

4.1 Introduction

In the previous chapter-3, preparation and characterization of geopolymer brick samples of different bottom ashes were discussed. For making geopolymer samples, very finer size particles are required to get the optimum geopolymerization (Chindaprasirt and Rattanasak, 2010). Therefore, the restrictions imposed for using bottom ash to prepare geopolymer samples are due to its coarser in nature as compared to fly ash (bin Mohd Sani and bt Muftah, 2011; Uçurum et al., 2011). That's why, adequate grinding of bottom ash is required to improve its pozzolanic activity for making geopolymer bricks, but which is not economical (Ramadoss and Sundararajan, 2014). Bottom ash has no self-binding properties due to its coarseness and absence of clay content as mentioned in Table 2.2 & Figure 2.3 (Marto et al., 2014). Hence, a suitable binder is required for making bricks. The property of binder should be such that which imparts strength inside bricks in green as well as fired condition. There are some commercially available binders, such as bentonite, metakaolin, normal clay (with plasticity index greater than 75) and also $\text{Ca}(\text{OH})_2$ which could be used for the production of bottom ash aggregate (Geetha and Ramamurthy, 2011, 2010; Pradhan et al., 2006). Fired strength depends upon the presence of low melting constituents produced at that particular temperature inside the bricks. Silica and alumina (main constituents of bottom ash) have high melting points which do not form any low melting constituents in the absence of suitable oxides like CaO , MgO etc. From the ternary diagram $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-FeO/Fe}_2\text{O}_3$, it is evidenced that lower melting phases could be produced in the presence of $\text{FeO/Fe}_2\text{O}_3$ (Schacht, 2004).

Iron ore slime is another waste material produced in iron ore beneficiation plant. It contains around 48–62% Fe which is being discarded in tailing ponds without any further

utility. This iron ore slime is not yet to be suitable for iron and steel making due to extremely fine size and presence of high amount of gangue constituents.

For the above-said reasons, iron ore slime could be utilized as a binder in making bricks due to the presence of extremely fine particles which could give green strength. The presence of FeO content could be helpful for formation of the lower melting constituents at a lower temperature of firing.

Therefore, the objective of the present chapter deals with the utilization of iron ore slime and coarse bottom ash for making of low temperature fired bricks and their characterization for their commercial uses.

4.2 Experiments for feasibility study

Before starting the details experiments, one trial test was performed to estimate the requirement of bottom ash fineness, iron ore slime content, firing temperature, etc. for achieving required optimum property suitable for insulation bricks. For that purpose, FBC bottom ash was selected under consideration due to its wider size range of the particles.

4.2.1 Preparation of test samples

Initially, bottom ash was screened out into two different fractions, i.e. a) Coarser (0.2 – 1 mm) and b) Finer (0.075-0 mm). Coarser and finer fractions were thoroughly mixed in different proportion (i.e. 25/50/75) with 0, 5, and 10% iron ore slime using optimum moisture content (OMC) as shown in Table 4.1. OMC was determined as per method mentioned in the previously reported literature (Rai et al., 2013).

Table 4. 1 Different mix proportion and operating parameters for making brick samples

Brick samples Code	Bottom Ash (%)		Iron ore slime (%)	Firing Temperature (°C)	Moulding pressure (MPa)	OMC (%)
	Coarse	Fine				
75C25F0IS	75	25	0	1000, 1100, 1200	15	30.0
75C25F5IS	75	25	5	1000, 1100, 1200	15	29.5
75C25F10IS	75	25	10	1000, 1100, 1200	15	29.0
50C50F0IS	50	50	0	1000, 1100, 1200	15	29.5
50C50F5IS	50	50	5	1000, 1100, 1200	15	29.0
50C50F10IS	50	50	10	1000, 1100, 1200	15	28.5
25C75F0IS	25	75	0	1000, 1100, 1200	15	29.0
25C75F5IS	25	75	5	1000, 1100, 1200	15	28.5
25C75F10IS	25	75	10	1000, 1100, 1200	15	28.0

Samples of 30 mm cubic shape brick samples were prepared by applying 15 MPa pressure for 10 seconds duration on hydraulic press (Capacity 50 ton). For the thermal conductivity measurement, 30 mm cubic sample was prepared. Dried samples were fired for two hours in an electric muffle furnace at 1000, 1100 and 1200°C respectively. The firing was performed at a very slow rate of heating and cooling (2°C/ minute) to avoid crack generation inside the samples due to the removal of moisture as well as phase transformation. The flow chart of sample preparation, as well as their characterization is shown in Figure 4.1. Photographs of samples after firing at a different temperature are shown in Figure 4.2.

4.2.2 Testing methodology of test samples

Required tests for accessing the quality of fired samples were performed. Compressive strength was measured using the low range universal testing machine (UTM) at a constant loading rate of 0.1 mm/min. The apparent porosity was determined using the 'Boiling Water method' as per ASTM C-20 (ASTM C20, 2010). The bulk volume was measured by measuring the dimensions by a slide caliper.

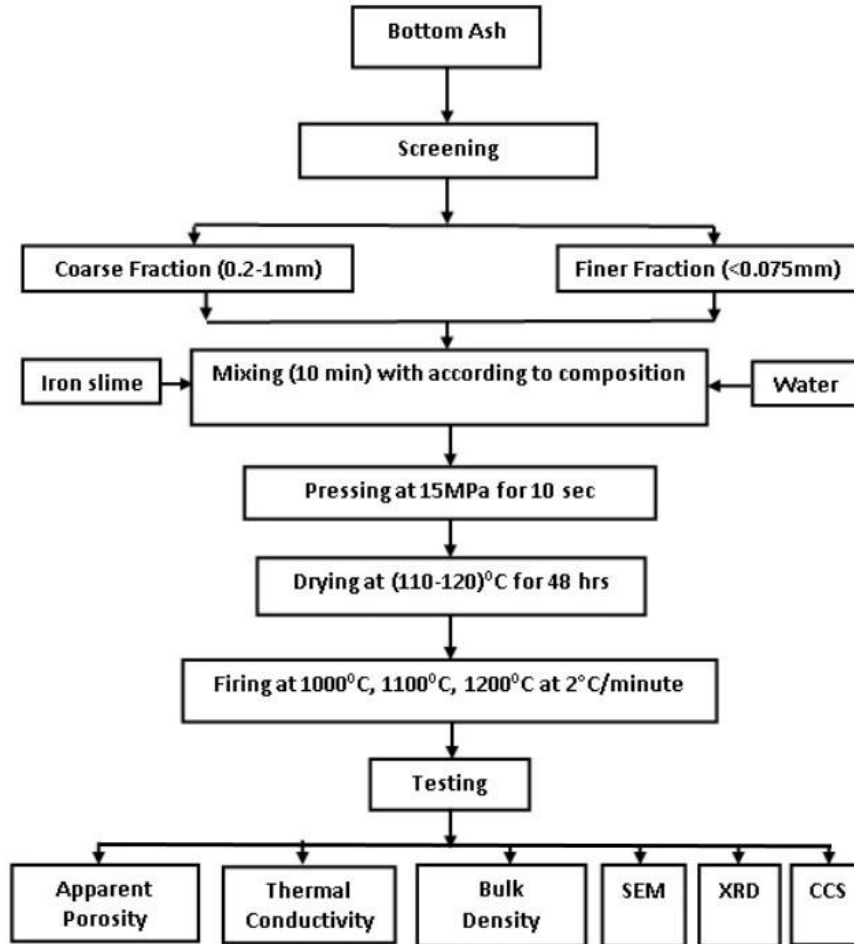


Figure 4. 1 Flow chart for preparation and characterization of brick samples

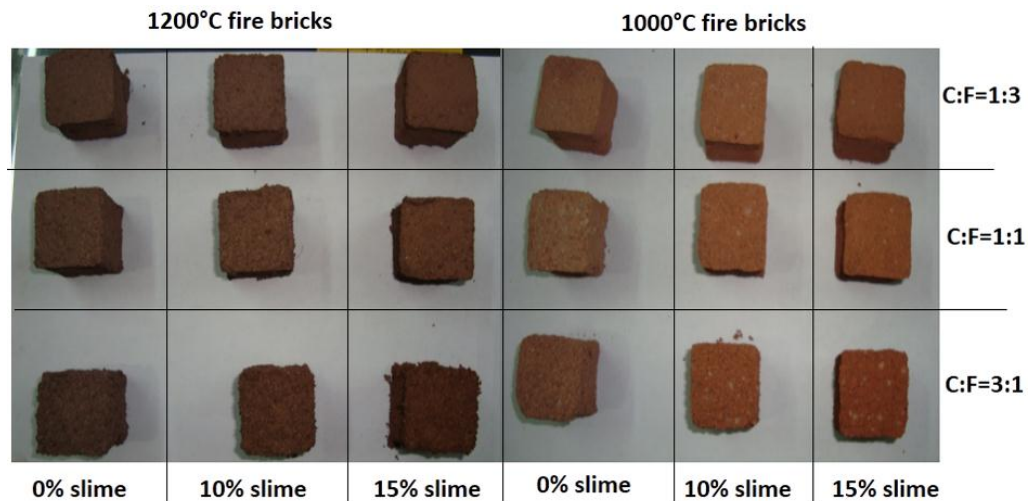


Figure 4. 2 Photograph of brick samples after firing at different temperature

Bulk density was measured by dividing the dry weight by bulk volume. The SEM imaging was performed on freshly broken test samples after compressive strength testing. XRD analysis was done on powdered sample by using CuK α as a primary emission radiation at the range of 20-80°. The thermal conductivity of the samples was measured at 600°C as per composite wall method. An apparatus was fabricated for performing thermal conductivity measurement experiment as mentioned in ASTM standard (ASTM C1363, 2011). Samples of known thermal conductivity (k_1) were procured and cut into the shape of 30 mm cube similar to brick samples to be tested. The samples were arranged in composite form and placed one face of it in front of hot side. Three thermocouples were fixed to measure the temperature of three different zone of composite structure as shown in Figure 4.3.

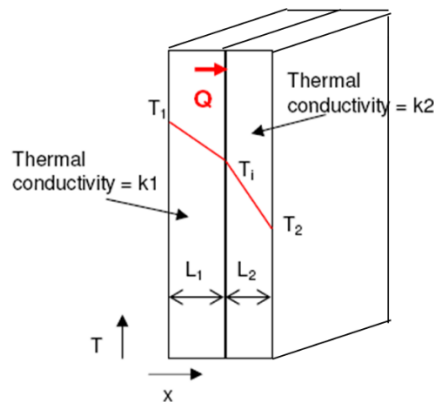


Figure 4. 3 Arrangement of samples for thermal conductivity measurement

The thermal conductivity was measured by the following formula using Fourier's law.

$$k_2 = k_1 \times \frac{L_1(T_1 - T_i)}{L_2(T_i - T_2)}$$

Where k_1 , L_1 , = thermal conductivity and thickness of the sample 1. k_2 and L_2 are the thermal conductivity and thickness of the sample 2. T_1 is the hot face temperature (furnace temperature), T_2 is the cold face temperature and T_i is the intermediate temperature of the

sample 1 and 2. All the values of k_1 , L_1 , L_2 , T_1 , T_2 , T_i are known and therefore thermal conductivity of unknown sample (k_2) was determined with the help of the above equation.

