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Effect of addition of talc on the sintering characteristics of fly ash based ceramic tiles

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Abstract

The sintering characteristics of fly ash–talc mixtures having 0–100% talc (w/w) in the presence of 10% (w/w) sodium hexa meta phosphate (SHMP) have been studied. In the fly ash rich mixtures, the presence of needle shaped mullite and rhombohedral aluminum phosphate phases is responsible for providing impact strength to sintered tile bodies. On gradual addition of talc, the decrease in the concentration of sillimanite and increase in the concentration of sodium magnesium phosphate crystals is responsible for the observed improvement in the impact strength of tiles. With increase in talc content, initially the % water absorption decreases to reach a minimum for a fly ash–talc mix containing 60% (w/w talc) where after it again starts increasing. The apparent density of the tile samples increases with increasing talc content in the raw mix due to densification as well as higher density of talc as compared to fly ash.

Keywords: Sintering; Talc; Fly ash; Phosphates; Tiles

1. Introduction

Coal fired thermal power plants are the main source of electricity supply in India. The large quantity of coal burned in these plants generates huge quantity of fly ash. On a per unit of energy production basis, Indian thermal power plants generate more fly ash as the Indian coals contain more silica than the coals found in other countries.[1] It is estimated that currently about 90 million tons of fly ash is generated[2] every year in India alone. Only a small quantity of the total fly ash generated is utilized in making bricks, concrete blocks or blending with cement.[3–5] The extensive research carried out in recent years for finding further applications of fly ash have led to the development of processes for making, hollow/masonry/concrete blocks,[6–8] glass ceramics,[9–11] ceramic wares,[12] silicon carbide, silicon nitride, sialon, β-cordierite,[13] mullite[14] and separation of cenospheres[15] for use as extenders for plastic compounds, synthetic foams with better mechanical properties, automobile industries, paints, coatings, fire and heat protection devices, etc. Attempts have also been made to use fly ash for making synthetic zeolites[16–18] and in poultry feed.[19] Since extremely large quantity of fly ash finds its way into fly ash ponds, which not only occupy valuable land resources but also pose a threat to ground water, there is a need to find applications where fly ash may find bulk utilization. Since chemically fly ash mainly comprises of silica and alumina,[12] it is an attractive candidate for making ceramic bodies such as wall and floor tiles, etc. However, the presence of silica in the form of quartz in fly ash causes low thermal shock resistance in the fired bodies and hence low impact strength. In our earlier paper[20] we investigated the effect of addition of pyrophyllite (ideal formula[21] $Al_4Si_4O_{10}(OH)_4$) to fly ash for making ceramic tiles making use of phosphate binding. In the present paper, we report the results of our studies on the addition of talc (ideal formula[21,22] ($Mg_3Si_4O_{10}(OH)_2$), a mineral belonging to the pyrophyllite family,[21] to fly ash for making sintered tiles.

2. Materials and methods

2.1. Raw materials and chemicals

The representative sample of fly ash, collected from the five electrostatic precipitators of the thermal power plant

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at Sarni over a period of two years, was prepared by the method of coning and quartering. The particle size analysis of the representative sample was carried out using the wet sieve analysis method. The analysis showed that 75% of the powder was below 48 μm and the balance 25% was in the particle size range 48–78 μm.

Talc mineral lumps were procured from mines located in Jabalpur District of M.P., India. The lumps were crushed manually to small pieces and then powdered in a ball mill of 2.5 kg capacity. The results of sieve analysis of the powdered sample showed that 1.8% of the sample was of +400 μm, the maximum percentage (i.e. 90.2%) was in the range −400 to 150 μm, 7.9% was in the range −150 to 75 μm and 0.1% was of −75 μm size. The X-ray diffraction studies of the sample confirmed it to be the tri layer monoclinic form of the talc mineral wherein a octahedral layer of magnesium ions is sandwiched between two sheets of SiO_4 tetrahedra.[23–25]

