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Utilization of Iron Ore Mines Waste as Civil Construction Material through Geopolymer Reactions

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Mothi Krishna Mohan, Anirban Roy and Arun Kumar*

Abstract

In India, due to fast pace development there is a drastic growth in the iron and steel industry. As of 2017, India is one of the largest producers of crude steel in the world. This has led to drastic increase in mining activity. Mining activity is responsible for generation of wastes, which can pose threat to the environment and its habitants. However, there is also a great potential for mines wastes to be utilized in construction industry, which can become an important ingredient for sustainable and eco-friendly development. In iron and steel industry, Iron ore tailings (IOT) and slimes utilization is still an area of challenge, because of the low content of iron oxide present in them, which is unsuitable for metal extraction. Usually particle size of slimes below 1 mm is not amenable for further metal extraction through conventional pelletization techniques. In the present study waste from two different iron ore mines have been tried for their utilization as a construction material through geopolymerisation technology. As a primary consideration, shapes made in the form of common bricks were tested for their densification behavior, compressive strength and water absorption. To reduce the cost, industrial wastes like fly ash, ground granulated blast furnace slag, and lime were tried in different batch compositions in addition to sodium silicate and sodium hydroxide. Relationship between compressive strength values with individual ratio of silica to alumina (Si/Al), silica to alumina with iron combined (Si/Al + Fe), and calcium to silica (Ca/Si) were developed. Based on the elemental ratios, critical threshold values were established that showed significant effect on the compressive strength of the final composite.

Keywords: IOT, geopolymer, GGBS, calcium, fly ash

1. Introduction

Numerous scientific studies indicated superiority of the ancient concrete as being much more durable than their modern counterparts made with ordinary portland cement. It has been observed that calcium silicate hydrate formed as a result of the hydration of modern portland cement deteriorates, while the ancient cement remains intact under identical conditions. French scientist, Davidovits [1] proposed

that the durability of ancient concrete was the result of the presence of alkaline aluminosilicates in the structure. Davidovits named this new class of cementitious material as geopolymers. This is a new generation material with diverse applications in the building industry. Geopolymers are produced from the alkali activation of an aluminosilicate source, for e.g. fly ash, metakaolin etc. These binders have similar chemical composition as the natural zeolitic materials but without the extensive crystalline zeolitic structure [1, 2]. It is formed by the polymerization of individual aluminate and silicate species, which are dissolved from their original sources at high pH in the presence of alkali sources and the products exhibit high mechanical strengths having the following general chemical formula.



where M is the alkaline element, which indicates the presence of a bond, z is 1, 2 or 3 and n is degree of polymerization. Theoretically, any alkali and alkali earth cation (Ca, Mg) can be used as a replacement of the alkaline element (M) in the reaction, However the majority of research has focused on the effect of sodium (Na⁺) and potassium (K⁺) ions [3], disregarding effect of alkaline earth cations (Ca, Mg). There have been many studies investigating the role of the Si/Al ratio, and how it relates to the mechanical properties of geopolymer. Theoretically, there should be a direct correlation with mechanical strength and silica content because increasing the amount of silica increases the amount of Si-O-Si bonds, which are stronger than Si-O-Al and Al-O-Al bonds [2]. However, it was found for metakaolin geopolymers with a Si/Al ratio lower than 1.40, the composites had a very porous matrix, which led to lower compressive strength. But when the Si/Al ratio was increased over 1.65 the composites showed an increase in strength. This increase was attributed to a homogenous microstructure in the geopolymer. Also in metakaolin based geopolymers it was found that the optimum strength was at an intermediate Si/Al ratio [4]. The reduction in strength for high Si/Al ratio mixes was the result of unreacted material, which was soft and acted as a defect in the binder phase [5].

Past research has also shown that the addition of calcium into metakaolin geopolymers has beneficial results for mechanical properties. But the role that calcium plays during the geopolymer reaction period has yet to be elucidated. It has been observed that both geopolymer gel and calcium silicate hydrate form during the reaction process [6, 7]. For metakaolin geopolymers, it appears that the alkali hydroxide concentration plays a vital role in determining if C-S-H forms in the geopolymer. At low alkali hydroxide concentration, the reaction product favors the formation of C-S-H, while at higher concentration (above 10 M) the reaction favors the formation of the geopolymer gel. This difference is due to the fact that the high hydroxyl concentration hinders the Ca²⁺ dissolution forcing the dissolved silicates and aluminum species to form geopolymer gel. On the other hand, when the OH⁻ concentration is low, the amount of Ca²⁺ dissolving increases and causes more C-S-H to form. Addition of calcium has been observed to accelerate the hardening process and increase the strength for fly ash based geopolymers. It was also observed that addition of calcium increases strength for geopolymers cured at ambient conditions, while it reduces mechanical properties of geopolymer cured at elevated temperatures, because the presence of calcium hinders the development of the three-dimensional network structure in the geopolymer gel. However, other research indicates that the presence of both C-S-H and geopolymer gel in a geopolymer could have beneficial effects on strength because the C-S-H phase act like micro-aggregates for the geopolymer gel and

forms a denser and more uniform binder. More research needs to be conducted to understand the effects of composition and nanostructure on mechanical properties of both the geopolymer gel and the C-S-H phases in the geopolymer. There is a lack of documented research involving geopolymeric reaction mechanisms occurring in natural systems like ore minerals consisting of calcium and alkaline minerals and in such systems it is probable that both C-S-H gel and geopolymeric reactions could be forming simultaneously. As a result, an investigation into the role of calcium in dictating the chemical mechanism will provide answers to the fundamental question as to whether two separate phases will be formed, or a new material will be produced.

