EFFECTS OF FLY ASH ADDITION ON THE MECHANICAL AND OTHER PROPERTIES OF CERAMIC TILES

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Abstract — The effect of fly ash additions on the mechanical properties of ceramic tile composition has been investigated. Fly ash addition in the range of 0-30wt% (Class-A) and 0-30wt% (Class-B) have been added into the tile body composition, wet milled, spray dried, shaped and fired at different temperatures (900, 1000, 1050 and 1100 °C). The MOR strength improved with increasing fly ash content and reached maximum when 30 wt% (Class-A) and 20 wt% (Class-B) fly ash used, and with greater additions it decreased. A linear correlation between strength development and Mullite formation was found. The tile with 30wt% fly ash (Class-A) and 20wt% (Class-B) have improved bending strength and have lowest porosities. The effects of fly ash incorporation on the mechanical properties of ceramic tiles and found that a small amount of fly ash addition improves the strength of the ceramic tiles.

Keywords — fly ash; ceramic tiles; mechanical properties; Mullite; MOR

1. Introduction

Recent industrial developments have drastically increased the amounts of waste materials [1], and many concerns have been raised regarding the treatment and disposal of waste materials. Fly ash produced in thermal power plants poses serious environmental problems. In 2012 about 10.5 million tons of fly ash generated in India to become a topic of the environmental and social interest [2]. Fly ash has been recycled through thermal treatment to be used for construction materials as aggregates, bricks, tiles and eco-cement [3,4].

The fly ash added ceramic files are low porosity, dense products with high technical performance [5-7], particularly with respect to abrasion and frost resistance, modulus of rupture and resistance to chemical attack. Due to the improved mechanical properties and aesthetic appearance, the last decade has shown a marked growth in fly ash added ceramic tiles.

The selection of raw materials for fly ash added ceramic tile is of utmost importance as it plays a vital role in ultimate product quality. A typical ceramic tile body (masse) consists of siO2 and Al2O3 as major oxides and CaO, MgO, Na2O, K2O and ZnO2 as minor components. Fe2O3 and TiO2 is kept to a minimum as they lead to a colored tile body. For supplemetning these compounds, the raw material is selected from a group of plastic and non-plastic minerals. Clayey minerals such as kaolinite, montmorillonite, illite etc. belong to the first group and contribute to strength development of green tiles. The second group consists of feldspar and quartz are used as flux[8] studied the influence if chemical composition on microstructural and mechanical properties of ceramic tiles.

The major constituents of fly ash are SiO2,Al2O3 and Fe2O3 with some minor constituents such as CaO, MgO, TiO2 and ZrO2 and thus may be considered as low cost resources materials for alumino-silicates. The potential of fly ash as a raw material for the ceramic industry has been reviewed by sent et al[9]. Uses of fly ash in ceramic tiles are reported in the literature [10-13]. Various efforts are being made at R&D institutes and universities to develop technologies for the gainful utilization of fly ash and the technical work done has gained prominence for further action towards commercialization.

On the basis of literature it was found that a controlled amount of fly ash addition improves the mechanical properties of ceramic tiles. Based on this observation, the present study was carried out to use fly ash as a source of alumino-silicate compounds to develop ceramic toles. Various composition have been developed using increasing properties of fly ash. The mechanical properties of tiles were studied with respect to fly ash content. Attempt has been made to correlate the properties with morphology (SEM) images.

2. Experimental Techniques
2.1 Chemical analysis

The ceramic tiles samples are made up of fly ash addition with different proportions (AS1 to AS4 and BS5 to BS7). The samples were obtained from a Government Ceramic Institute (ceramic plant), Vridhachalam, Tamilnadu, India. Upon collection, it was ground with a crushing machine. Ceramic tiles samples were subjected to chemical analysis with the aim to obtain accurate analysis for all elements present in the sample, in such a way that some of the elements were expressed as oxides which also reveal the type of the particles. The chemical analysis of the samples was made by using X-ray fluorescence (Bruker S4-Pioneer) instrument, Pondicherry University, Pondicherry, Tamilnadu, India. It was prepared by different mixture whose compositions are reported in Table 2.

