



Effects of flyash 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 30wt% (Class-A) and 20wt% (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.

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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. Flyash 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 flyash 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 flyash added ceramic tiles.

The selection of raw materials for flyash 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 SiO_2 and Al_2O_3 as major oxides and CaO , MgO , Na_2O , K_2O , and ZnO_2 as minor components. Fe_2O_3 and TiO_2 is kept to a minimum as they lead to a colored tile body. For supplementing 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 flyash are SiO_2 , Al_2O_3 and Fe_2O_3 with some minor constituents such as CaO , MgO , TiO_2 and ZrO_2 and thus may be considered as low cost resources materials for alumino-silicates. The potential of flyash as a raw material for the ceramic industry has been reviewed by sent et

al [9]. Uses of flyash 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 flyash and the technical work done has gained prominence for further action towards commercialization.

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

Experimental Techniques

Chemical analysis

The ceramic tiles samples are made up of flyash 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.

Mechanical Properties

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.

Porosity

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.

Modulus of rupture test (M.O.R)

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 tiles samples composition were dried and fired at 950, 1050, 1150 and 1250°C alongside with the ceramic tiles 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

$$\text{M.O.R.} = \frac{3PL}{2bd^2} \quad (\text{N cm}^2)$$

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.

In the present study

In order to assess the strength of the various proportion quartz additive ceramic ceramic tiles bodies obtained from Government ceramic institute Vridhachalam, Cuddalore Dt, Tamilnadu, India. From the four samples (class-A flyash and class-B added) of ceramic tiles bodies, which compositions were made with high strength and important factors affecting the quality of the ceramic tiles body 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.

Result and Discussion

The chemical composition of the masse, flyash-A and flyash-B was given in table 3.1. In terms of chemical compositions the SiO₂ was the most abundant component, followed by Al₂O₃. The masse also contains a reasonable amount of Potassium Oxide (K₂O) 3.985%. The Oxides K₂O, Na₂O, Fe₂O₃, 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 flyash (class-A and class-B) samples were the high CaO and SO₃² content in flyash-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) flyash, which is also suggested by the higher thermal loss [16].

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

From the chemical composition the sum of Fe₂O₃ and TiO₂ is 1.02% (masse), 4.24% (fly ash-A) and 5.24% (flyash-B). Many studies have described the influence of these mineralizes in enhancing the process of sintering of ceramic matrix and the formation mullite [17] Ti⁴⁺ and Fe³⁺ play an important role by either substituting Al³⁺ 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 flyash added ceramic tiles (Table 2 and 3) were tested to decide the sintering temperatures.

Water Absorption

From the tables 3.2 and 3.3, the water absorption values varied from 25.83% (AS₁) to 13.75% (AS₃) in flyash-A added ceramic tile samples. In respect of flyash-B added ceramic tile samples the values varied from 25.83 % (AS₁) to 13.26% (BS₆). 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 flyash (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 AS₄ (Table 3.2 and fig 3.1) and BS₆ (Table 3.3 and Fig 3.1). The samples of

ceramic tiles (flyash added) obtained from different proportions of flyash (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 flyash 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.

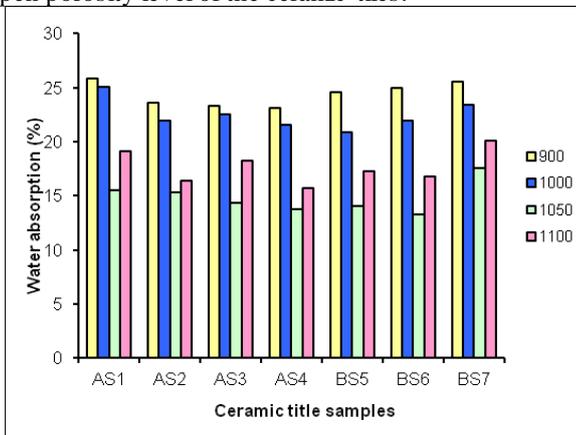


Fig. 3.1. Water absorption of ceramic tiles sintered at various temperatures

Porosity

From the tables 3.2 and 3.3 and fig3.2 the values of porosities of sample from AS₁ to AS₄ 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, Fe₂O₃ is partially reduced with the production of oxygen as the bloating gaseous phase, generating large pores within the fired body and determining a density decrease. The larger flyash organic content matter the greater the porosity and shorter the path among particles for gas diffusion.

Therefore, a higher flyash 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 flyash 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. flyash-A from 20% to 30% (as shown in Fig. 3.2). In respect of flyash-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 AS₄ and BS₆ show an appreciable improvement in their mechanical behavior when they reached 1050°C.