The fly ash and talc samples used in the present study were analyzed for their chemical composition following standard methods of analysis.[26] The fly ash (chemical analysis: SiO_2: 62.12%, Al_2O_3: 21.3%, Fe_2O_3: 5.55%, TiO_2: 1.38%, MgO: 1.58%, CaO: 0.53%, K_2O: 4.24% and LOI: 3.30%) and talc (chemical analysis: SiO_2: 60.35%, Al_2O_3: 1.86%, Fe_2O_3: 0.22%, MgO: 29.20%, CaO: 0.12%, K_2O: 0.80%, Na_2O: 1.2% and LOI 4.02%) were used as received, without any physical or chemical treatment. The fly ash and powdered talc sample were used wholly, without discarding any size fraction.

The alkaline phosphatic binder—sodium hexa meta phosphate (SHMP), used in the present study was of Laboratory Reagent grade (s.d. Fine-chem Ltd.).

2.2. Preparation of green tile samples and their sintering

The sintering characteristics of fly ash–talc mixtures having 0–100% talc (w/w) in the presence of 10% (w/w) sodium hexa meta phosphate have been investigated. For this purpose, the tile shaped green samples were prepared following the same procedure as followed for making green tile samples using pyrophyllite as additive to fly ash.[20] The green tile samples were dried in an air oven at 110 °C for 2–3 h and then fired in an electric muffle furnace. The heating program used for firing was as follows: heating from ambient to 950 °C at a rate of 10 °C/min → holding the temperature for 60 min at 950 °C → switching off the furnace to allow furnace cooling of the sample to ambient temperature. The temperature of firing (950 °C) was selected as the thermal analysis has shown[23] that due to dehydroxylation of talc, the formation of magnesium silicate with simultaneous reaction with SHMP is completed on heating to 950 °C.

2.3. Measurement of physical parameters of green and sintered tile samples

The physical parameters of sintered tile samples, viz., impact strength, apparent density, % linear shrinkage and percentage water absorption were determined. The procedure for measuring the impact strength of the sintered tiles was the same as followed for pyrophyllite based ceramic bodies.[27] The apparent density was evaluated by dividing accurately measured mass of the dry sintered samples by external volume (dimensional method[28]). The % linear shrinkage was evaluated using the formula:

$$\left[\frac{L_g - L_s}{L_g}\right] \times 100.$$

where L_g and L_s are the accurately measured length of green and sintered tile samples, respectively. The % water absorption of the sintered tile bodies was determined using the procedure outlined in the Indian Standards Specifications.[29] For this purpose, the sintered tile sample was dried to constant weight at 110 °C, cooled to room temperature in a desiccator and weighted accurately (W_1). The sample was immersed in distilled water with the help of a cotton twine and boiled for two hours. The heating was then removed and the tile was allowed to remain immersed in the water for 20 h, after which, the sample was taken out and excess water from the surfaces was removed by wiping with damp cloth. The sample was again weighted accurately (W_2) and the % water absorption was calculated using the formula:

$$\left[\frac{W_2 - W_1}{W_1}\right] \times 100.$$

3. Results and discussion

3.1. X-ray diffraction studies of tile compositions

The sintered tile samples made from various compositions of fly ash–talc mix were powdered using agate pestle and mortar. The X-ray diffractogram of the powdered samples was recorded with Philips make X-ray Diffraction spectrometer (Model 1710) using Ni-filtered Cu Kα radiation at 40 kV accelerating voltage and 30 mA current in 5–90° 2θ range. The identification of the phases present in the samples was made by comparing the experimentally observed inter-planar spacing (‘*d*’ values) and the intensity of the peaks with the ‘*d*’ values of the respective likely substances/phases given in the JCPDS files and search manual.[30]