In past, industrial wastes have been utilized in manufacturing of bricks. For instance, manufacturing of bricks using waste foundry sand at industrial scale has shown promising results [8]. Similarly, IOT can be a very favorable material for manufacturing of bricks at industrial scale. The suitability of IOT as a partial replacement of sand in mortar for masonry was studied. It was found that up to 20% IOT can be replaced for sand with desired compressive strength [9]. Masonry units made of IOT in compressed earth block as a replacement for natural sand at 25, 50 and 100% rates were evaluated [10]. Optimum mix proportion of soil, sand and cement was utilized for manufacturing of stabilized mud blocks. It was found that the water absorption increased with the increase in IOT content, but was within permissible limits. It is also reported that when 7% of cement is used, the wet compressive strength is 7 MPa. The experimental results showed that the significant amount of sand can be replaced by IOT without compromising the strength parameters [10]. Lamani S R et al. [11] investigated the utility of iron ore waste (IOW) in preparing non-fired bricks by using cement and fly ash. Bricks were prepared with different proportions of cement, fly ash, and IOW. The manufactured bricks were cured for 7, 14, 21, and 28d. Compressive strength and water absorption of bricks were evaluated. The results of the study reveals that mixture with 70% IOW, 15% cement and 15% fly ash shows the minimum required compressive strength and water absorption properties of bricks for a minimum curing period of 7d. The potential of IOT in sandcrete block was evaluated [12]. Maximum IOT used was 30%, which was replaced for sand in the production of sandcrete blocks. Accordingly the study reported increase in compressive strength with increased IOT replacements from 0 to 30%, and curing period from 7 to 28d. In another study the sustainability of IOT as a replacement to fine aggregate in mortar for masonry work was studied. It was found that the strength attained was approximately 37 MPa for the optimum combination of IOT and river sand. In self-compacting concrete the replacement of fine aggregate with IOT up to 40% and red mud by cement up to 4% was evaluated [13]. Accordingly the maximum compressive strength was achieved for 30% IOT mixtures [13]. Nagaraj and Shreyasvi [14], made an explorative study to prepare compressed stabilized earth blocks utilizing various proportions of mine spoil waste (MSW), quarry dust as aggregates, cement, and lime as stabilizers. In their study the researchers used 30–50% waste along with cement and lime. Cement varied in proportions of 6 and 8% with 2% lime. Blocks of 230 × 110 × 75 mm were prepared using Mardini press. The wet compressive strength and water absorption was evaluated for various curing periods of 7, 15, 30, 60 and 1800d. They concluded that wet compressive strength, water absorption and flexure strength of the blocks are meeting the requirements of Indian standards; accordingly they suggested that these blocks can be effectively used as eco-friendly blocks in construction activity [14].

In the present work, research is conducted with the addition of GGBS and lime (commercial grade) in IOT system with fixed amount of sodium silicate.

It is envisaged, that this will lead to a number of reaction products and the type and number of these products will be dependent on the experimental conditions and, more importantly, depending on the form of calcium present. It is anticipated that likely products to be formed will be calcium silicate hydrate and aluminum silicate geopolymers. Also the isomorphs nature of iron in combination with aluminum will most probably produce alkali (Al + Fe) geopolymers. It will be interesting to find the application of this waste as regards to its mechanical strength as construction material through calculation of Si/Al, Si/(Al + Fe), Ca/Si ratios. As regards to industrial application the present research explores the possibility of utilizing IOT for the production of eco-friendly bricks. These bricks are produced in Mardini block making machine. The formed bricks are kept in room temperature for extended time periods after which different properties are determined.

2. Part A: case study with BMM Ispat iron ore mines

2.1 Experimental

2.1.1 Materials

The materials used in this study include IOT, sodium silicate solution (Na_2SiO_3), lime, GGBS and potable water. The mine tailings were received in a sizes ranging ($<150\text{ }\mu\text{m}$) from the Bellary mining area (BMM Ispat). The physical properties of the mine tailings are determined as per the standard (IS: 2720 (part 3 & 7)). Ground granulated blast furnace slag (GGBS) is a by-product of steel industry. GGBS can be used as a replacement for, or be blended with portland cement. When blended with Portland cement it is called Portland slag cement. Addition of GGBS has shown improvement in properties of the cement like resistance to chemical attack which results in improved durability of concrete mixtures. The chemical composition of IOT and GGBS is presented in **Table 1**. **Table 2** presents the physical properties of IOT. **Figure 1** shows SEM images of IOT and GGBS, respectively. As seen the IOT surface shows random distribution of irregular particles, while GGBS surface exhibited aggregates which might have happened due to surface kinetics. **Figures 2 and 3** show the XRD patterns of IOT and GGBS. XRD pattern of IOT shows the presence

Chemical composition	IOT	GGBS
SiO ₂	9.02	34.16
Fe ₂ O ₃	66.56	1.99
Al ₂ O ₃	9.56	17.54
CaO	1.96	37.10
MgO	2.12	—
MnO ₂	1.15	—
TiO ₂	0.66	1.00
K ₂ O	—	0.31
Na ₂ O	—	0.57

Table 1.
Chemical composition of IOT & GGBS.

Fineness modulus	1.8
Specific gravity	2.34
OMC (%)	9.8
MDD (g/cm ³)	2.14
Plastic limit (%)	16.56
Liquid limit (%)	26.1
Plasticity index (%)	9.54

Table 2.
Physical properties of IOT and GGBS.

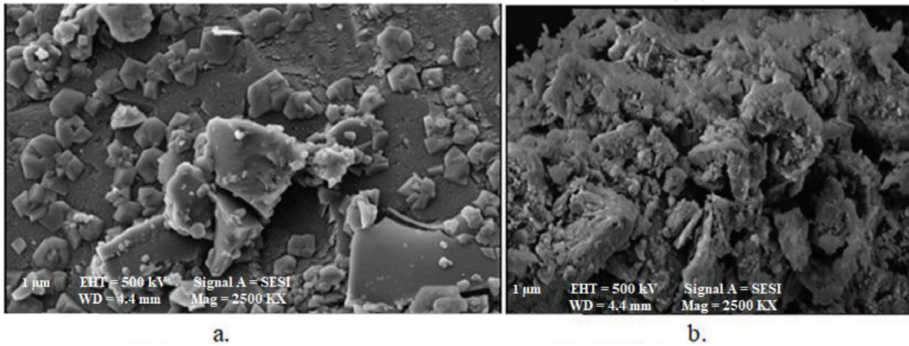


Figure 1.
SEM micrograph (a) IOT, (b) GGBS.