4.2.3 Results

Desired properties for assuring the engineering quality in constructional materials are described in following sections.

4.2.3.1 Cold crushing strength

The cold crushing strength (CCS) is the most important property for constructional materials. This test indicates the possible area of application for the products. This property depends on many factors, such as composition, fineness of the material, firing temperature, etc. Figure 4.4 demonstrated the CCS of samples, fired at different temperatures (1000, 1100 and 1200°C) with varying iron ore slime content (0, 5, 10 wt. %) and different fineness of bottom ash.

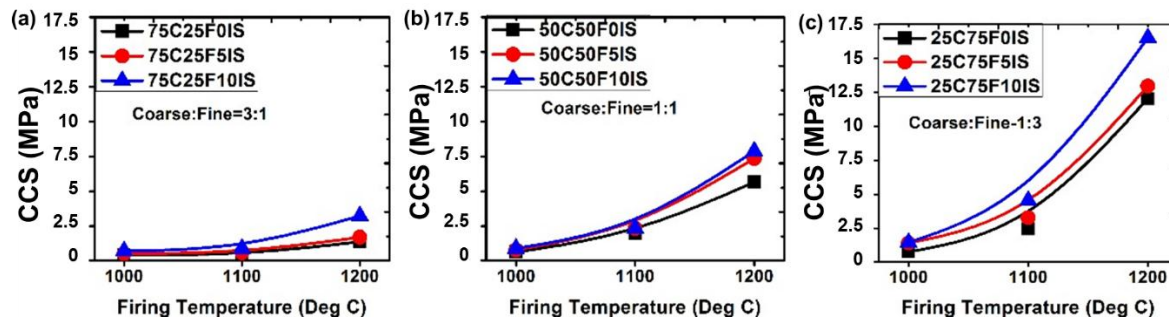


Figure 4. 4 Variation in compressive strength with different firing temperature, fineness of bottom ash and iron ore slime content

(a) Effect of firing temperature

The cold crushing strength (CCS) was increased from 0.40 to 1.36 MPa (~ 3.5 times) when the firing temperature was increased from 1000°C to 1200°C for coarser bottom ash samples without iron ore slime (i.e. 75C25F0IS). In the case of finer bottom ash samples without iron ore slime (i.e. 25C75F0IS), CCS was significantly increased from 0.76-12.05 MPa (~15 times)

for the same temperature range. In the case of 10 % iron ore slime added samples, CCS values were increased from 0.68 to 3.22 MPa (~5 times) in the case of coarser bottom ash (i.e. 75C25F10IS) whereas 1.44 to 16.53 MPa (~11 times) in the case of finer bottom ash (i.e. 25C75F10IS). Therefore, the temperature had a vital role in imparting the strength inside the samples. The fineness of bottom ash and incorporation of iron ore slime, both have influenced to increase the strength drastically.

(b) Effect of iron ore slime

The cold crushing strength (CCS) was increased by increasing iron ore slime content in the sample, even firing at a lower temperature (i.e. 1000°C). The CCS was increased from 0.40 to 0.68 MPa for coarser bottom ash samples (i.e. 75C25F0IS, 75C25F10IS) and 0.76 to 1.44 MPa for finer bottom ash samples (i.e. 25C75F0IS, 25C75F10IS), when iron ore slime was increased from 0 to 10 %. An interesting feature was observed when samples fired at 1200°C. At that temperature, the CCS was increased from 1.36 to 3.22 MPa (~2.5 times) in the case of coarse bottom ash samples (i.e. 75C25F0IS, 75C25F10IS) whereas it was increased marginally from 12.05 to 16.53 MPa (~1.5 times) in the case of finer bottom ash samples (i.e. 25C75F0IS, 25C75F10IS). It was found that the addition of iron ore slime is an encouraging effort for strength development in the coarser bottom ash samples.

4.2.3.2 Apparent porosity

Porosity is also an important property for constructional brick samples, mainly when producing lightweight aggregate for using in thermal insulation purposes. It depends on many factors, such as firing temperature, fineness, and composition of different raw materials. The effect of firing temperature, iron ore slime content, and its fineness, on the values of apparent porosity of fired samples were shown in Figure 4.5.

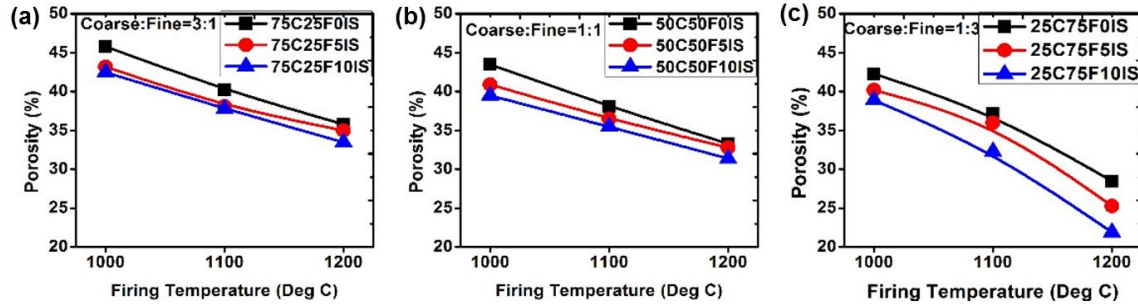


Figure 4.5 Variation in apparent porosity with different firing temperature, fineness of bottom ash and iron ore slime content

(a) Effect of firing temperature

It was observed that the apparent porosity was decreased marginally from 45.8% to 35.8% with increasing firing temperature from 1000°C to 1200 °C in case of coarse bottom ash samples (i.e. 75C25F0IS), but drastically decreased from 42.3 to 28.5 % in case of finer bottom ash samples (i.e. 25C75F0IS) without iron ore slime content. On the other hand, in the case of iron ore slime added samples, apparent porosity was decreased from 42.5 to 32.5 % for coarse bottom ash samples (i.e. 75C25F10IS) and 38.9 to 21.9% for finer bottom ash samples (i.e. 25C75F10IS) at the same temperature increment. Hence, firing temperature showed a noticeable effect for decreasing apparent porosity inside the samples. Apparent porosity difference became wider by increasing the fineness of the bottom ash as well as iron ore slime content.

(b) Effect of iron ore slime

With increasing iron ore slime content from 0 to 10%, the apparent porosity was decreased in all types of sample. The effect was more pronounced in the case of finer bottom ash samples, as compared to the coarser bottom ash. With increasing iron ore slime content from 0 to 10%, fired at 1000°C, the apparent porosity of the sample was decreased about 3% only (from 45.8 to 42.5%) in the case of coarser bottom ash (i.e. 75C25F0IS, 75C25F10IS). At 1200°C firing

temperature when iron ore slime was increased from 0 to 10 % in coarse bottom ash sample (i.e. 75C25F0IS, 75C25F10IS) apparent porosity was decreased ~ 2 % (from 35.8% to 33.5%). It was noticed from the above results that the decrease in apparent porosity with increasing iron ore slime content as well as firing temperature did not affect much in the case of coarse bottom ash samples. Similarly, with increasing iron ore slime content from 0 to 10%, fired at 1000°C temperature, apparent porosity was decreased ~3% (from 42.3% to 38.9%) for fine bottom ash samples (i.e. 25C75F0IS, 25C75F10IS). At firing temperature 1200°C, when iron ore slime was increased from 0 to 10% in fine bottom ash samples (25C75F0IS, 25C75F10IS) apparent porosity was decreased significantly ~ 6% (from 28.5 % to 21.9%). Hence, iron ore slime did not affect much on apparent porosity for coarser bottom ash samples, fired at any temperature; whereas affected greatly in case of finer bottom ash samples at higher firing temperature.

4.2.3.3 Bulk density

Bulk density is an important property for making the constructional bricks. It depends on porosity, presence of heavy materials and firing temperature. The values of bulk density of fired samples was shown in Figure 4.6 which illustrated the increasing trends with decreasing porosity and increasing iron ore slime content as well as firing temperature. The lowest bulk density was observed (1610 kg/m^3) in the case of coarse bottom ash samples having 0% iron ore slime and fired at 1000°C.

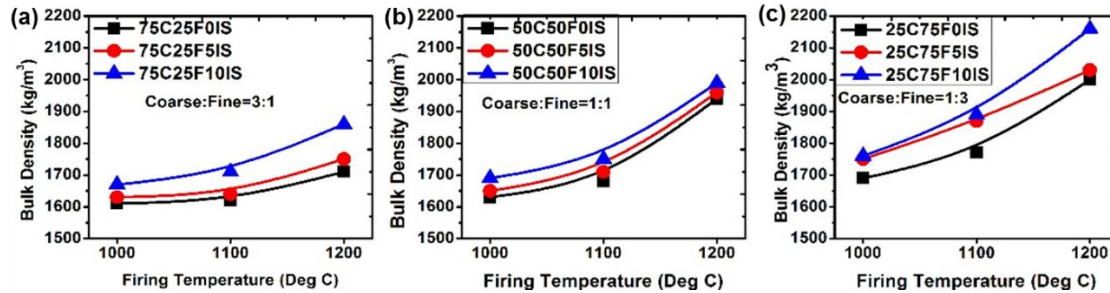


Figure 4. 6 Variation in bulk density with different firing temperature, fineness of bottom ash and iron ore slime content

(a) Effects of firing temperature

In case of coarse bottom ash samples with increasing firing temperature 1000 to 1200°C, the bulk density was increased by 6% (from 1610 to 1710 kg/m³) for 0% iron ore slime added samples (i.e. 75C25F0IS), whereas increased by 11% (1670 to 1860 kg/m³) for 10% iron ore slime added samples (i.e.75C25F10IS). However, in the case of fine bottom ash samples, it was increased to 13 % (1690 to 2000 kg/m³) for 0% iron ore slime added samples and 23% (1760 to 2160 kg/m³) for 10 % iron ore slime added samples. Like CCS, firing temperature also had a vital role for increasing the bulk density of the samples. The similar trend was observed while increasing the fineness of bottom ash and iron ore slime content.