A total of ten test piece for each composition were prepared to ensure the reproducibility of the measurements. Specimens were fired in a Laboratory electrical furnace simulating an industrial fast firing process in an air atmosphere involving basically: an average heating rate of 25 °C/min, a soaking temperature of 900, 1000, 1050 and 1100 °C hold an hour and the furnace cooling was performed by natural convection after turning the furnace off and leaving the specimen inside.

2.2 Mechanical Properties

2.2.1 Water absorption test

Water absorption is a key factor affecting durability of ceramic tiles samples. The less water infiltrates into a ceramic tile samples, the more durable is the ceramic tiles samples and the better is its resistance to the natural environment. The test specimens are ceramic tiles samples in the form of bars. The dry ceramic tiles samples were weighed and then submerged in water at a temperature between 55°C and 30°C. After 24 hours, the specimens were taken out of water. Then, the surface water of each specimen was wiped off with damp cloths and the specimens were weighed again.

Where,  
\[ W1 \] – weight of the dry specimen and  
\[ W2 \] – weight of the specimen after 24 hours of immersion in water.

2.2.2 Porosity

2.2.2.1 Role of porosity

The density or porosity affects a number of the properties of the ceramic tiles samples but probably the most important effect is its strength [14]. Highly porous ceramic tiles samples are mechanically weak. A ceramic tiles sample with the lowest porosity has the greatest strength, thermal conductivity and heat capacity. The water absorption method adopted to measure the porosity values of the ceramic body is described below. The samples were heated continuously in boiling water for about six hours and left to cool overnight which enables the pores to get filled up with water to saturation. The saturated specimens were then weighed by immersing in water as \( W1 \) and in air as \( W2 \). The samples were then placed in hot air oven at 200°C and dried for about six hours to remove the water contents completely and then weighed as \( W3 \). To standardize the values of the results the percentage of porosity was calculated using the relation:

\[
\text{Percentage of porosity} = \frac{W2 - W3}{W2 - W1} \times 100
\]

The stated procedure was repeated a number of times until consistency in the values were obtained and the average value was taken.

2.2.3 Modulus of rupture test (MOR)

A widely used method that measures transverse breaking strength to construction materials. The test is made on test bars the ends of which rest on knife edges while force is applied through a knife edge that is lowered midway between the ends. The breaking knife edges are moved by a dynamometer which measures the weight applied. Moduli of rupture tests were performed on a standard mechanical machine. Test specimens, measuring (16 X 8 X 1.5) cm for each ceramic tile samples composition were dried and fired at 950, 1050, 1150 and 1250°C along side with the ceramic tile samples. Each of them was placed one after the other on the bearing edges of the compression machine positioned 7.0 cm apart. Loads were then applied at the middle of the specimen, uniformly at 1.25 kgf per minute. The transverse breaking strength or modulus of rupture is calculate by the formula

\[
\text{MOR} = \frac{3PL}{2bd^2} \quad \text{(N cm}^{-2})
\]

where,  
\( L \) - the distance between two knife edges (cm),  
\( b \) - breadth of the specimen (cm),  
\( d \) - depth of the specimen, and  
\( P \) - breaking load in kg.

2.2.5 In the Present Study

In order to assess the strength of the various proportion quartz additive ceramic tile bodies obtained from Government ceramic institute Vridhachalam, Cuddalore Dt, Tamilnadu, India. From the four samples (class-A fly ash and class-B added) of ceramic tiles bodies, which compositions were made with high strength and important factors affecting the quality of the ceramic tile bodies were also discussed. This study discusses the strength analysis of the corresponding compositions and an attempt to interpret the chemical analysis of the composition in terms of their elemental composition. The chemical composition of the studied composition (determined by X-ray fluorescence at Pondicherry university laboratory Bruker S4-Pioneer) is present in Table 2. The behavior of the composition types regarding their chemical composition Vs their technological properties discuss in this paper.

3. Result and Discussion

The chemical composition of the masse, fly ash-A and fly ash-B was given in table 3.1. In terms of chemical compositions the SiO\(_2\) was the most abundant component, followed by Al\(_2\)O\(_3\). The masse also contains a reasonable amount of Potassium Oxide (K\(_2\)O) 3.985%. The Oxides K\(_2\)O, Na\(_2\)O, Fe\(_2\)O\(_3\), CaO and MgO are considered fluxes. They can influence the densification behaviour of the ceramic building materials during firing [15].