3.2. X-ray diffractogram of the sample made from 90 g fly ash + 10 g SHMP

The X-ray diffractogram of the powder sample of sintered tile made from 90 g fly ash + 10 g SHMP is shown in Fig. 1A. Sillimanite (Al_2SiO_5) ‘*d*’ values (3.36, 2.21), Quartz (‘*d*’ value: 3.35) and Mullite ($Al_6Si_2O_3$) (‘*d*’ values: 5.41, 3.35), are observed to be present as major phases. The minor phases observed to be present are sodium aluminum silicate (‘*d*’ values—4.27, 2.54), tridymite form of aluminum phosphate

(T-$AlPO_4$) (‘*d*’ values: 4.39, 4.15) and hematite (‘*d*’ value: 2.70). The presence of T-$AlPO_4$ crystals[31] and mullite needles may be responsible for providing mechanical strength to the samples whereas presence of sillimanite may tend to lower the strength.

3.3. X-ray diffractogram of the sample made from 90 g talc + 10 g SHMP

The X-ray diffractogram of the sample made from 90 g talc + 10 g SHMP is shown in Fig. 1A. Magnesium silicate ($MgSiO_3$) (‘*d*’ values—3.16, 2.86) and Crystobalite (SiO_2) (‘*d*’ value—4.05) are observed to be present as major phases. The formation of these major phases is caused by dehydroxylation of talc.[32,33] Sodium magnesium phosphate (‘*d*’ values—4.27, 2.75) is observed to be present as minor phase. The formation of sodium magnesium phosphate phase may be responsible for providing mechanical strength to the sample.

3.4. X-ray diffractogram of the samples made from (fly ash + talc) mix 90% + SHMP (10%) (w/w)

X-ray diffractograms of the samples made from (fly ash + talc) mix 90% + SHMP (10%) (w/w) are shown in Fig. 1B–D. The samples show the presence of quartz, sillimanite, mullite, sodium aluminium silicate, sodium magnesium phosphate, magnesium silicate, crystoballite, hematite and T-$AlPO_4$ phases. The content of these phases in the sample however depends, as evidenced from the respective peak heights, upon the ratio of fly ash:talc in the mix. With a decrease in the content of fly ash in the mix, as expected, the quantity of quartz, sillimanite, mullite, sodium aluminum silicate, hematite and aluminum phosphate decreases whereas that of sodium magnesium phosphate and magnesium silicate increases. The formation of crystobalite is observed to take place when talc content is 60% or more and increases with talc content in the mix. It is likely that the silica released during the conversion of talc to mag-

