1.5	Room temp.	27 MPa	[7]
3	Fly ash, GBFS, Aggregate Sand, quarry dust	10 M	0.65 - 0.75	2.0	Open air and 60°C in oven for 24 h	41.6 MPa	[10]
4	75 % GBFS & 25 % FA, sand coarse aggregate	5 M	-	-	Room temp.	60.5 MPa	[11]
5	FA : GBFS (1:1): nano silica (1 - 2 %)	3	0.3	0.48	Room temp.	62-72 MPa	[12]
6	FA 20 % GBFS 60 % GWS 20 % Sand 80 % Iron chips 20 %	10 M	2.5	-	Room temp.	10.1 MPa	[13]
7	FA: 20 %, clay / GBFS 70 : 10, 50 : 30, 30 : 50, and 10 : 70	8, 10M Calcium hydroxide (4, 8 %)	-	-	-	Thermal Cond 0.26 W mK^{-1}.	[14]
8	FA: GBFS (1:1), sand, gravels, Sand, wollastonite fiber	12 M	-	0.8	Room temp.	47 MPa	[15]
9	FA, GBFS, (60 / 40; 70 / 30), aggregates	8M	-	2.5	60°C in oven for 24 h	62.19, 42.36 MPa	[16]

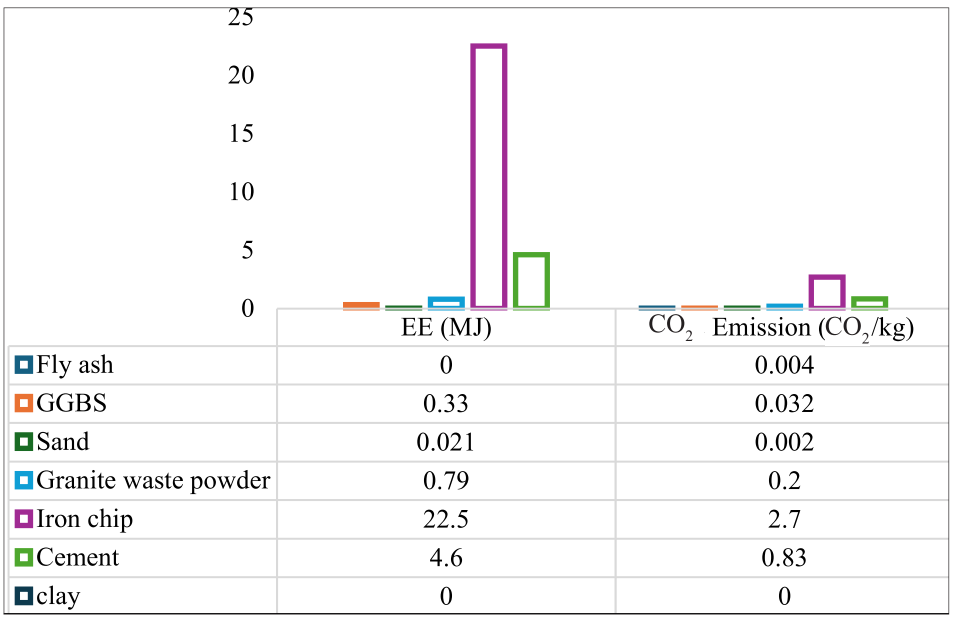

Fig. 1. Embodied energy and CO_2 emissions for raw precursors used in geopolymer [13].

solution were the two different concentrations used for activation. Depending on the solution's concentration, the mass of NaOH solids fluctuates. Chemical, mineralogical, and morphological analyses of the raw material's powdered fractions were performed. Standard wet analytical techniques [17 - 19] were used to ascertain the chemical characterization of the primary components present in the raw material. Mineralogical studied were carried out by X-Ray Diffraction (XRD) (Table 2). Minor elements in specific samples were determined by Inductively Couple Plasma Spectrometer (ICP), model IRISIntrepid II XDL, Thermo at JNARDDC, Nagpur. Chemical analyses of raw materials revealed the nature of major and minor elements in the raw materials. Dry density is the ratio of the material's dry weight (W) to its volume (V). For ease of reference, weight is expressed in kg and volume in m^3. In agreement with the IS 2720 [20], the dry densities of the major raw materials observed are listed in Table 2.