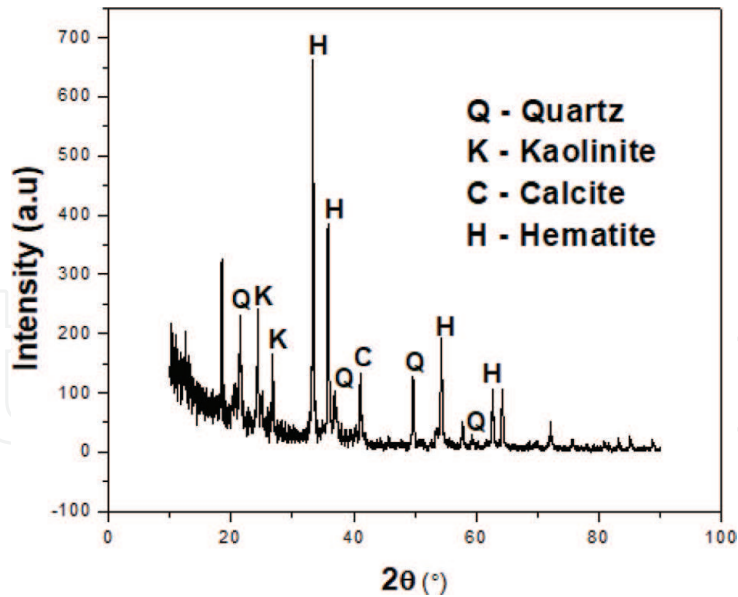


Figure 2.
XRD pattern of Iron ore tailing (IOT).

of kaolinite and calcite in addition to the crystalline phase of hematite. XRD pattern of GGBS shows highly amorphous nature, which is effective to control the geopolymeric reaction. The lime used in this study was commercial grade and slaked in nature, and the purity was more than 96%. Sodium silicate used in this study was procured from a local sodium silicate manufacturer in liquid form in concentration range of 3(SiO₂):1(Na₂O).

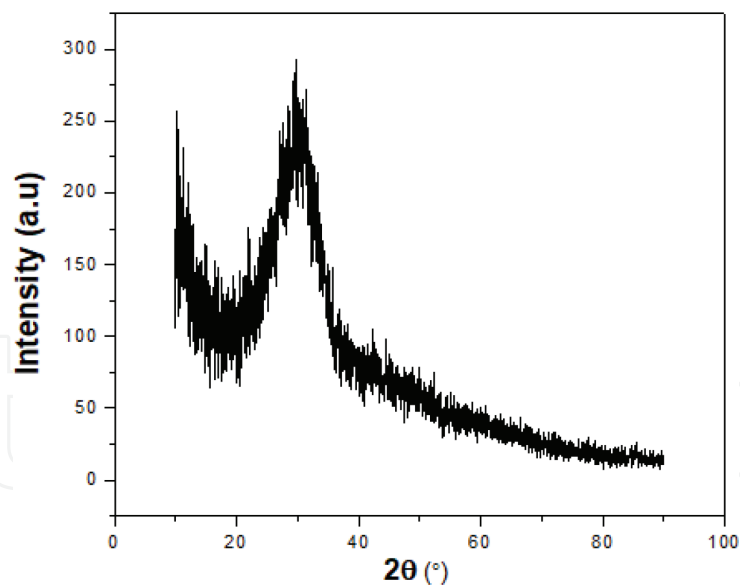


Figure 3.
XRD pattern of GGBS.

2.1.2 Preparation of brick samples

In present study standard sized brick (230 × 115 × 75 mm) were made by hand pressing in Mardini press as shown in **Figure 4**. The brick were cured at room temperature conditions for a period of 7, 14, and 28d. The variation of IOT was between 30 and 50% in increments of 5%. While the amount of lime was kept fixed at 5% and sodium silicate concentration was fixed at 20%. The addition of GGBS was proportionately decreased from 45 to 25% such that the total of IOT and GGBS for each composition was 75%. **Table 3** presents the details of mix specification. The schematic representation of brick making is shown in **Figure 5**.

2.2 Results and discussion

All the tests on the bricks were performed as per the IS:3495 standard. The maximum stress in the brick specimens were calculated by determining the maximum load at failure to the area of the bed surface of the bricks. Load was



Figure 4.
Mardini block making machine.

Mix designation	Quantity (%)			
	IOT	GGBS	Na ₂ SiO ₃	Lime
B1	30	45	20	5
B2	35	40	20	5
B3	40	35	20	5
B4	45	30	20	5
B5	50	25	20	5

Table 3.
Details of mix specifications.

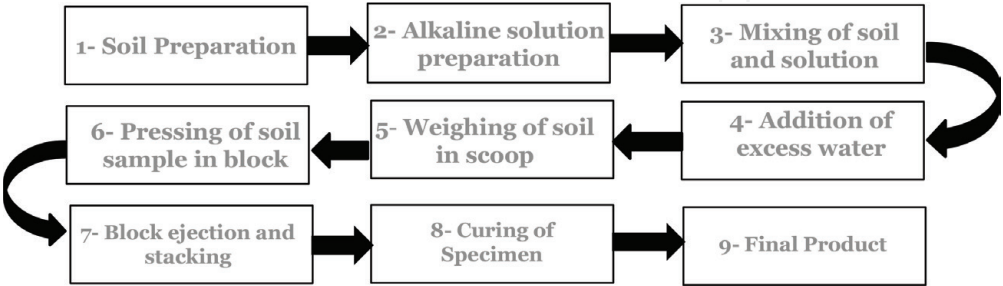


Figure 5.
Schematic representation of brick making.

applied axially at a uniform rate of 14 N/mm² per minute till failure. **Figure 6** shows the setup for compressive strength test. The results of the strength test are shown in **Figure 7**. Water absorption data is obtained by immersion of brick specimen in water for 24 h at room temperature, followed by drying of bricks in oven at 110°C. The water absorption is determined by obtaining the difference in weights of bricks before and after drying, to its dry weight in percentage. As per the IS: 3495 good quality bricks should not absorb more than 20% water by its weight. From the results of strength, density, and water absorption, it is seen that there is an increase in strength and density with increase in IOT (as observed in **Figures 7 and 8**). Similar trends in increase of water absorption and compressive strength for IOT bricks with increased IOT content were reported in past studies. Increase in water absorption as observed in **Table 4** can be attributed to the higher absorbing capacity of the tailing waste. The water absorption for IOT bricks



Figure 6.
Compressive strength test setup.