(b) Effects of iron ore slime

Iron is the major constituent present in the iron ore slime, and that’s why a small addition of iron ore slime increased the bulk density of samples to a great extent. At 1000°C firing temperature, bulk density was increased by 4% in both cases (from 1610 to 1670 kg/m³) for coarser bottom ash (i.e. 75C25F0IS, 75C25F10IS) as well as (from 1690 to 1760 kg/m³) for finer bottom ash samples (i.e. 25C75F0IS, 25C75F10IS) with increasing addition of iron ore slime from 0-10%. At firing temperature of 1200°C, the bulk density was increased by 8- 9% (from 1710 to 1860 kg/m³) in case of coarse bottom ash brick samples (i.e.75C25F0IS,

75C25F10IS) as well as (from 2000 to 2160 kg/m³) for finer bottom ash samples (i.e. 25C75F0IS, 25C75F10IS) with increasing addition of iron ore slime from 0-10%. It was found that the addition of iron ore slime affects adversely on increasing the unit weight of samples which may not be much suitable for construction applications.

4.2.3.4 Thermal conductivity

Thermal conductivity was measured at 600°C for the samples of different firing temperature, fineness of bottom ash and iron ore slime content and their trends are shown in Figure 4.7.

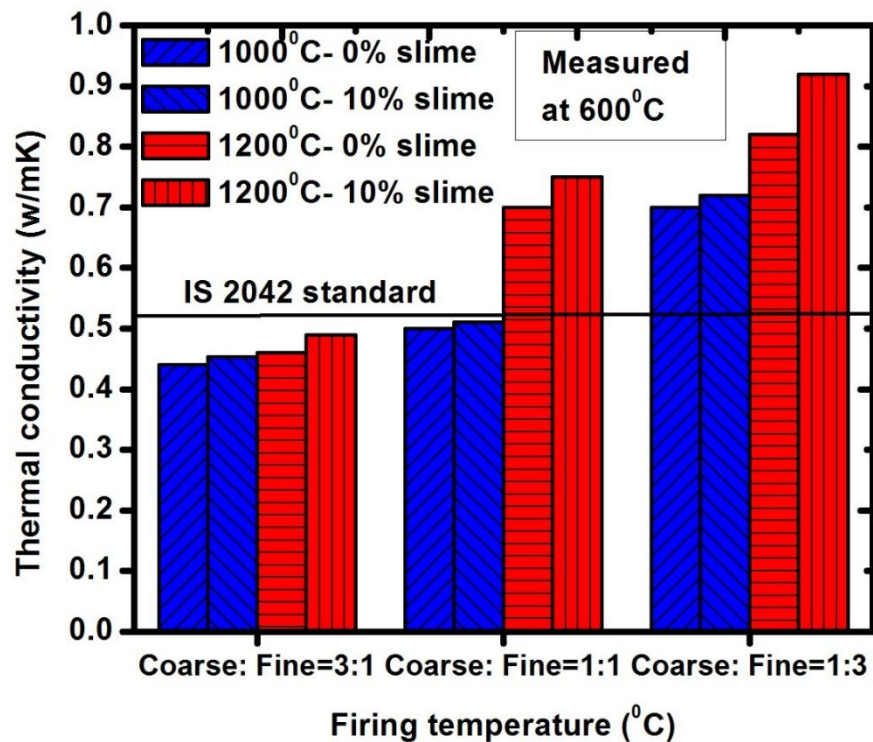


Figure 4.7 Variation in thermal conductivity with different firing temperature, fineness of bottom ash and iron ore slime content

It was observed from the above Figure 4.7 that the porosity in samples was decreased with increasing fineness of the bottom ash, resulting increased thermal conductivity in samples. The thermal conductivity was increased with iron ore slime added samples. High firing temperature

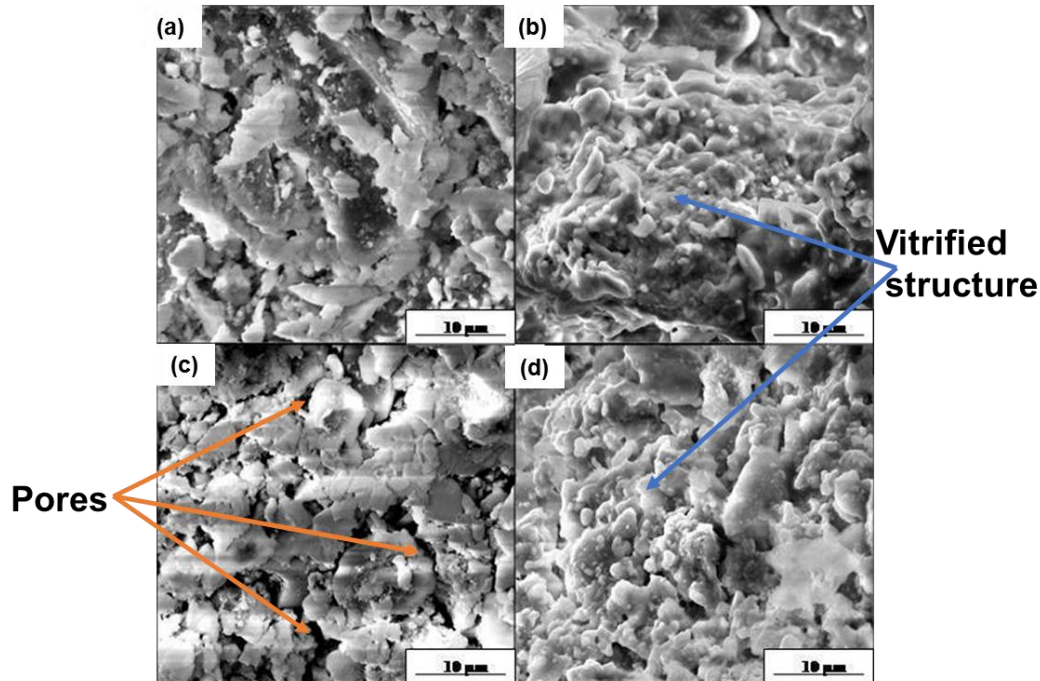
(1200°C) was more pronounced for increasing thermal conductivity of the samples. From the results of thermal conductivity, it was noticed that samples manufactured by bottom ash having ratio of coarse: fine 3:1, 10% iron ore slime and fired upto 1200°C lies in the category of A-type insulation bricks as per IS2042 (i.e. 0.52 w/mK) (IS 2042, 2006). The samples prepared by bottom ash having ratio of coarse: fine 1:1, 10% slime and fired at 1000°C also lies in the category of A-type insulation bricks. But remaining all types of the sample did not fall under the category of insulation bricks. Similarly, the addition of iron ore slime upto 10% and increasing fineness of the bottom ash, fired at any temperature (i.e. 1000°C, 1200°C), increased the thermal conductivity of the samples significantly which crossed the limit of standard insulation bricks.

4.2.3.5 SEM analysis

Two samples were chosen to have iron ore slime 0 and 10 wt.% of same fineness, fired at 1200°C for observing the effect of iron ore slime addition in the microstructural changes (i.e. 25C75F0IS and 25C75F10IS). Similarly two more samples of the same composition and fineness (i.e. 50C50F10IS), fired at 1000°C as well as 1200°C respectively were taken to see the internal structural changes occurred after firing. The SEM micrograph of above mentioned four samples are shown in Figure 4.8.

Samples without iron ore slime, fired at 1200°C, showed less dense and uncombined structure with compared to 10% iron ore slime, as shown in Figure 4.8a,b. With increasing iron ore slime content in the samples showed denser and glossy structure (Figure 4.8b). With 10% iron ore slime added samples, fired at 1000°C showed the loose un-combined matrix of material with rough surface characteristics having larger size voids (Figure 4.8c). With

increasing firing temperature 1200°C samples showed denser as well as glossier phases with reduced number of voids (Figure 4.8d).



(a-25C75F0IS fired at 1200°C; b-25C75F10IS fired at 1200°C
c-50C50F10IS fired at 1000°C; d-50C50F10IS fired at 1200°C)

Figure 4.8 SEM micrograph of different samples

Variation in microstructure due to the fineness of the bottom ash was observed in figure 4.8b and 4.8d respectively. Due to higher firing temperature (1200°C) and iron ore slime content (10%), the dense and glossy structure was observed in both samples (i.e. 25C75F10IS and 50C50F10IS). But increasing coarseness of the bottom ash samples (i.e. 50C50F10IS), more porosity was observed as compared to sample prepared by fine bottom ash samples (i.e. 25C75F10IS) as shown in Figure 4.8b,d.

4.2.3.6 Phase analysis

This is an important method for analyzing the presence of internal features (i.e. composition/phases) in the samples which are responsible for changing the properties in the

product. The X-ray diffraction patterns of fired samples having different iron ore slime content and firing temperature are shown in Figure 4.9.

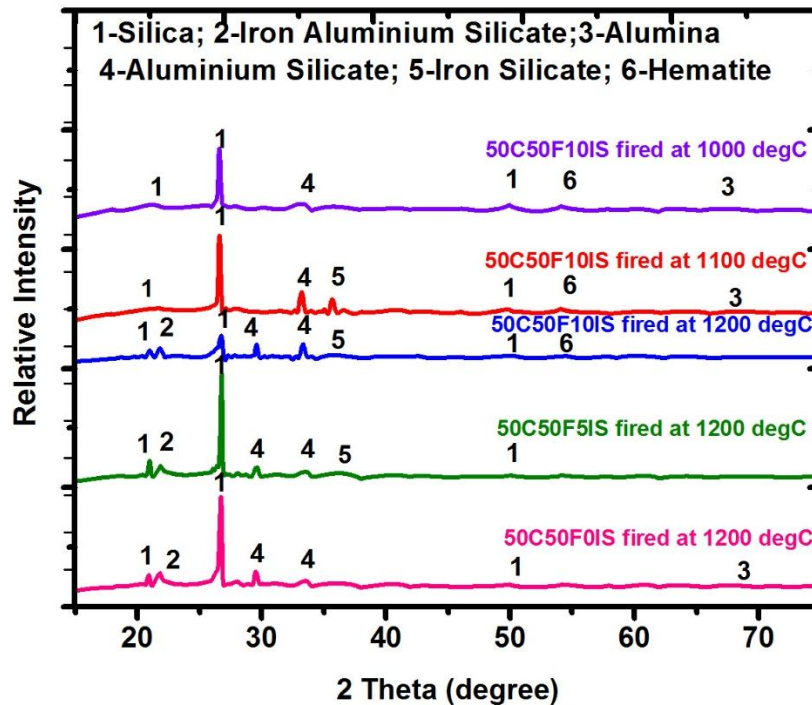


Figure 4. 9 XRD analysis of fired samples at different temperature and iron ore slime content

As expected, silica as an intense peak due to its major constituent was revealed by XRD. It exists as uncombined free form or combined as lower melting constituents. Lower melting constituents were produced mainly by reacting with alumina and iron oxide as aluminium silicate, iron silicate and iron aluminium silicate depending upon the composition and firing temperature.