Geopolymer Brick development

The dry GBFS and Fly ash were combined at a predetermined weight ratio in the beginning of the synthesis process (Fig. 2, Table 3), and then an activator, made up of a mixture of Na_2SiO_3 and NaOH solution prepared at 1.6 volume ratio. This mixture was then mixed with FA - GBFA binary blend at a alkali activator to solid weight ratio that depended on the composition of the matrix. At JNARDDC, a small pilot facility was put up to cast geopolymer bricks measuring 230 mm x 110 mm x 90 mm. To permit full reaction (i.e., principally the dissolution of reactive phases) between the powder

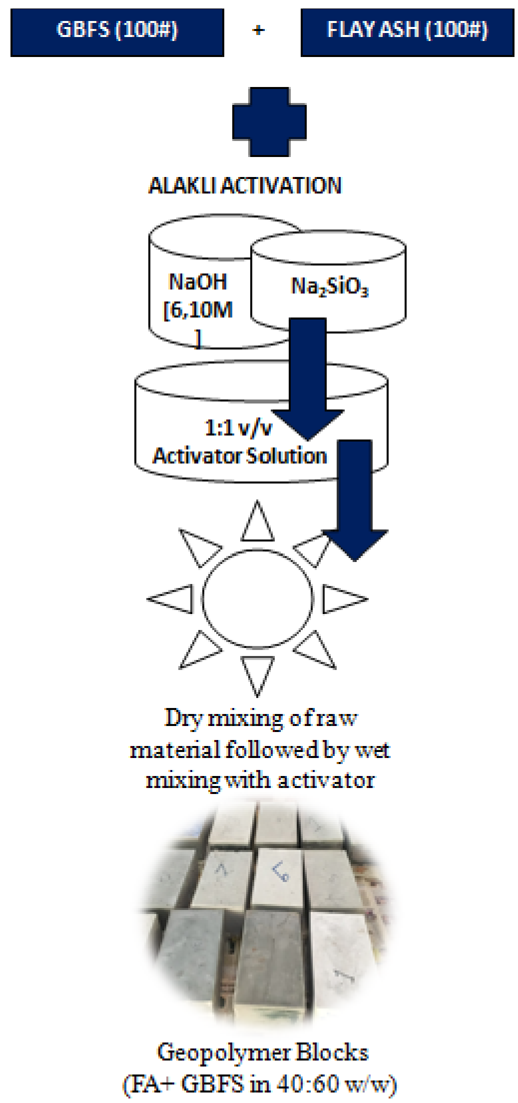

Fig.2. Schematic of process steps for geopolymer development.

Table 2. Chemical composition of FA and GBFS.

Raw Material	Composition, %							Dry density, kg m^{-3}
	Al_2O_3	Fe_2O_3	SiO_2	LOI	TiO_2	Na_2O	CaO	
Fly ash	27.28	5.60	57.06	0.35	1.82	0.24	0.33	1008.75
GBFS	14.28	1.20	33.84	ND	0.53	0.27	37.2	1338.19

Table 3. Compositional variation of geopolymer mix.