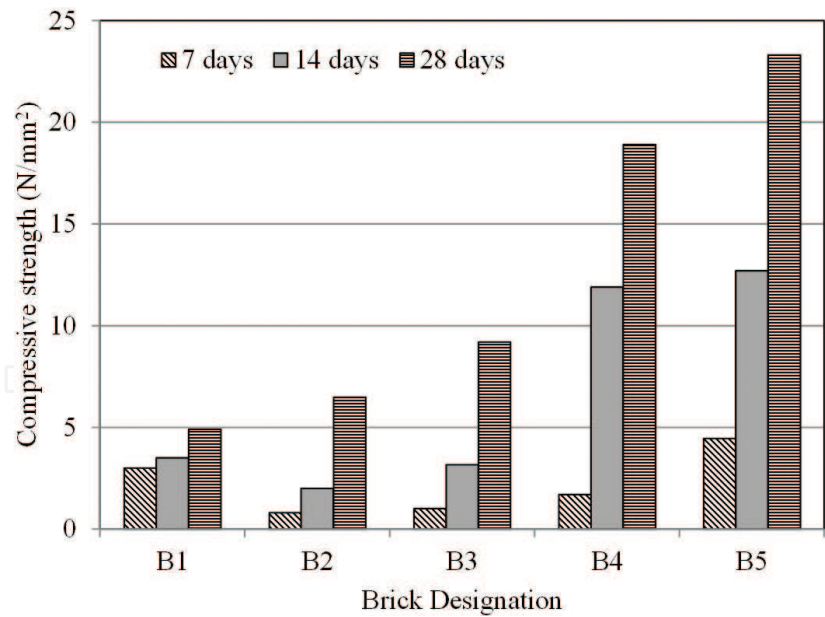


Figure 7.
Compressive strength of bricks at different curing period.

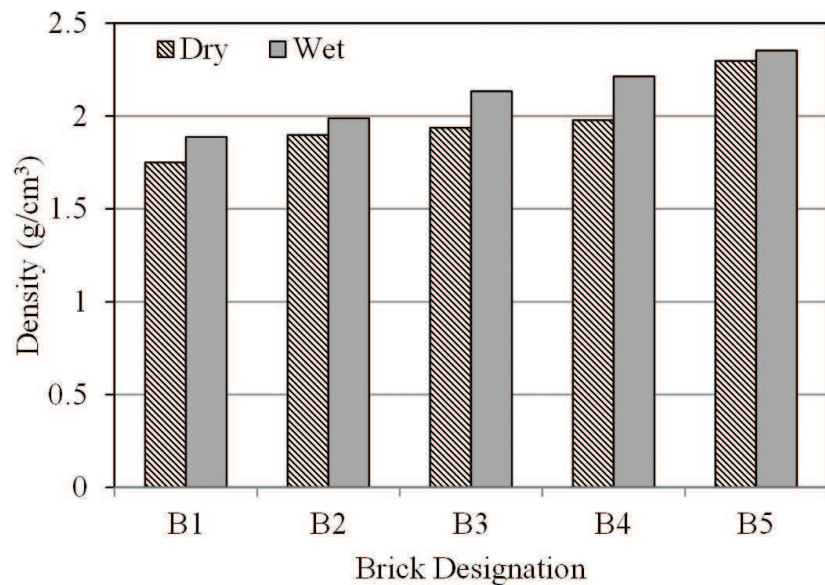


Figure 8.
Density of bricks.

Brick designation	B1	B2	B3	B4	B5
Water absorption	5.9	7.5	10.2	12.7	13.5

Table 4.
Water absorption of bricks.

are well below the limits of IS standard specifications. The relationship between Si/Al, Ca/Si, and Si/(Al + Fe) to compressive strength at different curing periods is shown in **Figures 9–11**, respectively. It is observed that there is a drastic fall in the compressive strength with increase in the Si/Al, Ca/Si, and Si/(Al + Fe) concentrations. At early curing periods (7d) the critical threshold values for Si/Al, Ca/Si, and

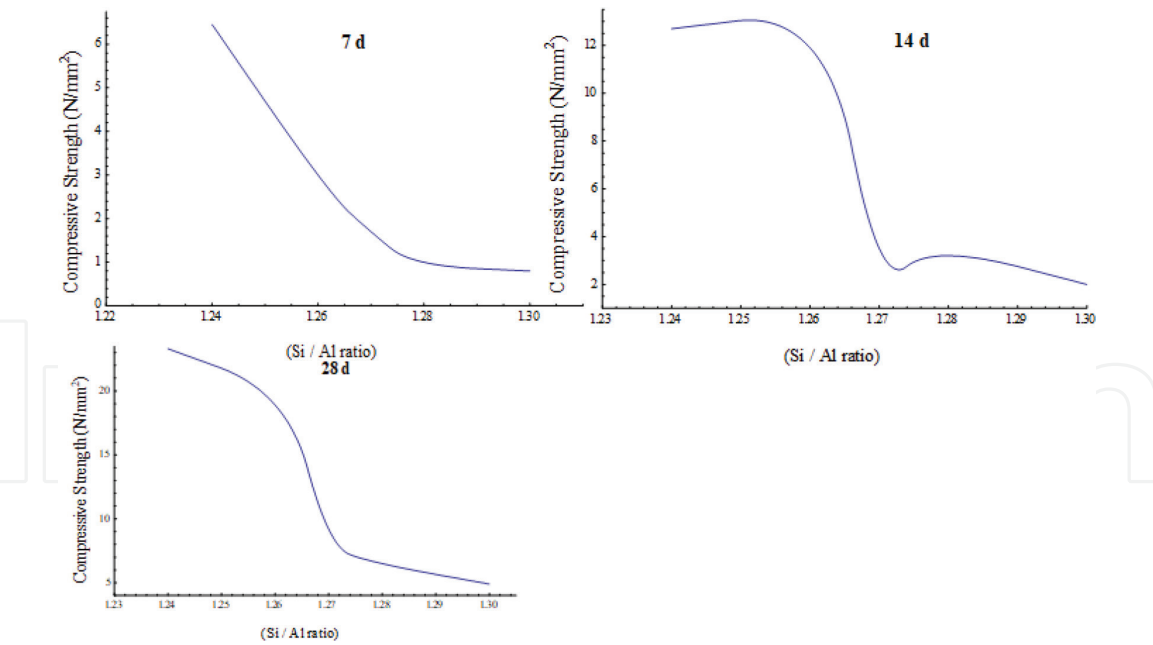


Figure 9.
Relationship between Si/Al ratio and compressive strength at different curing periods.