The main differences between the two fly ash (class-A and class-B) samples were the high CaO and SO\(_3\) content in fly ash-B sample. These differences suggest that the
higher amount of glass formed on the firing step can give rise to a lower porosity (Table 5.2) in the tile specimen prepared from (class-B) fly ash, which is also suggested by the higher thermal loss [16].

The other main differences between the masse and fly ash samples were the high CaO content (masse; 0.456% fly ash-A; 5.513% and fly ash-B; 8.351%), these suggest that the higher amount of fly ash additive added in the firing step can give rise to a higher porosity in the ceramic tiles.

From the chemical composition the sum of Fe$_2$O$_3$ and TiO$_2$ is 1.02% (masse), 4.24% (fly ash-A) and 5.24% (fly ash-B). Many studies have described the influence of these mineralizes in enhancing the process of sintering of ceramic matrix and the formation mullite [17] Ti$^{4+}$ and Fe$^{3+}$ play an important role by either substituting Al$^{3+}$ or by their integration into the structural interstices of the matrix.

The unfired tiles were prepared, and then fired at temperature 900, 1000, 1050 and 1100 °C for 6 hour in electric furnace respectively. The water absorption, porosity, modulus of rupture (some selected samples only) and compressive strength of fired fly ash added ceramic tiles (Table 2 and 3) were tested to decide the sintering temperatures.

3.1 Water Absorption

From the tables 3.2 and 3.3, the water absorption values varied from 25.83% (AS$_1$) to 13.75% (AS$_4$) in fly ash-A added ceramic tile samples. In respect of fly ash-B added ceramic tile samples the values varied from 25.83 % (AS$_1$) to 13.75% (BS$_5$). Water absorption as a function of firing temperature is shown in figure 3.1. From this figure it can be seen that the water absorption decreases when the percentage of fly ash (10, 20 and 30%) sample was less and also water absorption decreases with increase in the firing temperature.

The results show that for a firing temperature of 1050°C, the water absorption was decreased for the samples AS$_4$ (Table 3.2 and fig 3.1) and BS$_6$ (Table 3.3 and Fig 3.1). The samples of ceramic tiles (fly ash added) obtained from different proportions of fly ash (10, 20 and 30%) and they produced different values of water absorption.

Water absorption is a reliable indicator of the degree of the tile body sintering. The tables 3.2 and 3.3 give an example of the results of water absorption tests for one of the fly ash mix compositions (average for batches of 3) as a function of the firing temperature. While slight reduction in water absorption with progressive firing temperature is obvious, a substantial decrease in water absorption occurred between 1050°C, and 1100°C. Fig.3.1 depicts the water absorption values for the ceramic tiles. This physical property is very important, because it is related to the open porosity of the fired products. This is probably due to the reduction of the viscosity of the glassy phase, which accelerates the sintering process [15]. Hence, the glassy phase formed during firing fills the pores, and decreases the open porosity level of the ceramic tiles.

Fig: 3.1. Water absorption of ceramic tiles sintered at various temperatures

3.2 Porosity

From the tables 3.2 and 3.3 and fig3.2 the values of porosities of sample from AS$_1$ to AS$_4$ vary for different temperatures. Finally, since, water absorption is directly related to open porosity, its value decreases in the overall temperature range. Since the bloating is a process commonly used for the production of ceramic materials, e.g., aggregate for concrete it has been studied by several researches [18-20]. The mechanism of bloating by iron (III) Oxide reduction is well known; [21] gave a description of the fundamental role of iron oxide in the bloating of vitrified ceramic materials with iron (III) oxide content between 1 and 6 wt%.

At elevated temperature, Fe$_2$O$_3$ is partially reduced with the production of oxygen as the bloating gaseous phase, generating large pores within the fired body and decreasing a density decrease. The larger fly ash organic content matter the greater the porosity and shorter the path among particles for gas diffusion.

Therefore, a higher fly ash addition (30%) ratio increases the open pore volume and decreases the strength of sintering ceramic tile specimens (Tables 3.2 and 3.3).Fig.3.2 shows that the porosity of ceramic tile specimens increased with fly ash addition and decreased with the sintering temperature. With 1100°C sintering temperature the specimen porosity increased from 13.75% to 18.20% with decrease in added. fly ash-A from 20% to 30% (as shown in Fig 3.2). In respect of fly ash-B added ceramic tile samples the porosity value increased from 13.26% to 16.81% sintered at 1050–1100°C, (Fig.5.2). Only samples of the series AS$_4$ and BS$_6$ show an appreciable improvement in their mechanical behavior when they reached 1050°C.