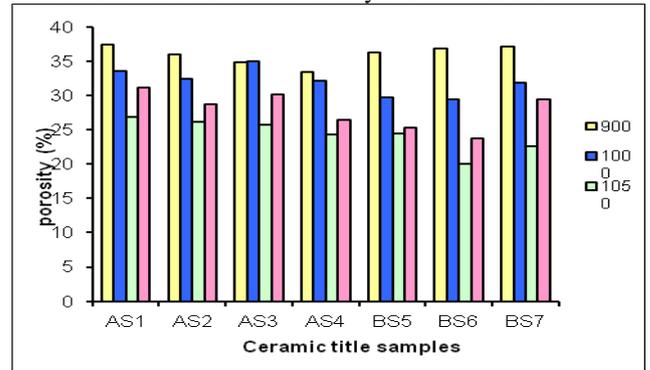


Fig. 3.2. Porosity of ceramic tiles sintered at various temperatures

Modulus of rupture (selected samples (AS₁, AS₄ AND AS₁, BS₆)).

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. (AS₄ and BS₆).

According to the fig.3.3 it was verified, 0% of flyash additive tiles (standard) while for 30% (AS₄) and 20% (BS₆) wt. % offlyash, a strength increase is appreciated. This high value may be attributed to the predistribution 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 tile bodies in the sintering process at high temperatures. After crystallization 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 flyash-A & flyash-B waste upto 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 flyash (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]

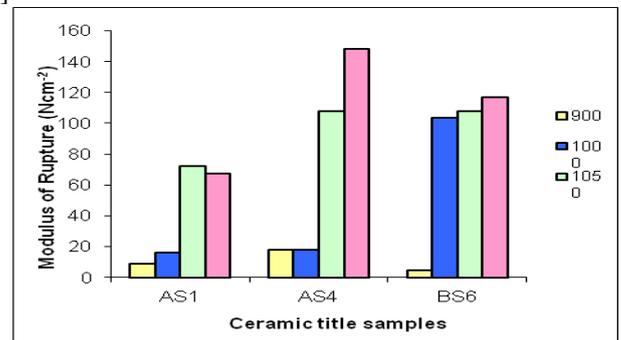


Fig. 3.3 Modulus of Rupture of ceramic tiles (selected samples) at various temperatures

Compressive Strength

In the present work, the compressive strength was mainly affected by sintering temperature effects. Fig.3.4 indicates that the higher compressive strength developed at 1050°C sintering temperature and 30% flyash –A addition (AS₄ and 20% flyash-B addition tiles samples, ranging from approximately 1500 kgf (AS₁ at 1100°C) to 3761 kgf (AS₄ at 1050°C) and 1500 kgf (AS₁ at 1100°C) to 2286 kgf (BS₆ at 1050°C).

Maximum compressive strength occurs at 1050°C sintering temperatures with 30% flyash-A addition and 20% flyash-B addition (Fig.3.4). The compressive strength decreased with the flyash-B content above 20%. The maximum compressive strength (Table 3.3) of ceramic tiles (flyash-B added) manufactured with 20% flyash is reached.

All ceramic tile bodies increase their compressive strength with temperature in approximately the same way. According to the fundamental of fracture mechanics [23] the larger defects has the lower materials strength. From our results, the ceramic tile samples (AS₁-AS₄) and (BS₅ – BS₇) have the quantity of higher water absorption where compared with other mixtures (AS₄& BS₆) which contain less value. Therefore, the compressive strength of the sintered specimen increased resulting in increasing the sintered specimen density.

When sintered at temperature 1050°C, the good quality and high compressive strength of building tiles were successfully manufactured from reservoir flyash served as the raw material above.

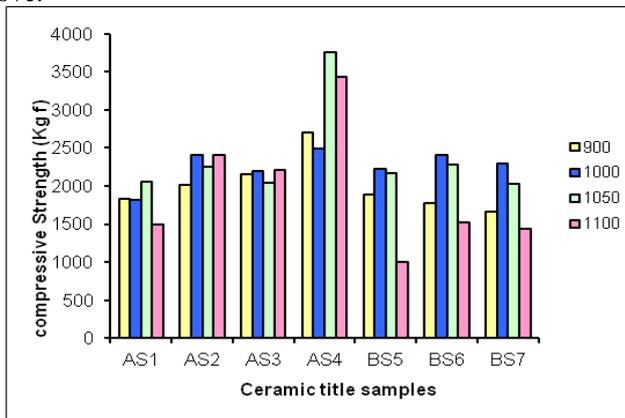


Fig: 3.4. compressive strength of ceramic tiles sintered at various temperatures

Maximum compressive strength occurs at 1050°C sintering temperatures with 30% flyash-A addition and 20% flyash-B addition (Fig.3.4). The compressive strength decreased with the flyash-B content above 20%. The maximum compressive strength (Table 3.3) of ceramic tiles (flyash-B added) manufactured with 20% flyash is reached.

The addition flyash (class-A and class-B) in ceramic compositions for production of tiles, upto 30% (flyash-A) and 20% (flyash-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, scanning electron microscopy of the samples AS4 and BF6 compositions fired at 1050°C were carried out and the micrographs 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.