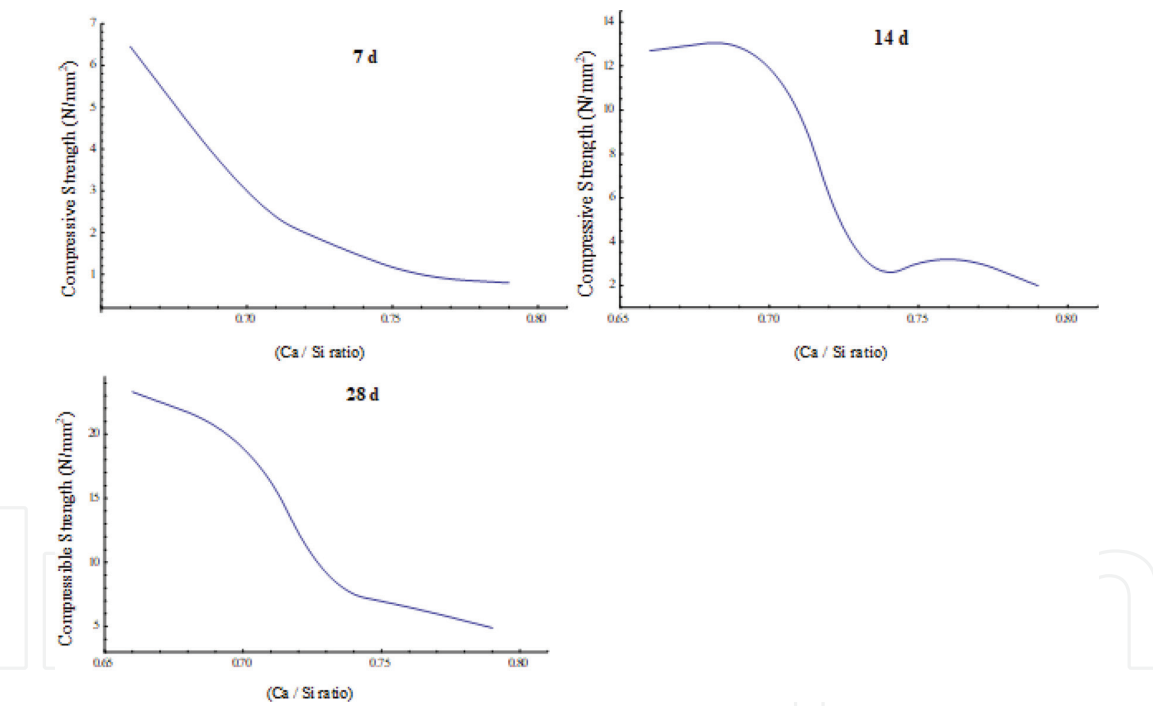


Figure 10.
Relationship between Ca/Si ratio and compressive strength at different curing periods.

Si/(Al + Fe) ratios were observed to be 1.25, 0.70, and 0.30, respectively, beyond the threshold values there was drastic reduction in the compressive strength of the bricks. Similarly, at later curing periods (14 and 28d) the critical threshold values for Si/Al, Ca/Si, and Si/(Al + Fe) ratios were observed to be 1.27, 0.73, and 0.34. **Figure 12** shows the SEM micrograph of brick specimen with 50% IOT. Sample cured at room temperature for 7d show amorphous phase with gel nature, but the increase in curing period changed the structure to crystalline with less degree of aggregation.

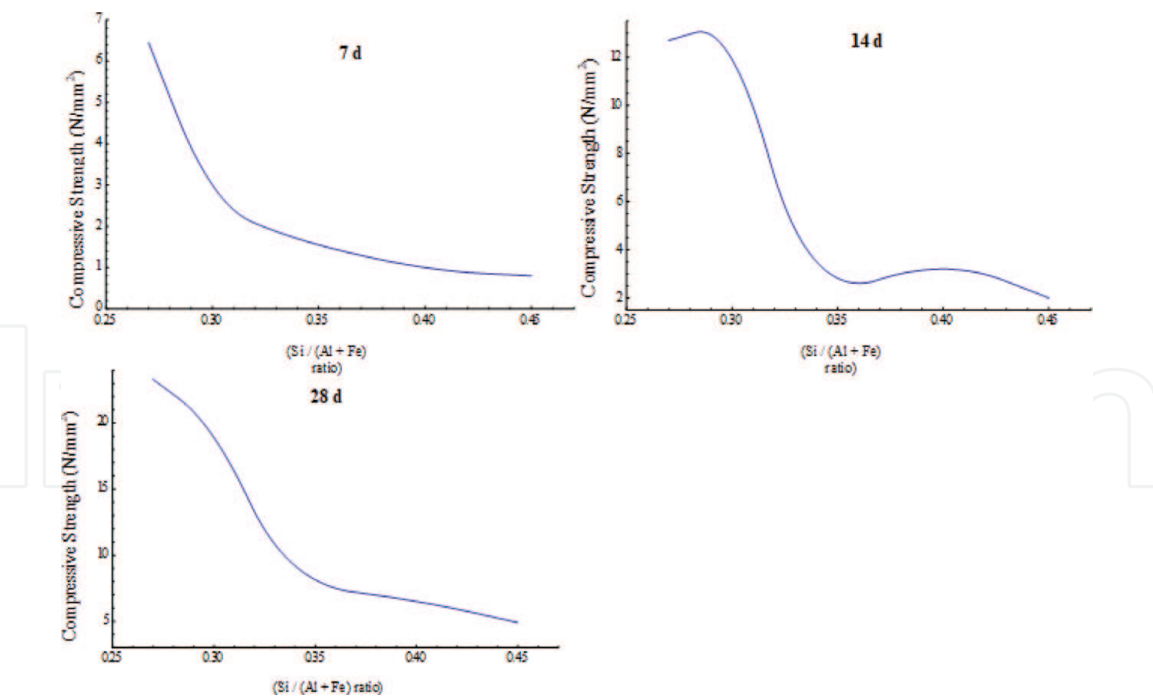


Figure 11.
Relationship between $\text{Si}/(\text{Al} + \text{Fe})$ ratio and compressive strength at different curing periods.