The formation of aluminium silicate was increased while increasing firing temperature from 1000°C to 1100°C, which was evidenced by large instanced peak as shown in XRD pattern. The height of the hematite peak was reduced by the formation of new iron silicate

peak. Fe_2O_3 was transformed to wustite (FeO) phases and entered into silicate lattice to form iron silicate phase (i.e. fayalite).

At 1200°C , free silica phase further decreased by forming more amount of aluminium silicate phases. Prominent iron silicate phases became shorter and a new phase appeared as iron aluminium silicate. Aluminium silicate could be changed its state as kyanite or andalusite or sillimanite depending upon the firing temperature.

The addition of iron ore slime did not exhibit any new phase transformation but produced a small change in the proportion of the iron silicate formation which was observed in higher iron ore slime content. Rest of the phase did not change inside the samples. But an increasing amount of iron ore slime enhanced the relative amounts of lower melting phases.

4.2.4 Discussions

It could be seen from the Figure 4.4 that the firing temperature, iron ore slime content, and fineness of bottom ash had a significant influence on the cold crushing strength (CCS) in the samples.

The CCS was increased with increasing firing temperature, iron ore slime content and decreased with increasing coarseness of the bottom ash. With increasing firing temperature in the presence of iron ore slime, lower melting constituents were formed in the samples as evidenced by XRD analysis (Figure 4.9). It is because of increasing amount of iron ore slime; more molten slags are produced by the formation of eutectic mixture of SiO_2 , Al_2O_3 , FeO etc. This molten slag compressed the internal space of fired samples, resulting in more dense glossy structure (Li et al., 2012; Xu et al., 2009; Zou et al., 2009). The formation of glossy phase structure indicated more efficient densification at higher firing temperature as evidenced by

SEM photographs (Figure 4.8). This tendency was usually observed in ceramic materials as the temperature increases, probably due to partial vitrification which started at $\sim 1050^{\circ}\text{C}$. (Eliche-Quesada et al., 2012; Kingery et al., 1976). The changes in porosities with firing temperature were noticed with the addition of iron ore slime content and fineness of the bottom ash. The amount of liquid phase (lower melting constituents) was formed during firing of the samples which filled up the open pores resulting decreasing porosity (Romero et al., 2008). Interparticle spaces are very less in the case of finer bottom ash samples compared to coarser bottom ash, resulting in easy entrapment of pores by lower melting constituents produced in the presence of iron ore slime during firing. For this reason, a drastic decrease in porosity was observed with increasing firing temperature. The coarser bottom ash samples are having a larger size of porosity, which could not be sealed fully by lower melting constituents after firing. That's why the presence of open porosity did not affect more in the case of coarse bottom ash samples.

In addition of 10 % iron ore slime at 1200°C fired sample produced a denser structure as discussed above due to decreasing porosity, resulted increasing the bulk density of samples. The bulk density of the fired samples was also increased with increasing amounts of iron ore slime due to the presence of iron oxides as well as the fineness of bottom ash. The Bulk densities of all types of sample were found higher than 1500 kg/m^3 (i.e. bulk density of conventional clay bricks) due to the presence of iron oxide content of the samples.

It was observed that at lower firing temperature (1000°C), bottom ash particles of samples did not stick each other hence there were still a lot of original lattice interspaces. There was not enough formation of lower melting phases at that temperature, resulting in more

loosely uncombined and the less dense structure was observed (Figure 4.8c). Hence, the CCS and bulk density were decreased, but porosity was increased simultaneously.

Due to the densification and formation of more glassy structure with increasing temperature, increased thermal conductivity was observed in the samples. The increasing fineness of bottom ash and iron ore slime content decreases the porosity but increases the thermal conductivity.

In raw materials (i.e. iron ore slime and bottom ash) silica, alumina, hematite have existed as a free phase or as a combined form. During firing at low temperature, the iron oxide of iron ore slime first reacted with silica to form iron silicate (fayalite) phases. Therefore, the peak height of hematite was decreased with increasing iron silicate content. At high firing temperature aluminium silicate phase may be formed by reacting silica with alumina. With increasing firing temperature and iron ore slime content, iron aluminium silicate phase may be formed by reacting with aluminium silicate and iron oxide.

4.2.5 Optimized method for preparing brick test samples

The following conclusions could be drawn based on the results obtained from the trial study:

For the preparation of test samples, iron ore slime was used which acts as an effective binder due to its fineness resulting increasing green strength of the samples made from even coarser bottom ash.

The presence of iron oxide in iron ore slime, forms iron silicate, iron aluminium silicate phases in the presence of silica, alumina at high temperature which imparted the fired strength drastically in the samples.

Inter-particle space due to coarser size of bottom ash was increased which increase the porosity in the samples. It was simultaneously decreased the bulk density of the samples. Therefore, the insulation property in the sample was enhanced due to the coarseness of the bottom ash. The samples were made by using coarser bottom ash and lower temperature of firing which may be met the requirement of A-type insulation bricks (0.52 w/mK) as per Indian Standard (as shown in Table B.5 of Appendix-B) (IS 2042, 2006).

From the above trial studies:

It was justified that the coarser bottom ash may generate enough porosity in the sample which would provide thermal insulation property in the samples.

Densification of the materials at high temperature was occurred, therefore lower temperature is preferred for getting higher porosity.

Strength of the bricks was increased by increasing firing temperature as well as iron ore slime content. Therefore, lower temperature and higher iron ore slime content could give the desired strength as well as porosity in the samples.

Based on the output of the trial tests, following final experiments were planned to perform towards making insulation bricks.

4.3 Experiments for Insulation brick sample making

4.3.1 Materials used and their characterization

In trial experiments, only FBC bottom ash and iron ore slime, were taken but in the present experiments, FBC along with PCC bottom ash and iron ore slime were selected. The detailed

characterization procedure and their results of above raw materials are mentioned in the previous chapter-2, in section 2.2.2.1 and 2.2.3.1 respectively.

4.3.2 Preparation of brick samples

FBC and PCC bottom ashes were screened out by 1 mm size sieve for making brick samples.

Iron ore slime was mixed in different proportion with bottom ash as shown in Table 4.2.

Table 4. 2 Different mix used for making brick samples

Brick samples Code	Bottom Ash (%)		Iron ore slime (%)	OMC (%)
	FBC	PCC		
F10	90	0	10	17
F20	80	0	20	16
F30	70	0	30	15
F40	60	0	40	14
F50	50	0	50	13
P10	0	90	10	20
P20	0	80	20	19
P30	0	70	30	18
P40	0	60	40	17
P50	0	50	50	16

A homogeneous mixture of above said dry mixtures with the addition of optimum moisture content (OMC) was prepared manually for 5 minutes. Due to laboratory limitation of raw materials and capacity of testing equipment, brick samples having 15 mm diameter and approximate 15 mm height were prepared in the mould by applying uniaxially pressure of 5MPa for 10 second using through hydraulic press. After demoulding, samples were kept in air drying for 48 hours. The firing of brick samples was carried out in an electrical furnace for different holding time at 800, 900, 1000, 1100 and 1200°C temperature respectively. The firing was performed at a very slow rate of heating and cooling (5°C/ minute) to avoid generation of cracks in the brick samples due to moisture removal as well as phase transformation. Initial few brick samples were fired at different holding time (i.e. 60 to 720 minutes) to see the effect

and optimizing the holding time to get adequate strength with other properties. For determination of thermal conductivity 30 mm cube sample was prepared as mentioned in trial experiments.

4.3.3 Testing methodology

Brick samples were prepared and characterized for different properties like compressive strength, apparent porosity, bulk density, thermal conductivity, microstructure, and phase present as per methods described in the section 4.2.2 of this chapter. Permanent linear change (PLC) property was also evaluated by measuring the linear change in the sample with the help of slide caliper before and after heating. Besides the insulation bricks properties, water absorption characteristics were also examined for the suitability of building bricks as per ASTM C-67 (ASTM C67, 2013).

4.4 Results & Discussions

4.4.1 Properties of green brick samples

Volume change in the green brick samples made from both types of bottom ash was evaluated. It was observed that the PCC bottom ash green brick samples showed a drastic decrease in volume due to porous structure than FBC bottom ash as shown in Figure 4.10. The volume of PCC bottom ash brick samples is more due to porous structure. For the same reason, moisture requirement is also more. No significant changes was observed in the strength property after aging up to 100 days of oven dried samples as shown in Figure 4.11.

The photographs of green, dried as well as fired brick samples at different temperature were shown in Figure 4.12. With increasing firing temperature, the colour of samples was

changed from red to brown. At higher firing temperature the shrinkage inside the brick samples was also observed.

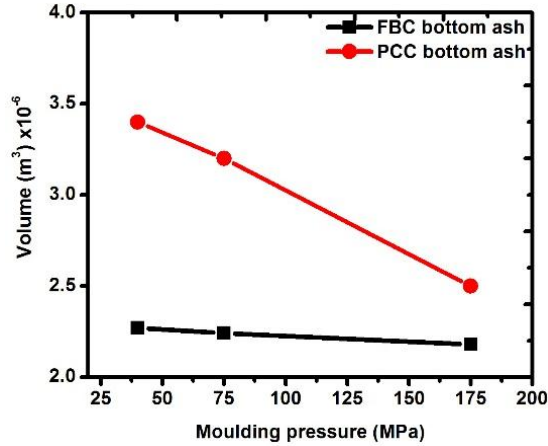


Figure 4.10 Green brick samples characteristics

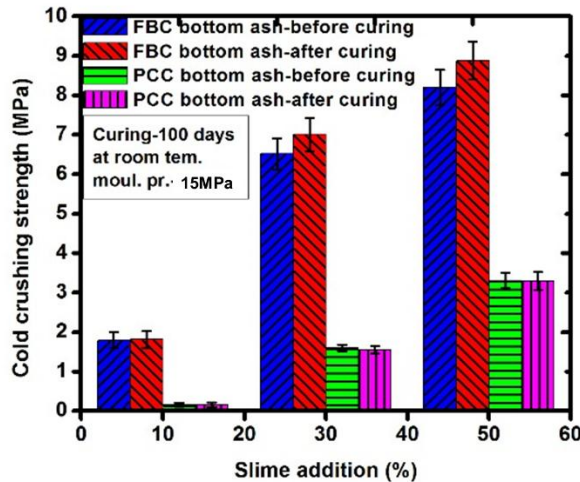


Figure 4.11 Effect of ageing on oven dried brick samples

4.4.2 Properties of fired brick samples

IS 2042 standard describes the classification of insulation bricks based on their service temperature condition where it could be used successfully (IS 2042, 2006). There are three categories based on the property of thermal conductivity, porosity, cold crushing strength, etc.

such as Grade-A, B, C respectively (as shown in Table B.5 of Appendix-B). Based on the properties achieved in the different brick samples in the present studies are categorized as per the specification of insulation bricks (IS 2042, 2006) as well as building bricks (ASTM C62, 2013), which are described in the following sections.