Sr. No	Constituent raw material	Abbreviation	Composition, %	NaOH : Na_2SiO_3, v/v
1	GBFS	Geo-Brick 1	100	6 M : 6 M (1 : 1)
2	Fly ash + GBFS	Geo-Brick 2	40 : 60	6 M : 6 M (1 : 1)
3	GBFS	Geo-Brick 3	100	10 M : 6 M (1 : 1)
4	Fly ash + GBFS	Geo-Brick 4	40 : 60	6 M : 2.5 M (1 : 1)
5	Fly ash + GBFS	Geo-Brick 5	40 : 60	10 M : 6 M (1 : 1)

and activator solution, resulting to the development of flowable geopolymer slurry, the mixture was then fully mixed for at least 15 minutes using a magnetic stirring bar at 1 : 1 ratio. To create geopolymer specimens for further mechanical and microstructural analysis, the geopolymer mixture was then placed into a brick mold. After that, the geopolymer brick was given 28 days to cure in a laboratory environment, or at normal temperature and air pressure. The specimens were subsequently demolded, and then they were cured in an exposed environment.

For geopolymer brick development GBFS was primary raw material along with fly ash with alkali activator i.e., sodium hydroxide and sodium silicates. To utilize GBFS to the fullest, mix trials initiated with 100 % GBFS with lower molarity values. Next trails consisting of inclusion of fly ash along with GBFS in 40 : 60 ratio. The considered mix proportion for different geopolymer brick (Geo-Brick) is provided in Table 3.

Testing of Geo-brick

The geopolymer brick was put through several tests for dry density, compressive strength, water absorption, and efflorescence in accordance with the guidelines provided by IS 3495 [21]. The outcomes were analysed in accordance with IS 1077 [22]. Using a Universal Testing Machine (UTM), the compressive test was carried out. For analysis, the average of three samples was estimated. To comply with IS 1905 [23], prism tests were conducted.

RESULTS AND DISCUSSION

Table 2 displays the results of the chemical analysis performed using X-Ray fluorescence (XRF), as well as the dry density of the homogenized GBFS sample and fly ash. Fly ash can be classified as siliceous fly ash (Class F) based on its chemical composition [24]. The GBFS and fly ash raw precursors underwent the XRD examination. The dominant mineralogical phases of the raw material that are most suited for the active formation of geopolymers have been determined using XRD. The major mineral phases identified by XRD in FA and GBFS are quarts (SiO_2), mullite ($2Al_2O_3SiO_2$), calcium oxide (CaO), magnesium oxide (MgO) and alumina (Al_2O_3). For quick alkali-mineral phase interactions leading to gel formation and subsequent three-dimensional rearrangement of silicon and aluminium atoms, materials with higher concentrations of amorphous silica and alumina-containing mineral phases were used. SEM analysis confirmed the presence of the spheroidal particles in FA, but the majority of the GBFS particles have an abnormal shape with distinct edges and angles (Fig. 3).

After 28 days of ambient curing conditions, the generated geopolymer bricks (Fig. 4) were evaluated for compressive strength, water absorption, and efflorescence.

The test cubes were put through a 100 KN load limit under UTM. The minimum average compressive strength specified by IS 3495 is 3.5 MPa. After geopolymerization, it has been found that the material's fineness and density