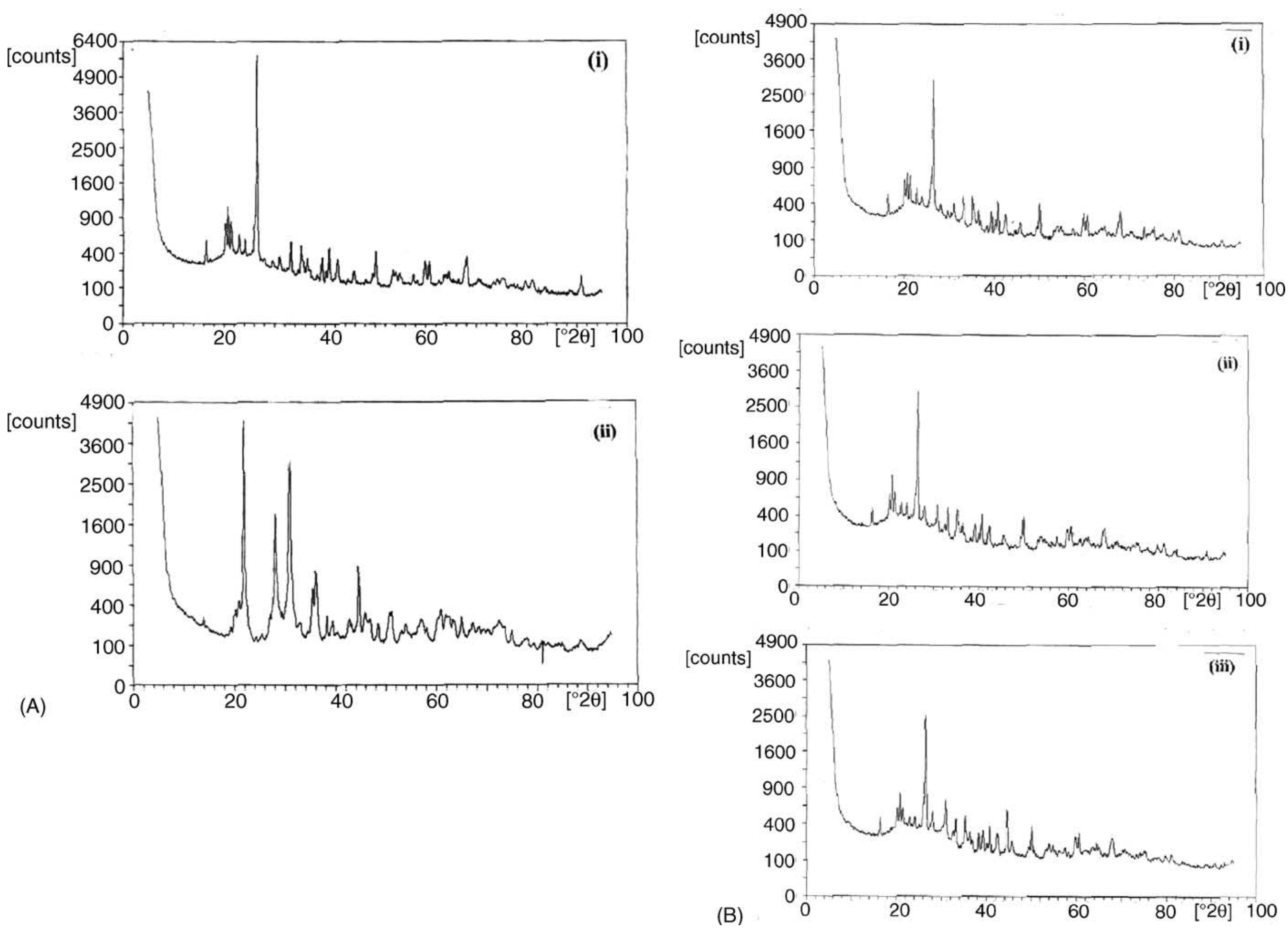

Fig. 1. X-ray diffraction pattern of powdered tile samples made from: (A) (i) 90% fly ash + 10% SHMP (w/w) and (ii) 90% talc + 10% SHMP (w/w); (B) 10% SHMP + 90% fly ash–talc mix (mix contains (i) 10% (ii) 20% and (iii) 30% talc); (C) 10% SHMP + 90% fly ash–talc mix (mix contains (i) 40% (ii) 50% and (iii) 60% talc); (D) 10% SHMP + 90% fly ash–talc mix (mix contains (i) 70% (ii) 80% and (iii) 90% talc).