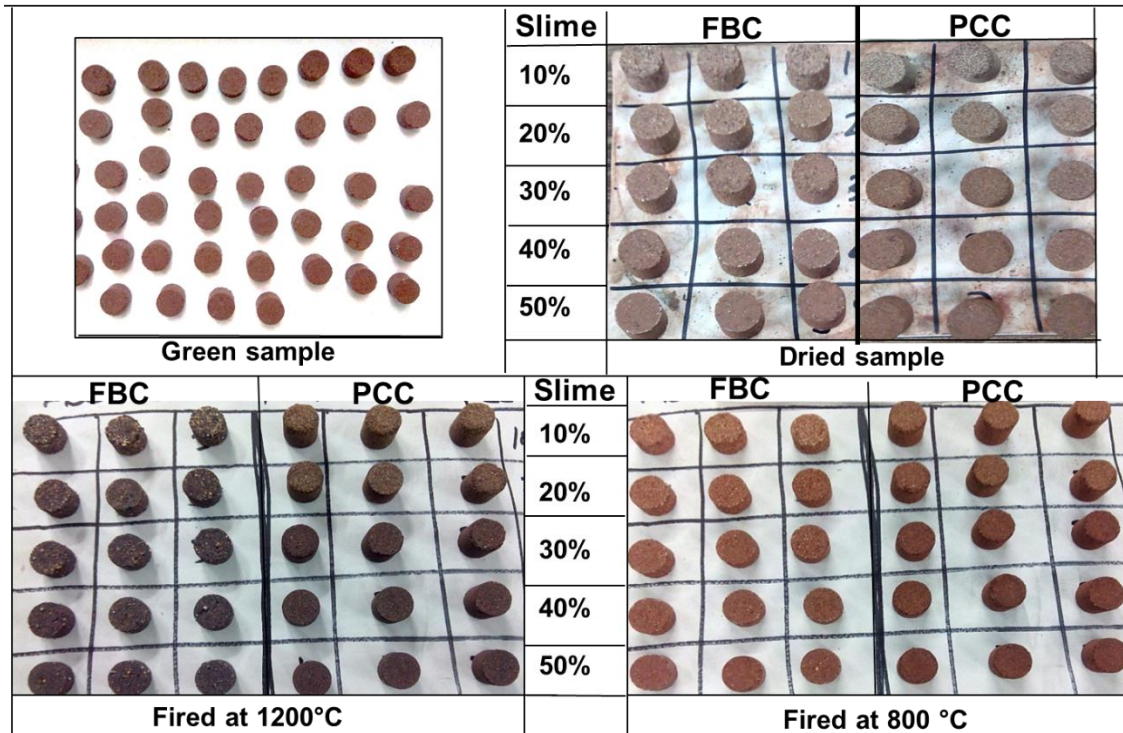


Figure 4.12 Photographs of brick samples at different conditions

4.4.2.1 Effect of firing time

In trail experiments, the firing time of brick samples was maintained for two hours. Firing schedule was maintained by the different author as well as standard tunnel kiln are different for clay bricks as well as insulation bricks (Demir et al., 2005; Taha et al., 2016; Tayler and Diadone, 2011). Therefore an experiment was conducted to get the optimum firing time for obtaining strength and porosity. It was observed that the samples of 360 minutes firing time

showed the maximum strength as well as comparable porosity, required for insulation bricks (as shown in Figure-A.1 of Appendix-A). Therefore, firing time was fixed to 360 minutes for all the remaining experiments in the present work.

4.4.2.2 Effect of firing temperature

(a) Cold crushing strength (CCS)

It is the main property to assess the suitability of its use for any type of bricks as per their application area. The requirement of compressive strength is relatively lower than the building bricks, but insulation property should be high. Increasing firing temperature and iron ore slime content at constant firing time (360 minutes) increased the strength values for both types of brick samples as shown in Figure 4.13. But, the strength was drastically increased at 1200°C fired brick samples having even lower iron ore slime content. It was observed that in the case of FBC bottom ash brick samples, the strength increment is more as compared with PCC brick samples. All the brick samples were made by FBC bottom ash which fulfill the criteria of crushing strength requirement of insulation bricks as per specification grade-A of IS2042 standard (IS 2042, 2006). It also fulfilled the crushing strength requirement of building bricks standard as per MW category of ASTM C62, except a few batches having a lower temperature of firing with a lesser amount of iron ore slime (ASTM C62, 2013) (as shown in Table B.4 of Appendix-B). In the case of PCC bottom ash brick samples, except some batches having lower iron ore slime content, all the brick samples fulfilled the criteria of crushing strength requirement of insulation bricks standard as per grade-A of IS2042 (IS 2042, 2006). But except few batches having higher iron ore slime content and firing temperature, remaining brick samples did not fulfill the compressive strength requirement of building bricks standard as per grade MW of ASTM C62 (ASTM C62, 2013). It is due to the formation of lower melting

phases at higher firing temperature which increases the strength. The increasing iron ore slime content enhances the bonding between the coarse bottom ash particles, resulting in more strength as it was observed in feasibility experiment tests as mentioned in section 4.2.

PCC bottom ash having the popcorn like porous structure, therefore the overall strength increment inside the brick samples was less than FBC bottom ash brick samples on similar conditions.

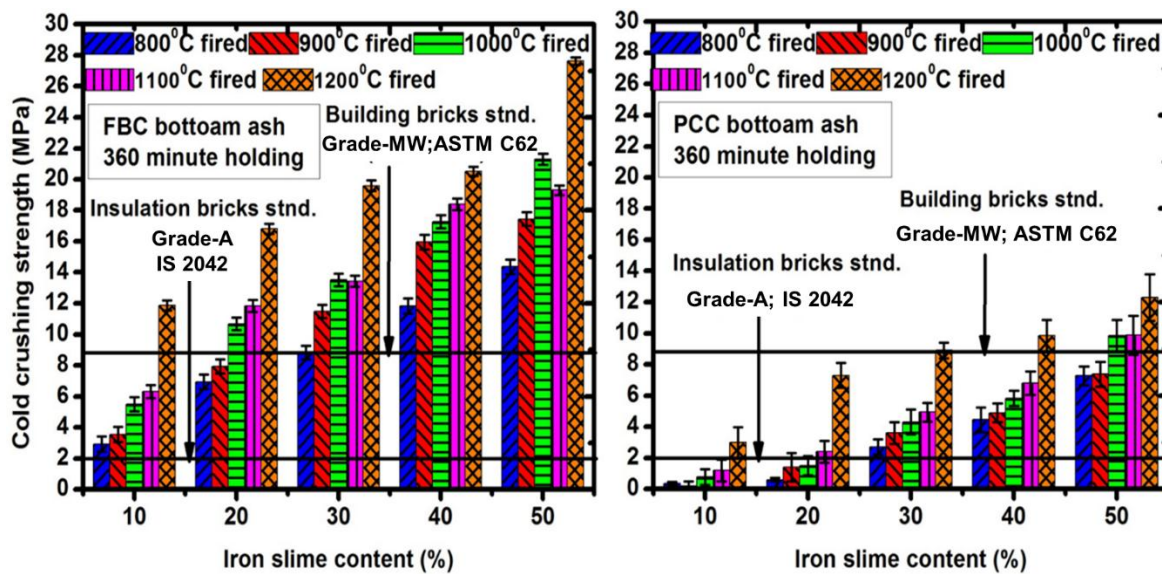


Figure 4.13 Effect of firing temperature on cold crushing strength

(b) Apparent porosity (AP)

These parameters generally measure to know the suitability of insulation bricks standard. The amount of lower melting phases was increased with increasing firing temperature and iron ore slime content in the brick samples which enters into the interparticle spaces of the coarse bottom ash, resulting in decreases in the porosity values of the brick samples (Figure 4.14). Except lower temperature fired (up to 900° C) brick samples, all the brick samples were made by FBC bottom ash which did not fulfill the criteria of insulation bricks as per grade-A of IS

2042 standard. But all the brick samples were made by the PCC bottom ash which fulfilled the criteria of insulation bricks as per grade-A of IS2042 standard (IS 2042, 2006).

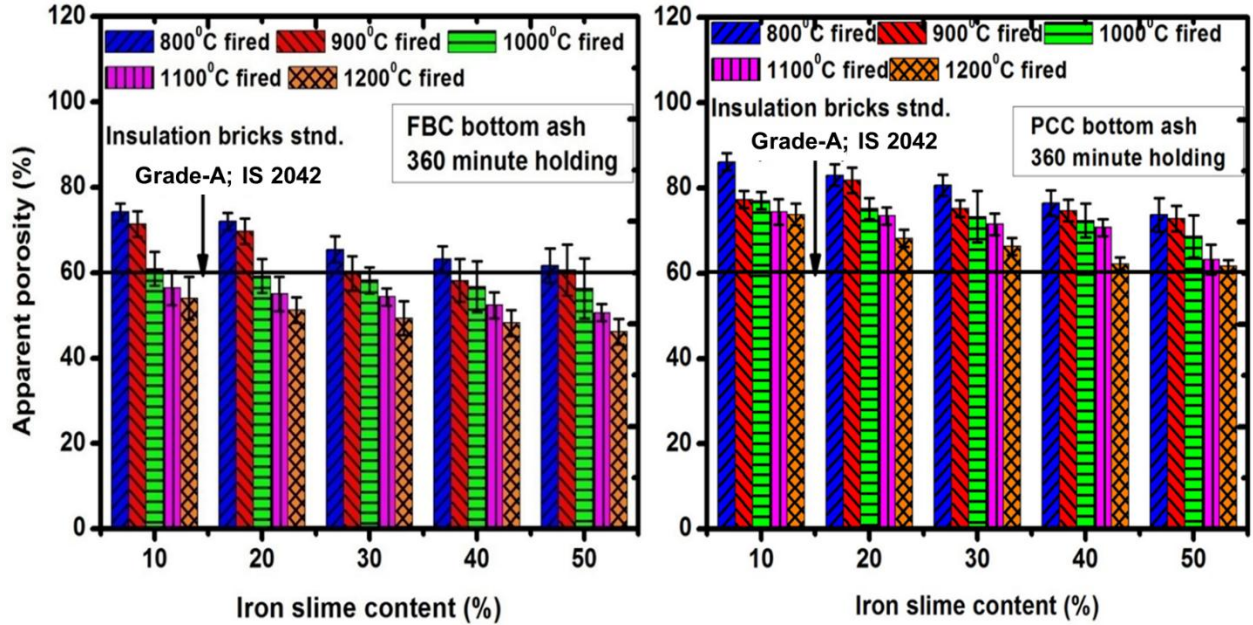


Figure 4.14 Effect of firing temperature and slime content on apparent porosity

(c) Water absorption (WA)

The property of water absorption is used for determining in the building bricks standard. To know the suitability of building bricks standard for both FBC and PCC bottom ash brick samples the water absorption values were also determined and the trends are shown in Figure 4.15. Increasing firing temperature and iron ore slime content decreased the water absorption as observed in the case of porosity.