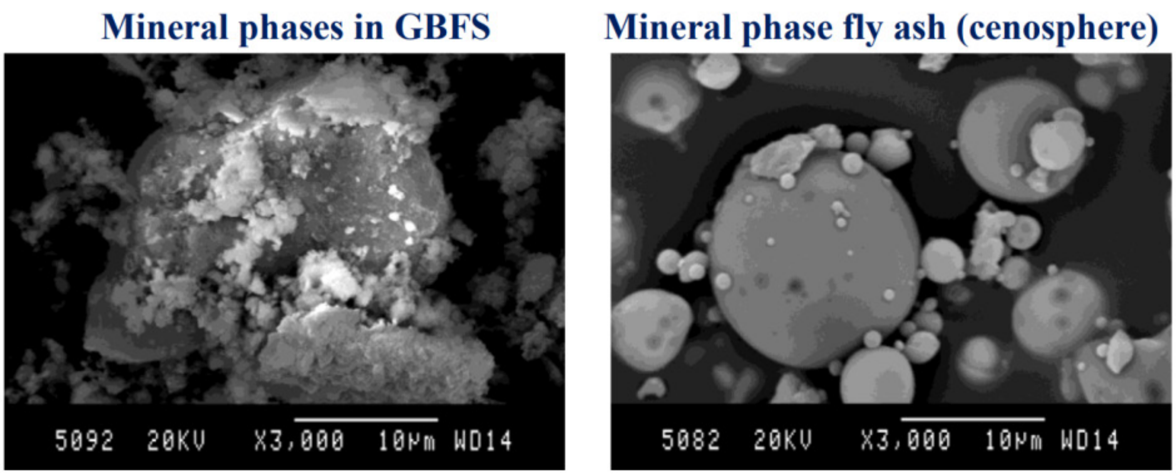

Fig. 3. Mineral phases in GBFS and FA using SEM.

Fig. 4. Geo-brick from FA and GBFS combination.

are crucial in producing a dense composition [21]. Fig. 5 provides the compressive strength of various mix patterns. The maximum compressive strength obtained was 30.35 MPa with GBFS mix Geo-brick 1. The Geo-brick 5 mix composition showed minimum compressive strength of 21.46 MPa which is lying in a class 20 as per IS 1077 [22]. With the addition of 40 % fly ash in GBFS at constant molar concentration (6 : 6), the compressive strength does not change significantly. The change in sodium hydroxide molarity from 6 M to 10 M for GGBF brick (Geo-Brick 1 and Geo-Brick 3) and Fly ash, GBFS composition (Geo-Brick 2 and Geo-Brick 5) resulted in decrease in compressive strength by 18.58 % and 27.88 % respectively. Mix composition of GBFS and fly ash (Geo-brick 1, 2 and Geo-Brick 3, 5) showed that addition of fly ash does not improve upon the strength of brick. These results can be justified by results obtained by Sasui et al. which revealed the synthesis of CSH gel was dependent on the reactive Ca from the GBFS source, leading to the increased strength [9]. While the Ca in the fly ash did not contribute to the creation of C-S-H bonds. One of the variables for enhancing strength and microstructure was the addition of Na_2SiO_3, which hastened geopolymerization by delivering the amorphous silica from the Na_2SiO_3 source. Thus, it was discovered that geo-Brick 4 had decreased compressive strength. When compared to 100 % FA based geopolymer, the produced gels in the GBFS incorporated geopolymers displayed more compact and homogenous morphologies, leading to higher strength behaviour [25].

The percentage of water that a specimen absorbs in relation to its weight is known as water absorption. After being immersed in cold water for 24 h, the bricks must pass the test described in IS 3495 without absorbing more water than 20 % by weight up to class 12.5 and 15 % by weight for higher classes [21]. Except for Geo-brick 3 and 5, every mix composition demonstrated water absorption within the acceptable range [22]. Average density decrement is 7.69 %, 5.91 %, 6.85 % and 6.74 % for Geo-brick 1 to Geo-brick 5 respectively (Fig. 6).

To ascertain whether extra soda (unreacted alkali) was present in the geopolymer specimens made with the chosen mix designs, an efflorescence test was performed. When the bricks were tested using the IS 3495 technique, all the geopolymer mix had a low efflorescence rating (Fig. 7) [21]. Geo-bricks 2 and 5 had low efflorescence, whilst others had nil. Table 4 briefed the overall Indian standard requirement with the achieved results.

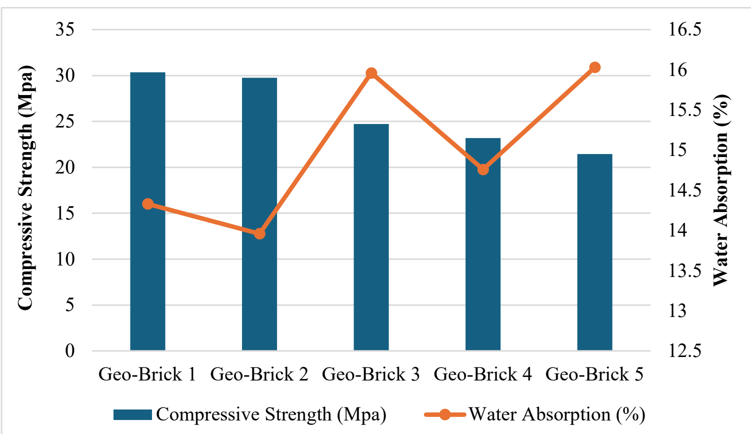

Fig. 5. Compressive strength and water absorption test results for Geo-Bricks.