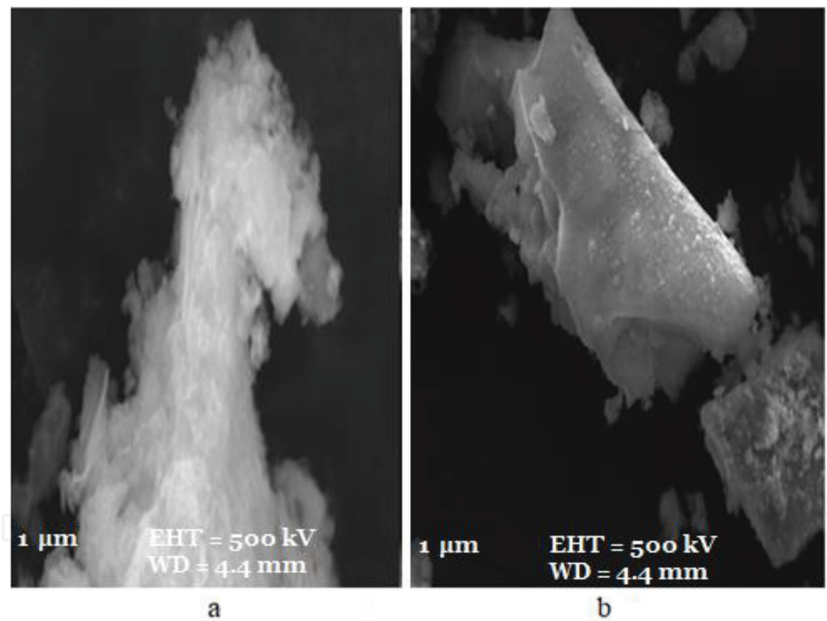


Figure 12.
SEM micrograph of brick specimen with 50% IOT (a) 7d curing, (b) 28d curing.

3. Part B: case study with JSW Steel mines Ltd

3.1 Experimental

3.1.1 Materials

Raw materials are important in geopolymer formation and materials rich in Si and Al are the primary requirement. These two materials react with alkaline solution (comprising of sodium silicate and sodium hydroxide) for dissolution of Si and Al to form reactive precursor necessary for mechanical strength. Combination

of these two materials has traditionally been used for geopolymer formation. The role of calcium either in formation of C—S—H gel giving cementitious bond or as a substitute in Al and Si as partial replacement towards geopolymerisation reaction is yet a controversy. In the current study as a calcium source; GGBS, fly ash and slag sand were used in the various batch compositions.

Fly ash is a waste by-product from coal burning power plants for generation of electricity. It has pozzolanic properties and is widely used in cement making industries as an admixture in portland cement. GGBS is a byproduct from iron making industries namely blast furnace. GGBS can be used as a replacement for, or be blended with portland cement. When blended with portland cement it is called portland slag cement. Addition of GGBS has shown improvement in properties of cement, like resistance to chemical attack. However, the quality of slag depends on certain conditions. For instance, slow cooling of the slag results in more crystallized product, while rapid cooling results in desired non-crystallized product which exhibits enhanced reactivity. Slag sand is an admixture of blast furnace slag and other calcia based alumina-silicate compounds produced by Jindal Steel Ltd., Karnataka, India. The materials are generally used in civil engineering construction for early development of strength in concrete. IOT is a low grade iron ore generated in iron ore mines after sorting out of good quality ore (>65% hematite).

Water glass, also known as sodium silicate, contains compounds of sodium oxide (Na₂O) and silica (SiO₂) and forms a glassy material that is soluble in water. Water glass can be produced as both an aqueous solution and as solid material. It is produced when burning sodium carbonate and silica sand in a furnace at temperatures between 1000 and 1400°C. The viscosity of the solution depends on the ratio of SiO₂ and Na₂O; the higher the concentration of both, the more viscous is the solution. Water glass dissolves in water and produces an alkaline solution which is glassy in nature and colorless. Due to the alkali properties, water glass will react under acidic conditions and form a hard glassy gel, which is a very useful as bonding agent. Sodium hydroxide (NaOH), also known as caustic soda is a white material commonly found in the form of pellets, granules or flakes. NaOH is highly soluble in water and because of its high alkaline activator levels; it is normally used in geopolymer reactions. The NaOH concentration has a significant effect on the compressive strength of geopolymers.

In the present study 8 and 10 M NaOH solution were used. The NaOH was delivered by Merck (Germany). **Table 5** presents the chemical composition of all the raw materials used. **Figure 13** shows the XRD patterns of different raw materials. XRD analysis was performed to understand the nature of the material. Sharp and intense peaks of samples A and D, is attributed to its crystalline nature. Broad diffraction peaks of samples B and C, exhibited its amorphous nature. The full width at half maximum (FWHM) of a peak is inversely proportional to crystallite size. Lower crystallite size of samples B and C, compared to samples A and D is obvious from the diffraction patterns.

	SiO ₂	Al ₂ O ₃	CaO	MgO	MnO ₂	TiO ₂	Fe ₂ O ₃
Fly ash	66.87	23.34	1.17	0.31	—	—	4.41
GGBS	31.79	17.07	38.78	6.23	—	—	0.49
Slag sand	30.73	16.32	38.47	6.41	—	—	0.56
IOT	16.05	6.34	1.52	0.28	1.20	0.38	44.82

Table 5.
Chemical composition of raw materials.