It is observed from the Figure 4.15 that the value of water absorption in FBC brick samples fulfilled the criteria of building bricks standards as per grade MW of ASTM C62. On the other hand, due to higher porosity of PCC bottom ash brick samples which showed the higher water absorption values hence all the batches did not fulfill the criteria of water absorption for building bricks as per grade MW of ASTM C62. Only those brick samples, fired

at high temperature (1200°C) and more slime content (50%) which blocked the porosity hence satisfies the criteria of water absorption.

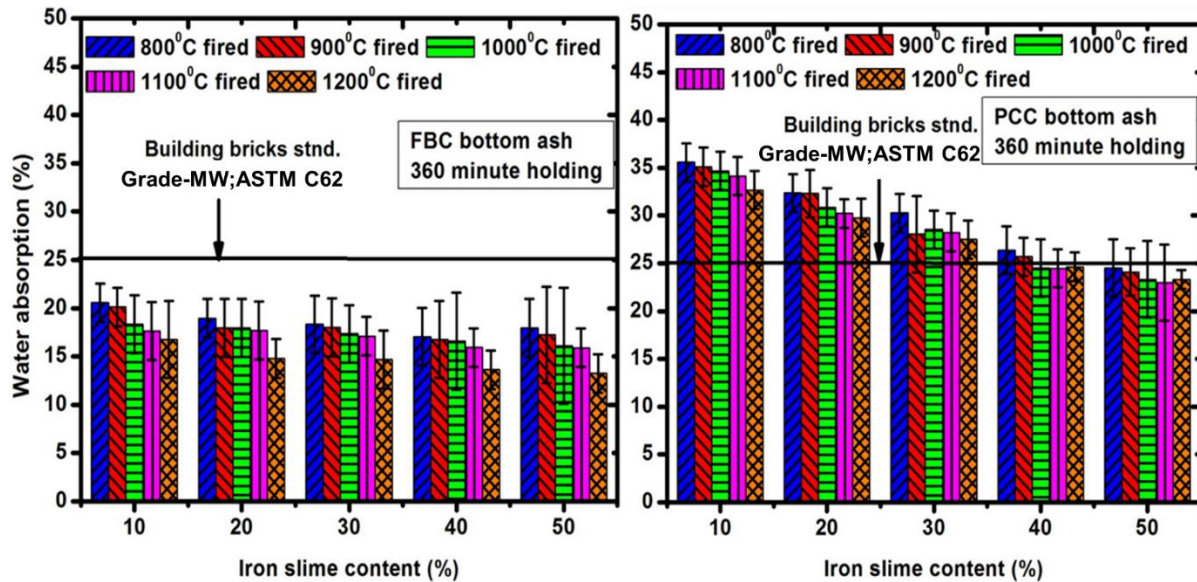


Figure 4.15 Effect of firing temperature and slime content on water absorption

(d) Bulk density (BD)

The value of bulk density for bricks are not as stringent as per standard, but it should be as low as possible because it exerts load through the wall of any construction and also increases the cost of transportation. In the present study, the bulk densities of fired brick samples were measured, and values were shown in Figure 4.16.

As described above that, with increasing firing temperature and iron ore slime content, the formation of lower melting phases occurred which fills the inter-particle spaces, resulting shrinking of the brick samples occurred which caused the increased bulk density value. Therefore, with increasing iron ore slime content and firing temperature, the bulk density values were increased in both types of brick samples (Figure 4.16).

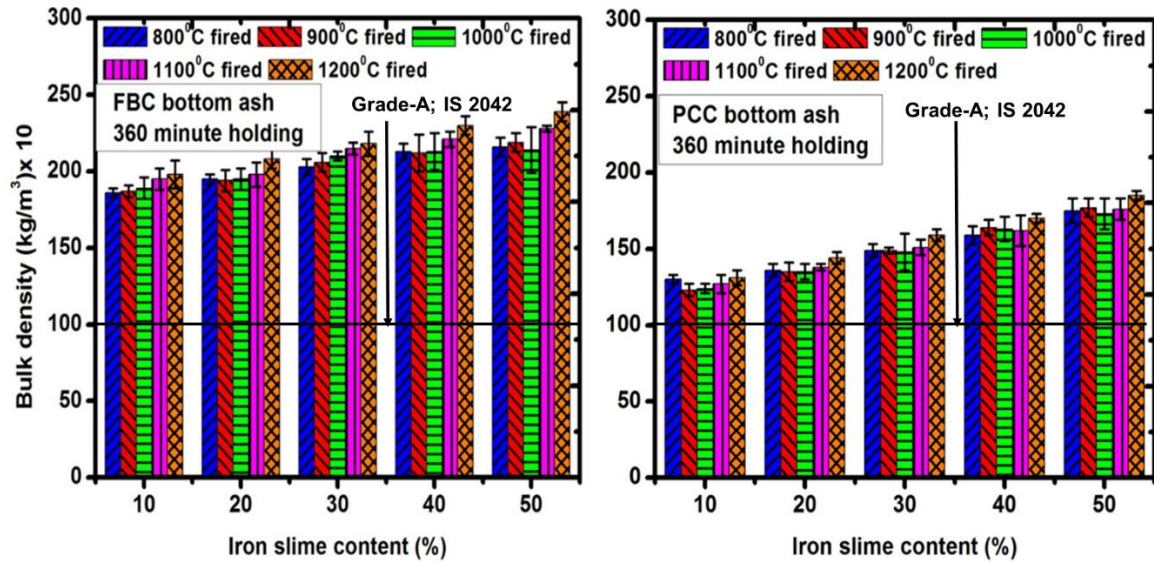


Figure 4.16 Effect of firing temperature and iron ore slime content on bulk density

In comparison to FBC, the brick samples of PCC bottom ash were showed relatively lower apparent density values which are due to its porous structure. There is no such strict standard, mentioned for the requirement of bulk density regarding building bricks, but the requirement for grade-A of IS 2042 standard for insulation bricks should be $<1000 \text{ kg/m}^3$ (IS 2042, 2006). In this respect, PCC bottom ash samples will be more preferred for making insulation bricks which could decrease the wall load during furnace construction. In the present study due to the use of iron ore slime content, increases the bulk density value beyond 1000 kg/m^3 , due to the presence of iron oxide inside the iron ore slime.

(e) Permanent linear change (PLC)

Permanent linear change is determined in the case of insulation bricks standards. Insulation bricks experiences the expansion or contraction (concerning linear change) behavior at a service temperature which should be minimum for sustainability of the wall structure. In the present study, it was observed that at lower temperature fired bricks have least PLC values in the case of all the brick samples as shown in Figure 4.17. It was noted that the PLC values

were drastically increased after increasing iron ore slime content beyond 20% with increasing firing temperature. If compared the values of FBC bottom ash brick samples to PCC brick samples, PCC brick samples have low PLC values. It may be due to the expansion during heating could be compensated by the presence of huge pores inside the brick samples.

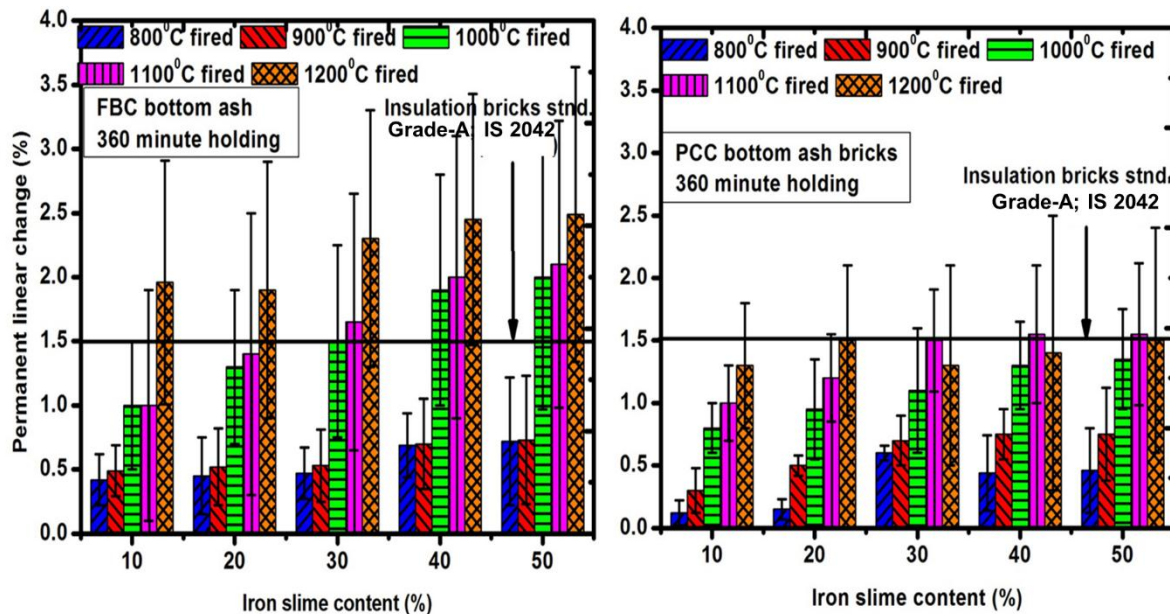


Figure 4.17 Effect of firing temperature and iron ore slime content on PLC

(f) Thermal conductivity (TC)

For the determination of thermal conductivity, 30 mm cubic samples were prepared from both FBC and PCC bottom ash using 10, 30 and 50% iron ore slime separately with keeping all other parameters similar to the cylindrical brick samples (designated as TC samples). The TC samples were fired at 800, 1000 and 1200°C for 360 minutes. The cold crushing strength and apparent porosity of above TC samples were determined. The obtained values were compared with the values of standard brick samples (cylindrical sample made previously for the property of CCS, AP, WA, BD, PLC determination). The results are shown in Figure 4.18. From the figure, it is cleared that the properties of TC samples lies in the range of cylindrical brick

samples with respect to CCS and apparent porosity only. Based on the results of cold crushing strength and apparent porosity of cubic TC samples, it was found identical with the value of cylindrical bricks samples.