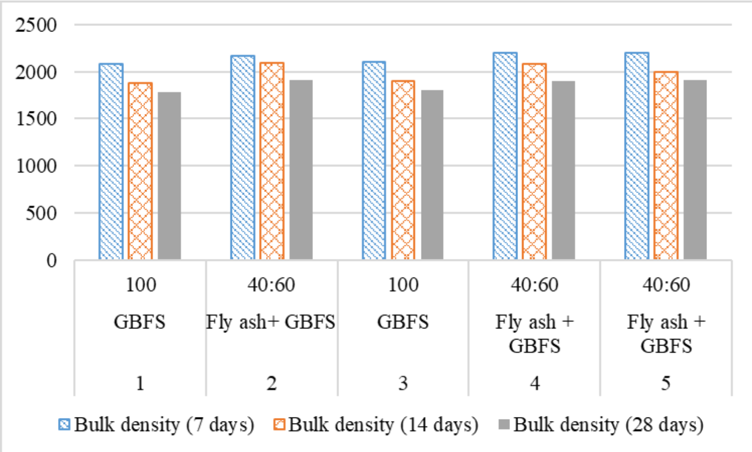

Fig. 6. Density, kg m^{-3} variation with different proportion.

Prism Test

For the field application of the developed bricks, as masonry wall construction becomes expensive, prim test was carried out as per IS 1905 to check the strength of the masonry [23]. The height-to-thickness (h/t) ratio for prism specimens should be between 2 and 5, and the minimum height should be 40 cm, according to IS 1905 [23]. The test is performed at 28 days with the evenly distributed load 350 KN min^{-1} as per IS 1905. During the prism test major focus is on observing the crack patter. The crack pattern in the prism which was observed on the assembly cast as per the requirement (Fig. 8). Fig. 9

Fig. 7. Efflorescence test on Geo-Brick samples.

Table 4. Geo-brick compliance with Indian standards.

Testing Parameters	IS code Specification	Code No.	Obtained Value	Observations
Compressive strength, MPa	The compressive strength should not be less than 3.5 MPa	IS 3495 part I [21]	Approximate 30.35 MPa	Compressive strength is nearly 10 times equal to minimum requirements. Hence can be in the range of High-class bricks
Efflorescence test	Moderate or no efflorescence is acceptable	IS 3495 part II [21]	No Efflorescence	The insoluble salts are not coming on the surface of the bricks. Geopolymer bricks are safe in terms of efflorescence
Water absorption	The average water absorption should not be more than 20 %	IS 3495 part III [21]	Less than 20 %	As less water absorption means it is preventive in terms of dampness
Bulk density	Density should not be more that 2000 kg m^{-3}	IS 2720 [20]	Less than 2000 kg m^{-3}	It satisfies the density criteria of concern IS code

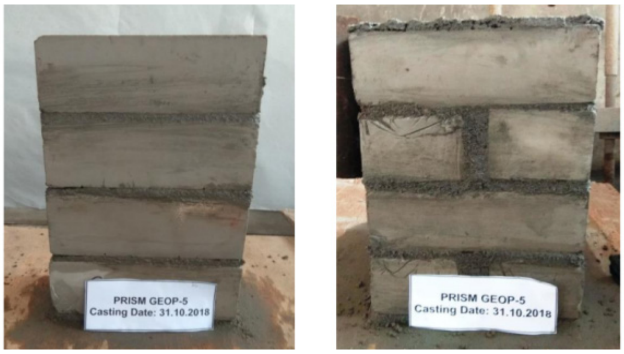

Fig. 8. Single and double brick geopolymer prism.