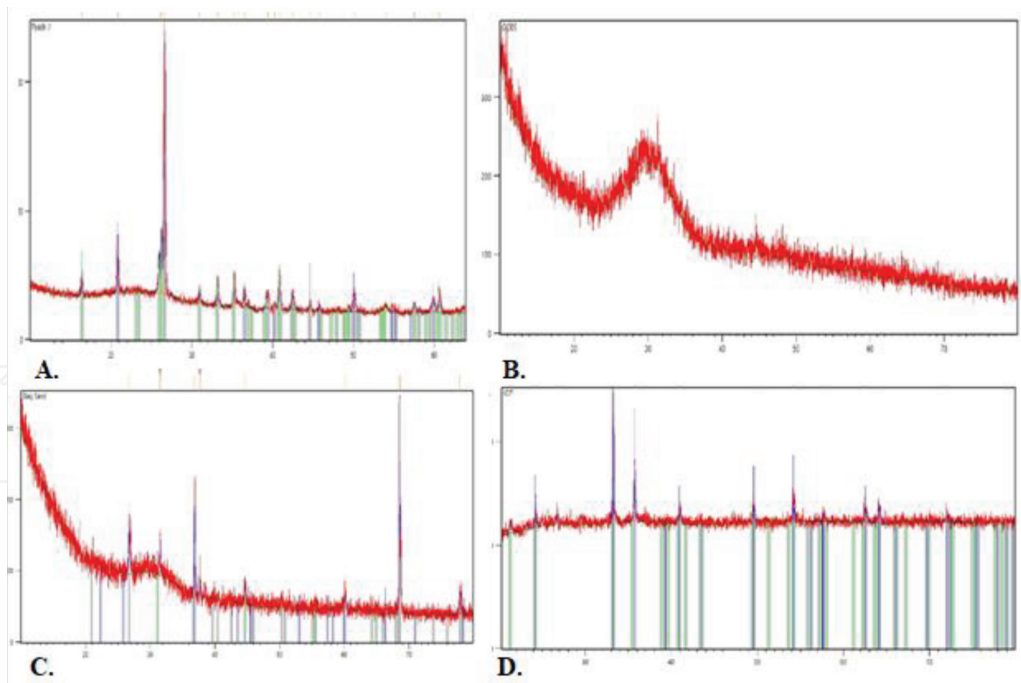


Figure 13.
XRD patterns of (A) fly ash, (B) GGBS, (C) slag sand, and (D) IOT.

3.1.2 IOT geopolymer with fly ash, GGBS and slag sand composition

Table 6 presents the details of proportions made with IOT, fly ash, GGBS, and slag sand. The amount of fly ash and GGBS was kept constant as 15% in all the compositions. As seen in **Table 6**, IOT increased from 20% (GB1) to 40% (GB3). The Ca/Si ratio was increased and varied between 0.504 (GB-1) and 0.368 (GB-3). All these compositions were made with 8 M alkaline solutions. Similarly, another set of composition with 10 M alkaline solution was made. IOT varied from 20% (GB4)

Sample designation	GB1	GB2	GB3	GB4	GB5	GB6
IOT (%)	20	30	40	20	30	40
Slag sand (%)	40	30	20	40	30	20
Fly ash (%)	15	15	15	15	15	15
GGBS (%)	15	15	15	15	15	15
Alkaline sol. (concentration)	10 (8 M)	10 (8 M)	10 (8 M)	10 (10 M)	10 (10 M)	10 (10 M)
NaOH sol. (%)	2.04	2.04	2.04	1.8	1.8	1.8
Na ₂ SiO ₃ sol. (%)	1.37	1.37	1.37	1.71	1.71	1.71
Extra water (%)	3.8	3.8	3.8	3.69	3.69	3.69
Si (mol)	0.767	0.730	0.693	0.770	0.733	0.696
Al (mol)	0.068	0.063	0.058	0.068	0.063	0.058
Si/Al (molar ratio)	11.279	11.587	11.948	11.324	11.635	12.000
Ca (mol)	0.387	0.321	0.255	0.387	0.321	0.255
Ca/Si (molar ratio)	0.504	0.439	0.368	0.502	0.438	0.366

Table 6.
Details of mix proportion with fly ash, GGBS, and slag sand.

to 40% (GB6) with 10 M alkaline solution. The ratio of Si/Al varied between 11.32 (GB-4) and 12.00 (GB-6). The Ca/Si ratio varied between 0.502 (GB-4) and 0.366 (GB-6).

3.2 Results and discussion

The compressive strength versus Ca/Si ratio at different curing periods for the different compositions is shown in **Figure 14**. The general trend of increase

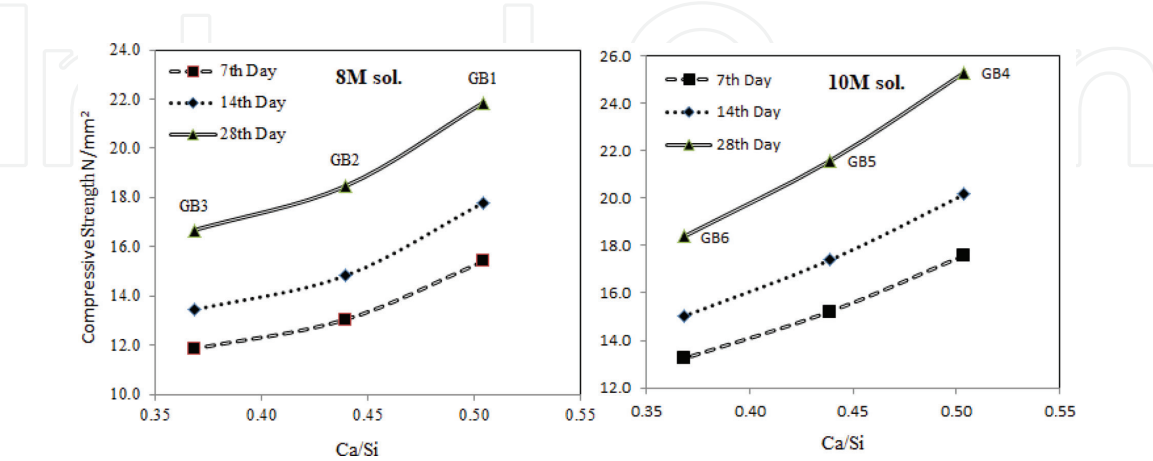


Figure 14.
Compressive strength vs. Ca/Si.