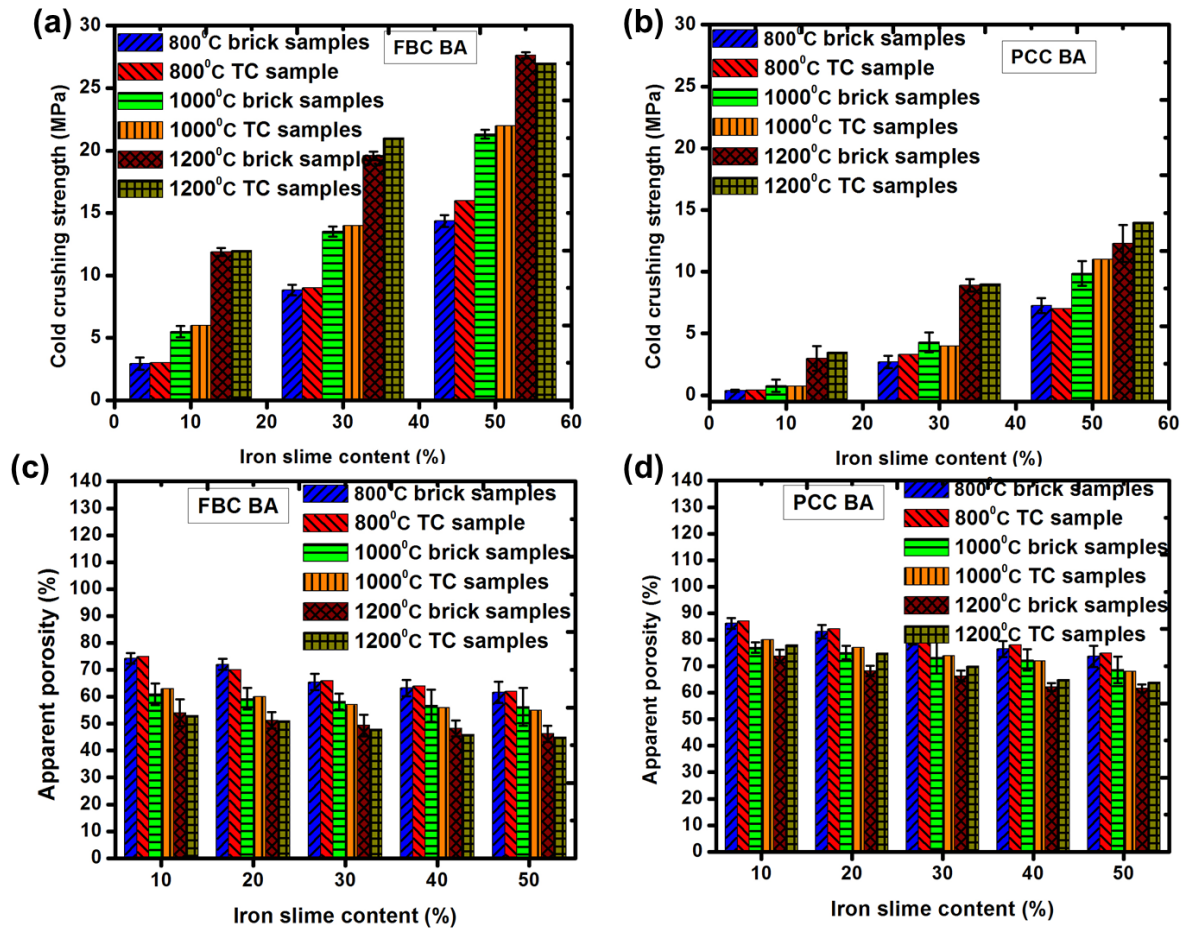


Figure 4.18 Comparison in properties of standard brick samples for thermal conductivity measurement with brick samples

The data on thermal conductivity of TC samples were generated at temperature for 200, 400, 600, 800 °C and calculated values are reported in Table B.6 of Appendix-B and trends are shown in the Figure 4.19 and 4.20 for FBC and PCC bottom ash respectively. In all types of brick samples, it was observed that with increasing measuring temperature, thermal conductivity values are increased successively. Increasing firing temperature and iron ore slime content increased the thermal conductivity of all the TC samples.

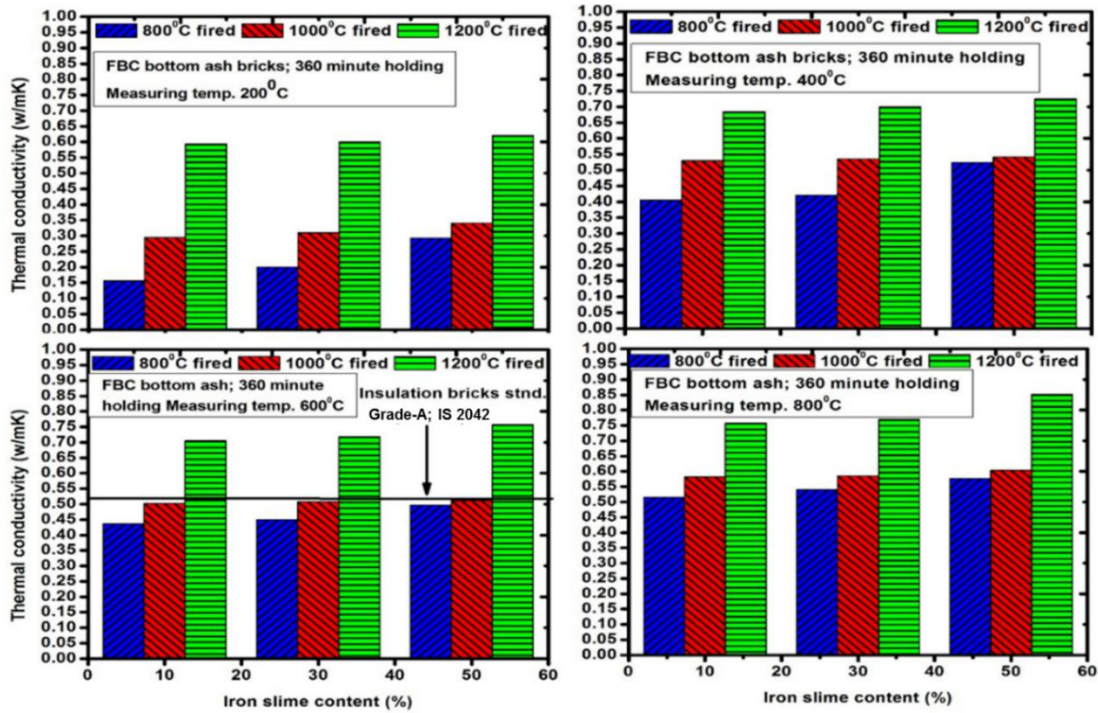


Figure 4. 19 Effect of firing temperature and slime content for thermal conductivity measured at different temperature for FBC bottom ash samples

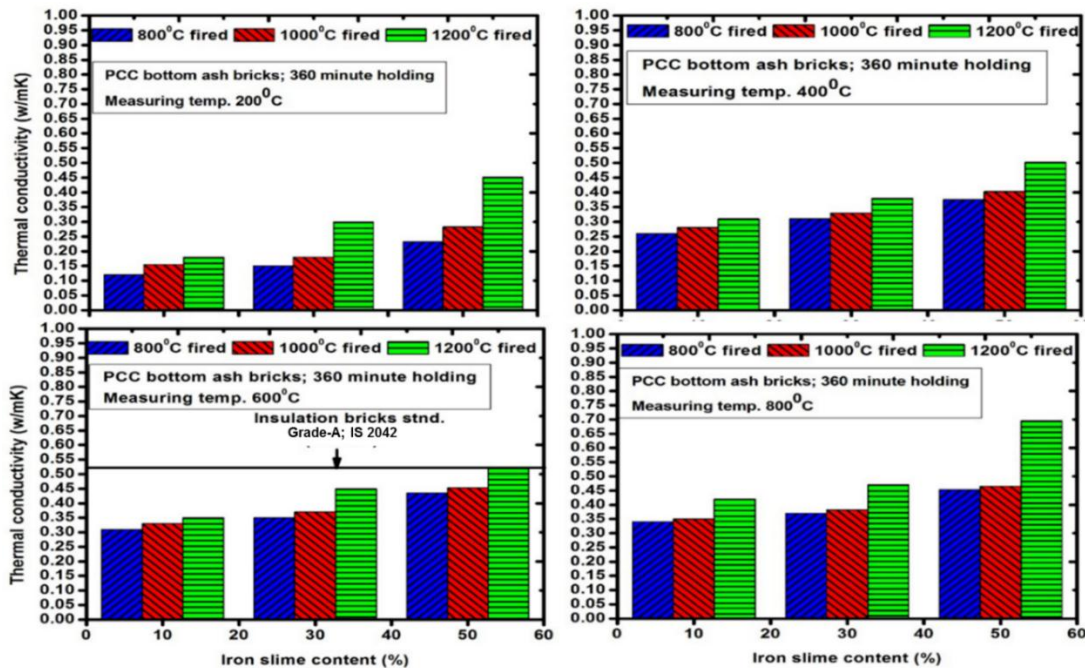


Figure 4. 20 Effect of firing temperature and slime content for thermal conductivity measured at different temperature for PCC bottom ash samples

The thermal conductivity for PCC, as compared to FBC, showed the lower values due to the presence of the porous structure.

TC samples made with FBC bottom ash, fired upto 1000°C were fulfilled the requirement of thermal insulation property for bricks as per grade-A of IS 2042 standard. But TC samples made with PCC bottom ash fired upto 1200°C, satisfied the criteria of insulation bricks (IS 2042, 2006).

(g) Phase analysis

Phases present inside the brick samples of FBC and PCC after firing for 360 minutes at different temperatures and iron ore slime content were observed which were shown in Figure 4.21.

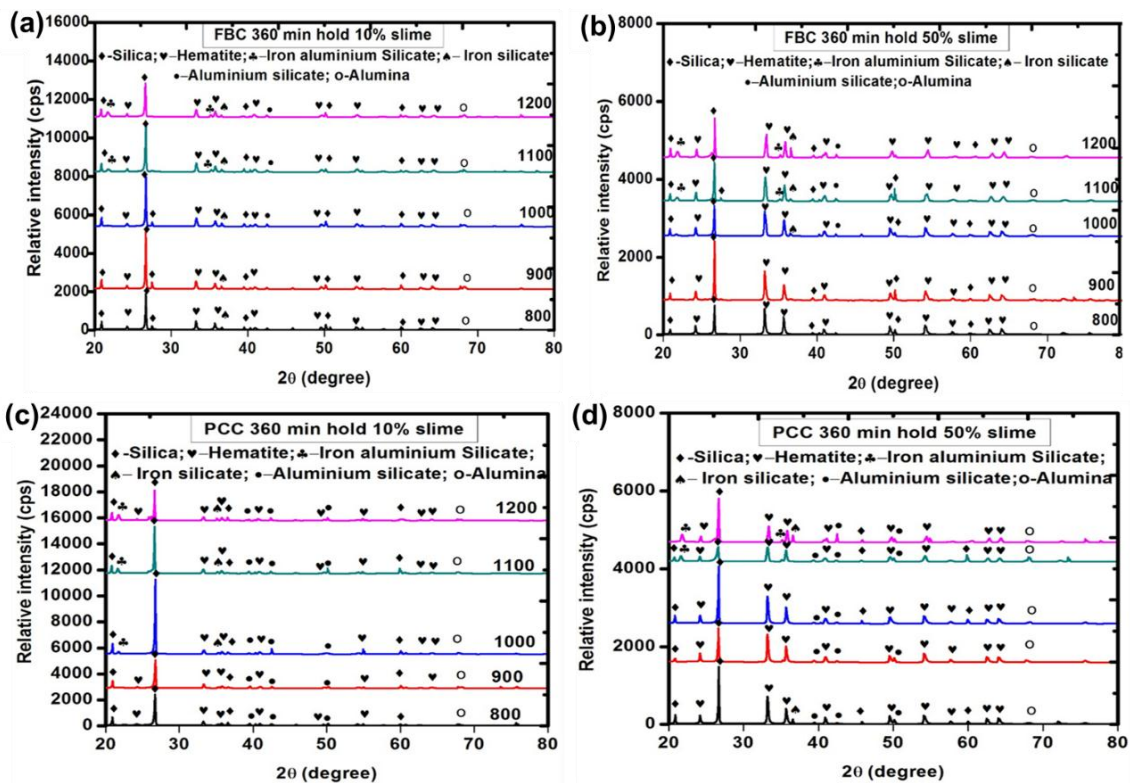


Figure 4.21 XRD analysis of fired FBC and PCC brick samples at different firing temperature and iron ore slime content

With increasing the firing temperature, more strength forming phases were found in all the brick samples. Iron ore slime content further enhances the amount of lower melting constituents as it was observed in trial experiments. But the amount of phase present are mere than the trial tests (120 minutes) and also observed the increased strength due to the increased holding time (i.e. 360 minutes) which may be allowed for the completion of phase transformation in the brick samples.