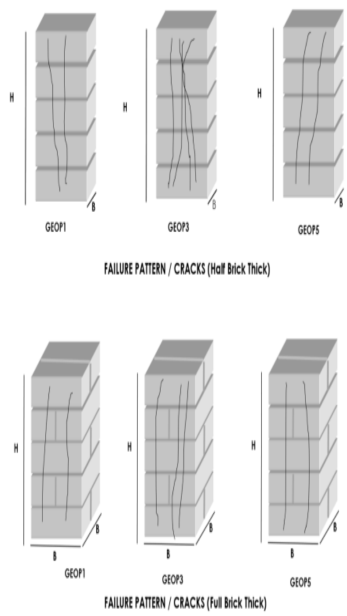

Fig. 9. Crack patter observed in the GBFS-FA geopolymer prism.

showed crack patter in half and full brick thick prism, which is mostly in the middle of the prism vertically. This is because Poisson's effect caused the composite specimen to produce an outward bursting force [26]. Displacement verses load is modelled in Fig.10.

Embodied energy and cost estimation

Four Geo-Brick 1, 2, 3, 5 were considered for embodied energy and cost estimation. Only energy required for the raw material is considered for estimation for energy. Fly ash has no embodied energy because it must be collected from flue gas in India [27]. An embodied energy of 0.31 MJ Kg^{-1} has been considered for GBFS considering grinding after quenching [28]. According to the SPLINE LCI datasheet, sodium hydroxide has an embodied energy of 20.5 MJ Kg^{-1}. It is assumed that sodium silicate has an embodied energy of 5.37 MJ Kg^{-1} [29]. Only GBFS and Fly ash - based brick is used for calculations of cost and embodied energy which showed high strength. The embodied energy for Geo-brick 2 is found to be on lower side followed by Geo-Brick 2 compared to other bricks. Estimated embodied energy of Geo-Brick 2 is 5 % higher than Geo-brick 2 which is having 2 % strength variation. While Geo-brick 3 and 5 showed 33 % and 41 % higher embodied energy compared to the lowest values of Geo-Brick 2.

The cost of Geo-Bricks was calculated based on

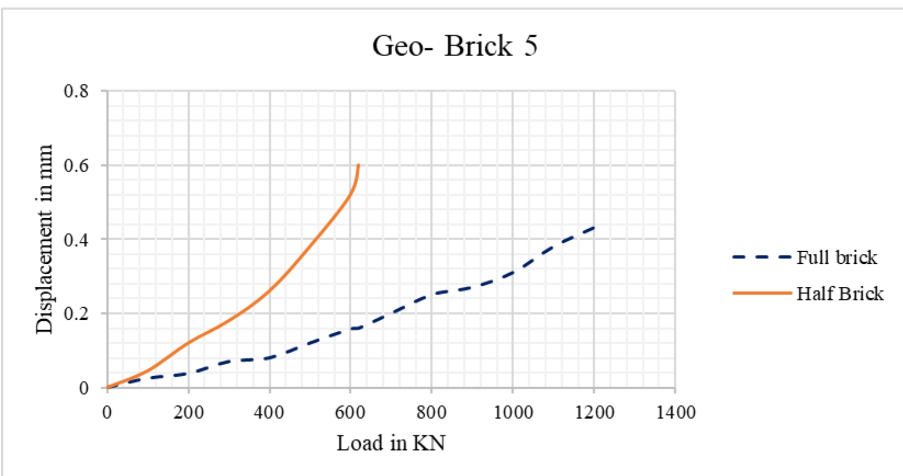

Fig. 10. Displacement vs. load in the GBFS-FA geopolymer prism.

Table 5. Geopolymer brick vs. burnt clay and fly ash cement bricks comparison.