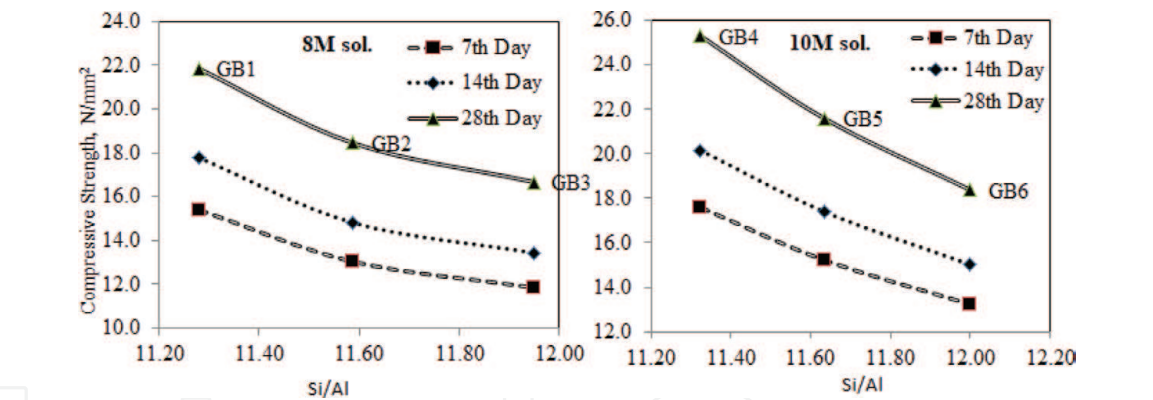


Figure 15.
Compressive strength vs. Si/Al.

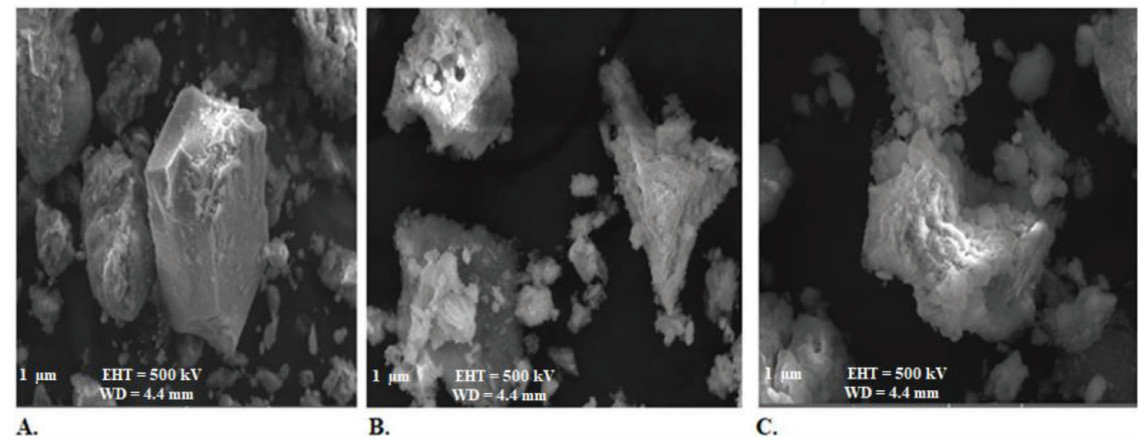


Figure 16.
SEM micrographs of composites with (A) GB1, (B) GB3, and (C) GB6.

in compressive strength with increase in time period (7, 14, and 28d) is observed. However, with increase in the amount of IOT (from 20 to 40%) there is a fall in compressive strength. It is observed that in all the different samples the Ca/Si decreases with increase in IOT content. This was further attributed to decrease in compressive strength of the brick specimens. Almost identical trends were observed for the specimens with 10 M solution. **Figure 15** shows the compressive strength behavior of the bricks and its relationship with Si/Al ratio. It will be observed that with increase in Si/Al ratio there is decrease in compressive strength.

Figure 16 shows the SEM micrographs of different composites. The SEM micrograph of GB1 reveals the crystalline nature of the material with irregular distribution of shapeless particles. Crystalline nature of the material may positively influence the compressive strength. GB3 sample also exhibited shapeless structures with irregular distribution. The aggregation due to surface kinetics is more compared to GB1. Due to high concentration of NaOH, GB6 is highly aggregated with less number of independent particles. The compressive strength of GB3 was found to be less due to low calcium content, less concentration of sodium hydroxide and irregular distribution of particles with some degree of aggregation.

4. Conclusion

In the current study, IOT from two different sources were used to make brick samples in laboratory. The characterization on IOT from different sources revealed the variation in the chemical composition of the raw hematite tailings. In the first sample the combined ($\text{SiO}_2 + \text{Al}_2\text{O}_3$) was found to be 18.6%, and that of hematite was 66.56%, similarly second sample showed ($\text{SiO}_2 + \text{Al}_2\text{O}_3$) to be 22.4% and hematite was 44.8%. With first IOT sample, bricks were manufactured with GGBS, sodium silicate, and lime. In the brick specimens with IOT, GGBS, sodium silicate, and lime there was an increase in compressive strength with increased IOT content. The trend in increased strength was similar for different curing periods. A maximum of 23 N/mm^2 was observed for bricks made with 50% IOT, 25% GGBS, 20% Na_2SiO_3 , and 5% lime. The relationship between Si/Al, Ca/Si, and Si/(Al + Fe) to compressive strength at different curing periods were developed. From the developed ratios it was observed that at early curing periods (7d) the critical threshold values for Si/Al, Ca/Si, and Si/(Al + Fe) ratios were 1.25, 0.70, and 0.30, respectively, beyond the threshold values there was drastic reduction in the compressive strength of the bricks. With second IOT sample, bricks were manufactured with GGBS, fly ash, slag sand, Na_2SiO_3 , and NaOH (8 and 10 M). With increase in IOT content there was decrease in compressive strength of the brick specimens. The maximum compressive strength of 25.7 N/mm^2 was observed for bricks containing 20% IOT, 40% slag sand, 15% fly ash, 15% GGBS, Na_2SiO_3 , and NaOH (10 M). The limited results of the current study shows the effective utilization of waste materials like IOT, GGBS, fly ash along with geopolymerisation results in more eco-friendly and environmental sustainable building material.

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