(h) Microstructural analysis

Micrographs of FBC and PCC bottom ash brick samples having 10% and 50% iron ore slime content fired at 800°C and 1200°C for 360 minutes are shown in Figure 4.22.

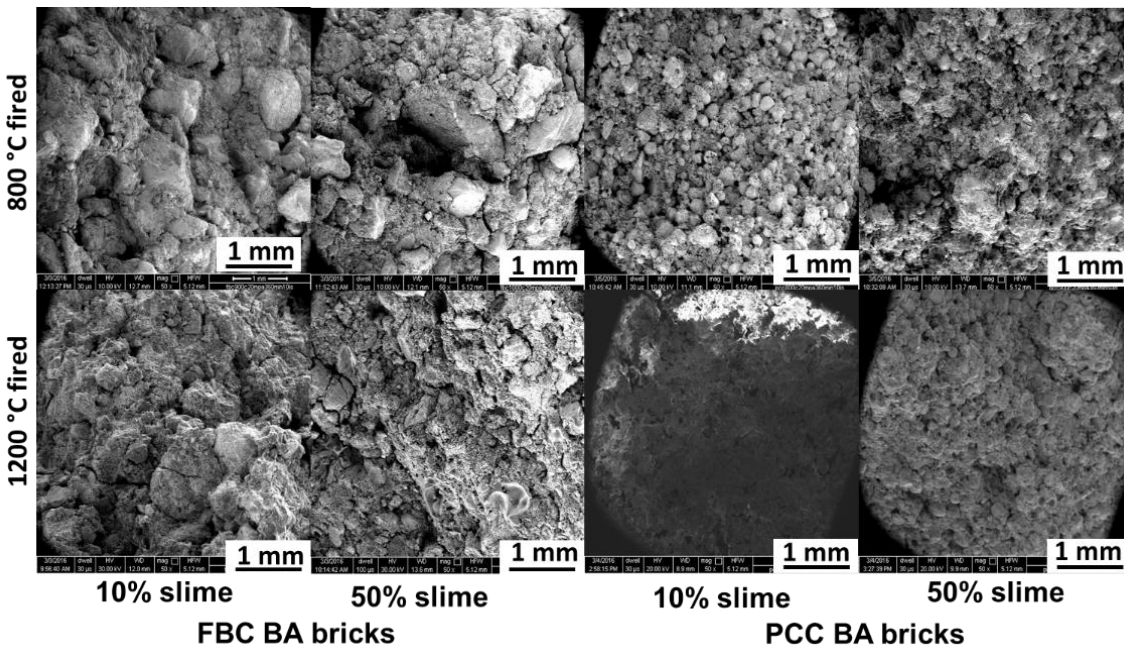


Figure 4. 22 SEM analysis of different bottom ash samples at different firing temperature and iron ore slime content

From the figure, it was evidenced that, the particle size of FBC bottom ash brick samples are coarser than PCC bottom ash although selected particle size for both cases were below one mm. The PCC bottom ash brick samples visualized as the more porous structure than FBC

bottom ash. With increasing firing temperature and iron ore slime content, the densification, as well as lower porosity, were observed in both types of bottom ash sample. But due to enough porosity in PCC bottom ash brick samples, all pores could not be blocked by the lower melting phases produced during firing. Therefore, densification effect in the case of PCC bottom ash was not pronounced. For the above said reasons, increased strength properties and decreased porosity were observed more in the case of FBC bottom ash brick samples in comparison to PCC bottom ash samples.

4.4.3 Tentative bonding mechanism

Based on the results obtained from the performed experiments as mentioned in the previous section 4.4.2.2(a-h), a tentative bonding mechanism could be predicted inside the brick samples as considering different variables shown in the Figure 4.23.

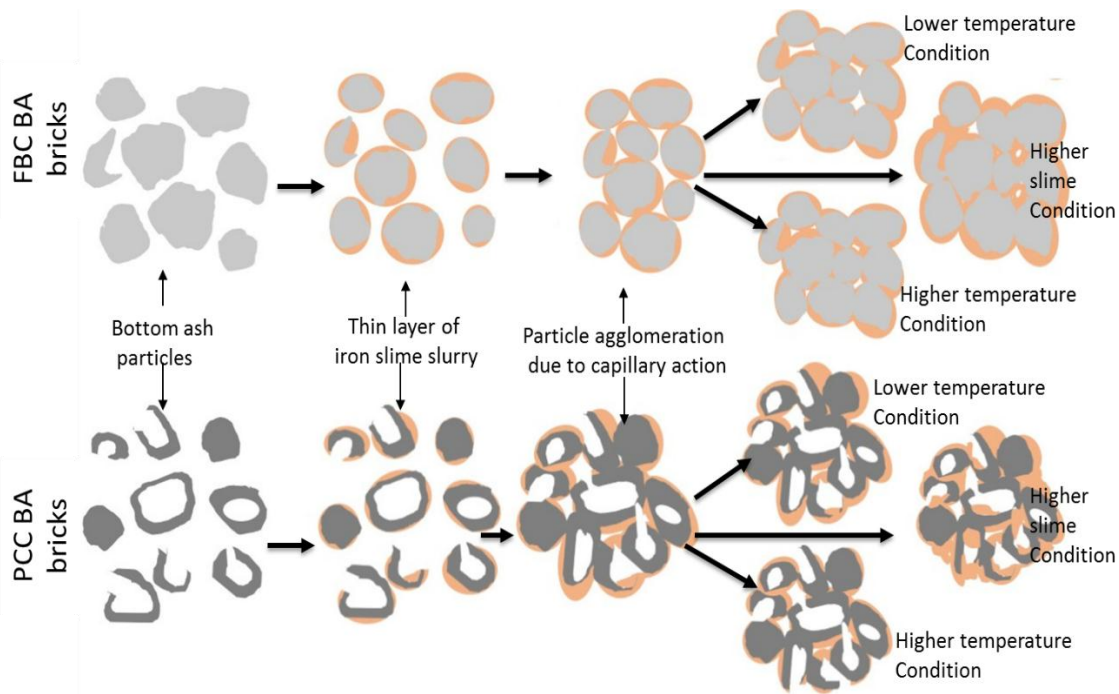


Figure 4.23 Tentative bond formation mechanism for different bottom ash samples at different firing temperature and iron ore slime content

Particles of bottom ash when comes in to contact with iron ore slime and water, a thin layer of iron ore slurry may be formed around the particle of bottom ash. Due to the capillary action of water, particles tends to agglomerate each other. Therefore after pressing the mixture of particles inside the mould, a green strength may be developed due to the closeness of the particles in the green brick samples. After drying, moisture was removed leaving very fine iron ore slime powder as a binder around the particles of bottom ash which provides the oxide bonding effect. At lower firing temperature condition (up to 900°C), the oxide bonding remains unaltered but slight increment in strength may be observed. With increasing the firing temperature (from 1000°C), the constituents of silica, alumina and iron oxide in the brick samples may be converted into different lower melting constituents, resulting in gradual increased in the strength properties. At still higher firing temperature (1200°C), the formation of lower melting constituent's may be increased which fills the inter-particle space of the brick samples. It resulted the shrinkage in volume which imparts more strength and density with less porosity values. These phenomena may be accelerated due to the presence of iron ore slime. Iron ore slime has very fine structure which may easily block the porosity and help to form the lower melting constituents resulting higher strength and density with decreasing porosity.

In the case of PCC bottom ash brick samples, due to the presence of porous popcorn like structure, significant breakage of the particles may be happened during pressing for making brick samples in the mould. Due to the enhanced porous structure of the PCC bottom ash, which does not fill entirely by the lower melting constituents or iron ore slime content, therefore, porosity values could not decrease to a greater extent as compared to FBC.

4.5 Conclusions

The following conclusions could be drawn based on the results obtained from the present studies:

1. The brick samples with iron ore slime as a binder was found to be effective in increasing the green crushing strength of the brick samples made from even coarser bottom ash particles.
2. The increasing firing time up to 360 minutes at all temperature were found to increase the cold crushing strength values. With further increased time, little decrease or no significant change was observed.
3. The increasing firing temperature upto 1200°C and iron ore slime content (max 50%) in the brick samples yielded high cold crushing strength value may be due to the formation of low melting constituents. For that reason lower water absorption and porosity and increased density was observed.
4. The minimum water absorption (13% for FBC and 23% for PCC), minimum porosity (46% for FBC and 62% for PCC), and maximum density (2390 kg/m³ for FBC and 1850 kg/m³ for PCC) were observed for 1200°C fired, 50% iron slime content brick samples.
5. Due to the porous structure of PCC bottom ash, the cold crushing strength, as well as bulk density was found to decrease and porosity increased as compared to FBC bottom ash brick samples.
6. The presence of iron oxide in iron ore slime appeared to form iron silicate, iron aluminium silicate phases in the presence of silica, alumina at high temperature which could be imparting the crushing strength in the fired brick samples.

7. The brick samples prepared by PCC bottom ash was found to fulfil the requirements of insulation bricks as per grade-A of IS: 2042, while brick samples made from FBC bottom ash fulfilled the requirements of building bricks as per grade-MW of ASTM C62 standard.
8. The insulation brick samples could be used in low to medium temperature applications up to 600°C which means that these bricks could be effectively utilized at the outer cold facing side of the furnaces wall as insulation bricks.