Brick type	Material composition, wt. %					Physical Properties		
	CL	FA	GBFS	Sand	CM	Density, kg m^{-3}	Crushing strength, MPa	Water absorption, %
BCB	90	-	-	10	-	1600	3.5	15
FAB	-	40	-	50	10	1800	6.5	10
Geo-Bric 1	-	-	100	-	-	1779	30.35	14.33
Geo-Bric 2	-	40	60	-	-	1915	29.76	13.96
BCB: Burnt clay brick; CL: Clay; FAB: Fly ash brick; GBFS: Ground granulated blast furnace slag; CM: Cement								

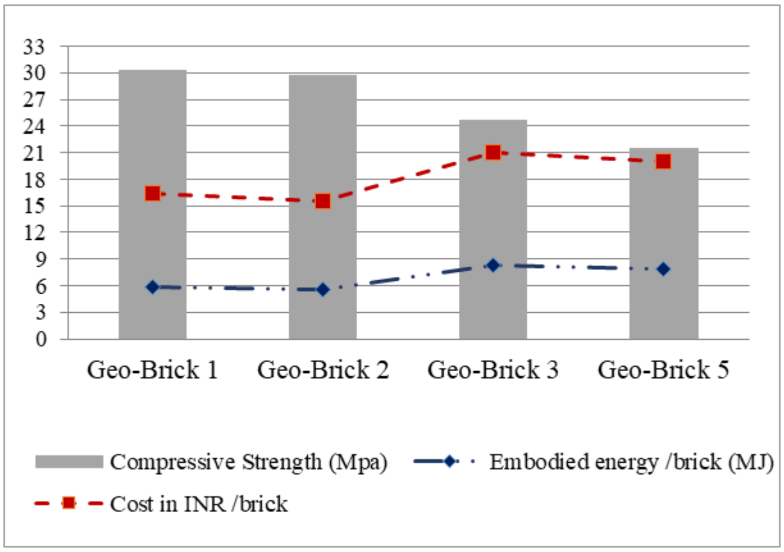

Fig. 11. Compressive Strength, Embodied Energy and Cost per geo-brick.

pilot plant run and the details of cost estimation is shown in Fig. 11 [30]. The cost of expenditure for the plant production of geopolymer brick was calculated based on raw material, energy consumption, transportation cost, labour. It was found that the overall cost of production of Geo-Brick 1 and Geo-Brick 2 is INR 16.4 and INR 15.5 respectively (Fig.11). While for Geo-Brick 3 and 5 cost is slightly high INR 21 and 20 respectively.

The developed brick showed very high strength and fulfilling the basic requirements set by IS 1077 [22]. Though the brick cost is on higher side, but these can be used where achieving high strength is a criterion of the structures. This cost is almost equivalent to other commercially available masonry products (AAC blocks). Geo-Brick 1 and 2 are found to have higher strength, and fulfilled the criteria set by Indian standards also have lower embodied energy, cost. These two bricks are then compared with commercially available Burnt Clay brick (BCB) and Fly ash brick (FAB).

Further comparisons were made between the geo brick and commercially available burnt clay (BC) and fly ash (FA) bricks (Table 5). An earlier study on the BC and FAB's properties was considered [31, 32]. The resulting compressive strength is significantly higher than that of bricks made of burned clay and fly ash. Since the desired characteristics were obtained at room temperature, less energy will ultimately be needed for heat curing. Although water absorption virtually falls within the range allowed by IS regulations. Overall, it is conceivable to produce the developed geopolymer brick at room temperature with reduced molarities utilizing GBFS as the main raw material. These geopolymer bricks can be easily employed for building whenever a higher brick strength is required.