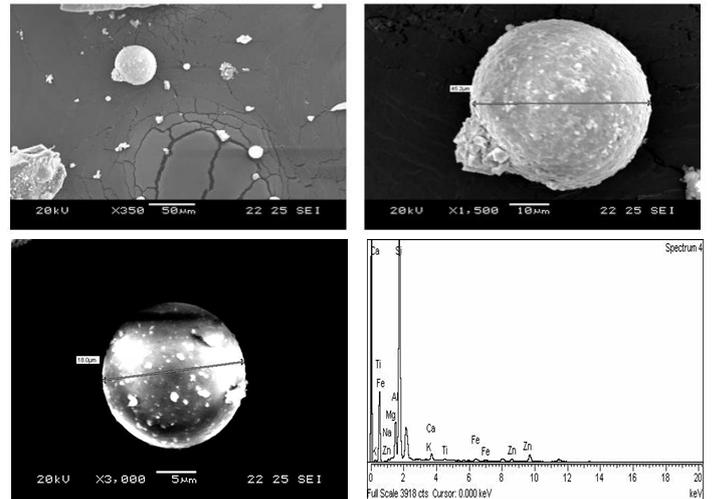


Fig: 3.5 SEM images (Different Magnification) Of Neyveli Lignite Fly Ash Sample (AS)

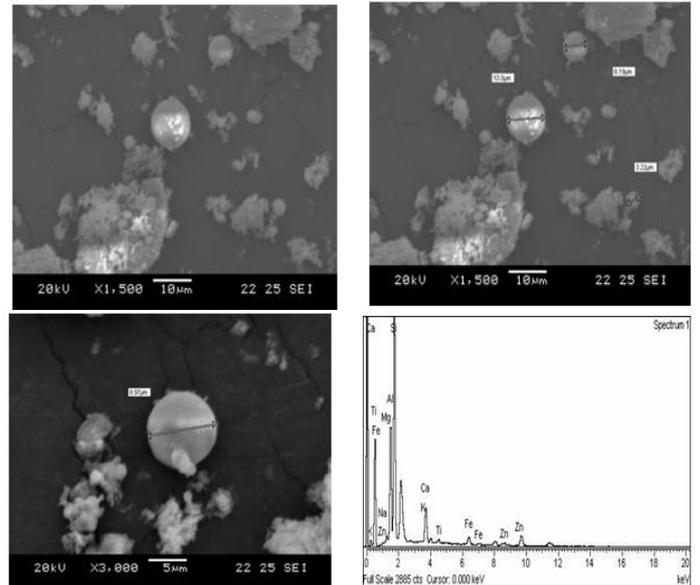


Fig: 3.6 SEM images (Different Magnification)Of Neyveli Lignite Fly Ash (class - B) Sample (BS)

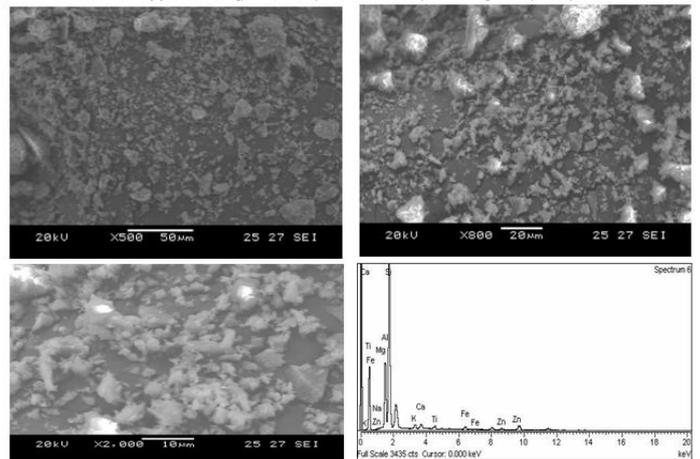


Fig:3.7 SEM images and EDX spectra of ceramic tile made from masse with 30% fly ash [class A (AS₄)] sintered at 1050° C

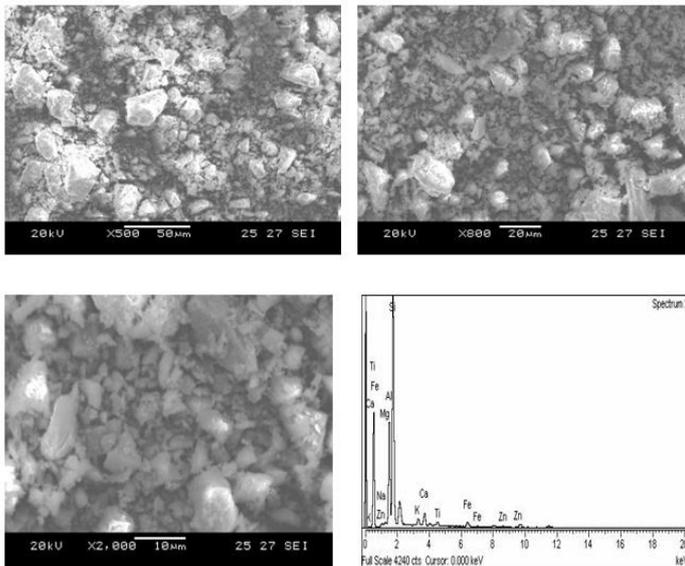


Fig: 3.8 SEM images and EDX spectra of ceramic tile made from masse with 20% fly ash [class B (BS₆)] sintered at 1050° C

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 and economical attractive alternative.

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