Fig: 3.2. Porosity of ceramic tiles sintered at various temperatures

3.3 Modulus Of Rupture (Selected Samples (AS$_1$, AS$_4$ And AS$_1$, BS$_6$)).

The change of Modulus of rupture (MOR) values, with increase of the firing temperature from 900 to 1100°C are given in tables 5.2 & 5.3 and Fig.5.3. The obtained results
show that the increase of firing temperatures (1050°C) lead to the increase of MOR values. (AS₁ and BS₆). According to the fig.3.3 it was verified, 0% of fly ash additive tiles (standard) while for 30% (AS₂) and 20% (BS₆) wt. % of fly ash, a strength increase is appreciated. This high value may be attributed to the pre distribution and the vitrification level of the tile body. However, as shown in Fig.3.3, bending strengths of glazed tiles improved to 1.5 times than standard tiles. Glazes melted tightly into body tiles in the sintering process at high temperatures. After crystallization they were rearranged, melted glazes formed a hard layer on the surface of tile, which could improve the bending strength of tiles. It was verified that all samples with fly ash-A & fly ash-B waste up to 30% have values in agreement to literature [22]. According to tables.3.2 and 3.3, it can be observed a decrease in the water absorption and an increase in the modulus of rupture with the elevation of the firing temperature, which is related with the fusion and vitrification of the fly ash (A&B) that acted as fluxes in the studied temperatures.

The increased amount of liquid phase as higher temperature certainly affects negatively the mechanical strength. These results correlate well with the samples microstructure, the absence of carbonate and thermal transformation within Al₂O₃-SiO₂ composition, cause a formation of a considerable amount of amorphous/glassy phase and sealed porosity. Consequently, these conditions cause higher values of bending strength in the ceramic tile samples compared to that of the standard values [22].

Maximum compressive strength occurs at 1050°C sintering temperatures with 30% fly ash-A addition and 20% fly ash-B addition (Fig.3.4). The compressive strength decreased with the fly ash-B content above 20%. The maximum compressive strength (Table 3.3) of ceramic tiles (fly ash-B added) manufactured with 20% fly ash is reached. The addition fly ash (class-A and class-B) in ceramic compositions for production of tiles, up to 30% (fly ash-A) and 20% (fly ash-B) in weight, and firing at 1050°C they can be used to produce ceramic tiles within the acceptable limits for industrial production.

Since, the AS₄ (ceramic tiles with 30% fly ash class-A) and BS₆ (ceramic tiles with 20% fly ash class-B) compositions have shown the best properties compared to all other compositions, SEM analysis of the samples AS₄ and BS₆ compositions fired at 1050°C were carried out and are shown in figs. 6.12 and 6.17. The dense microstructure is characterized by a very small number of pores. Interlocked Mullite and quartz crystals embedded in glassy matrix. The formations of needle shaped clusters of crystals are also observed in micrographs. EDAX analysis revealed that these crystals are of Mullite and quartz compositions. This dense microstructure is responsible for good mechanical properties of tiles.
Fig: 3.5 SEM images (Different Magnification) Of Neyveli Lignite Fly Ash Sample (AS)

Fig: 3.6 SEM images (Different Magnification) Of Neyveli Lignite Fly Ash (class - B) Sample (BS)

Fig: 3.7 SEM images and EDX spectra of ceramic tile made from mass with 30% fly ash [class A (AS)] sintered at 1050°C

Fig: 3.8. SEM images and EDX spectra of ceramic tile made from mass with 20% fly ash [class B (BS)] sintered at 1050°C

Conclusions

The effect of fly ash additions on the properties of ceramic tiles has been studied. During firing, fly ash powder waste accelerates the densification process, with some positive effects (lower porosity, water absorption) combined with higher compressive strength. The reduction in strength for the tiles containing more than 20% (fly ash class-B) is due to increased glass phase content. A linear correlation between Mullite formations and strength development was found. The firing temperature (1050°C) of fly ash added ceramic tiles was the most important advantage of ceramic tiles and makes the use of fly ash an economical attractive alternative.

References

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