Circular Economy approach for power and steel industries

Cement has proved to be generating a high amount CO_2 emission. Many alternative to conventional ordinary Portland cement, in the form of industrial waste utilization has given promising outcomes. Geopolymer is the one which has strengthened this process of controlling CO_2 emission. Geo-bricks utilized FA and GBFS as primary precursor alone without further inclusion of any additives (sand, nano clay). This type of binary blended geo-brick will surely benefit for increasing waste utilization and geopolymer production. This also provide added value, both economic and environmental to the power section (FA) and steel sector (GBFS) thus supporting a circular economy (Fig. 12). From waste to geo-brick development, achieved many benefits, like potential to replace cement (FA brick as compared to geo-brick) as can be observed from Table 5. Achieving economy, as in utilizing complete waste from two industries, and developing a market ready product is practically possible. This type of approach will surely eliminate deposit at landfill, certify the reuse and recycling of power and steel industry waste.

CONCLUSIONS

The investigation showed that using GBFS to create geopolymer bricks has a lot of potential. In comparison to geopolymer bricks with fly ash added, the increase in wt. % of GBFS led to an increase in compressive strength of geopolymer bricks as high as 30 MPa. The geopolymer bricks developed using 100 % GBFS and 40 % : 60 % fly ash, GBFS have shown almost similar properties as well as lower embodied energy and cost. Overall, it was determined that the bricks were a better alternative to commercially available burnt clay and fly ash bricks due to their high strength. Traditional bricks' environmental pollution and waste management problems can be effectively resolved by using geopolymer bricks made of fly ash and GBFS. The newly created geopolymer bricks are more suited for usage in high strength applications in building.

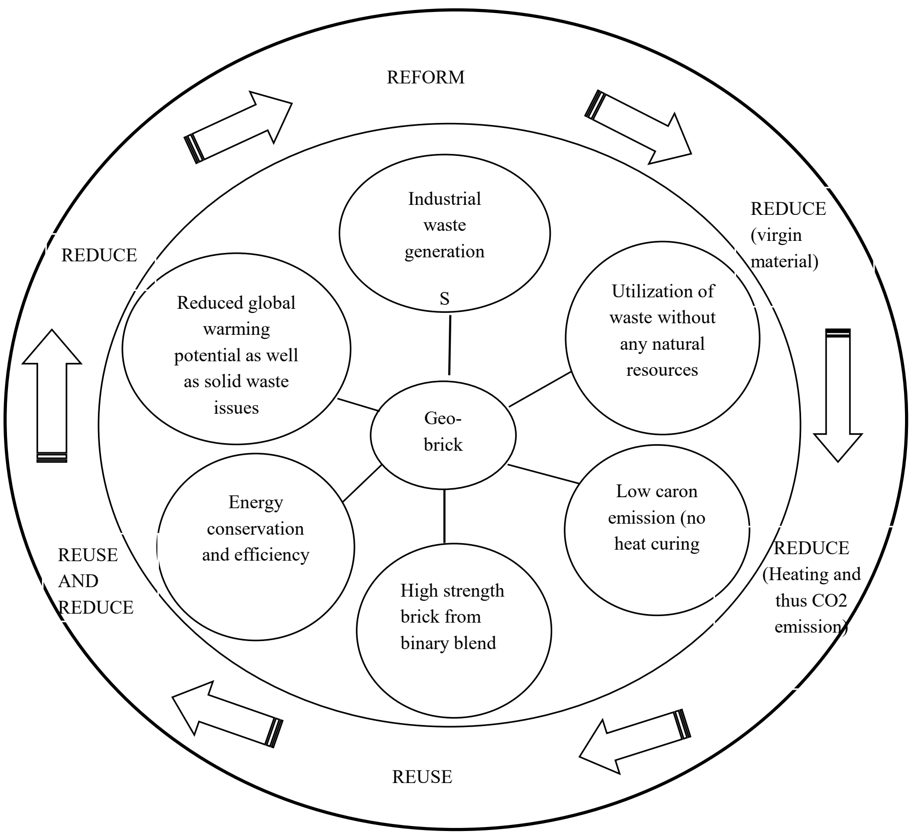

Fig. 12. Circular economy approach for power and steel industries.

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