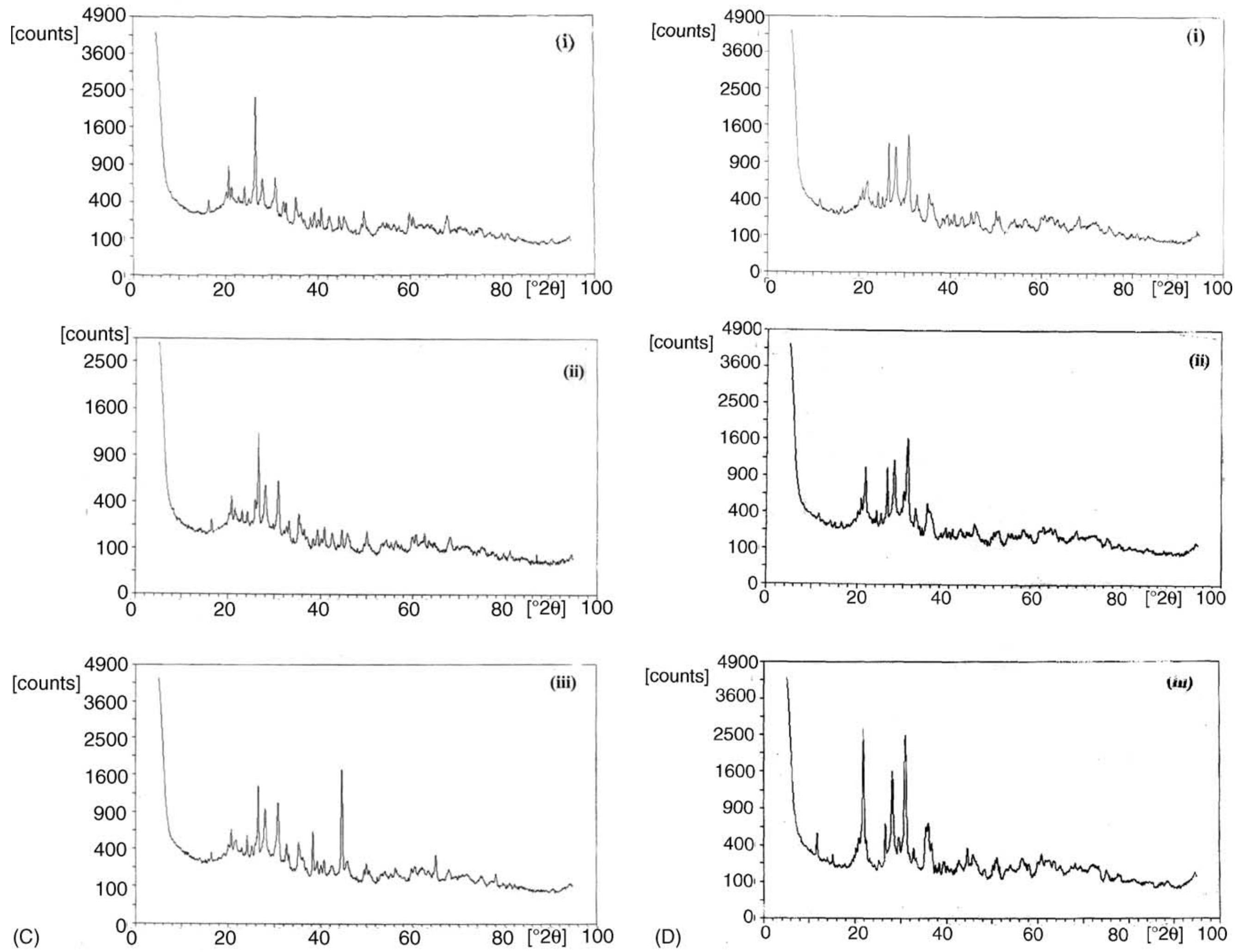

Fig. 1. (*Continued*).

nesium silicate in compositions containing 10–50% talc is utilized in forming sodium aluminum silicate in preference to quartz content of fly ash. Further, with gradual decrease in fly ash content in the mix., the availability of aluminum also decreases and hence the excess silica released by talc conversion gives rise to crystobalite phase.

3.5. Impact strength of fired ceramic tiles

The plot of impact strength versus percentage of talc in the tile samples made from fly ash–talc mix containing 0–100% talc (w/w) is shown in Fig. 2. The impact strength of the samples is observed to increase with gradual increase in the talc content in the mix. The strength in the sintered tiles made from fly ash+SHMP and talc+SHMP compositions is mainly due to the formation of T-$AlPO_4$ and sodium magnesium phosphate respectively. Further, it is notable that sample made from talc has much higher impact strength than those made from fly ash. Thus, a gradual increase in impact strength with increase in talc concentration in the mix may be attributed to increase in the sodium magnesium phosphate

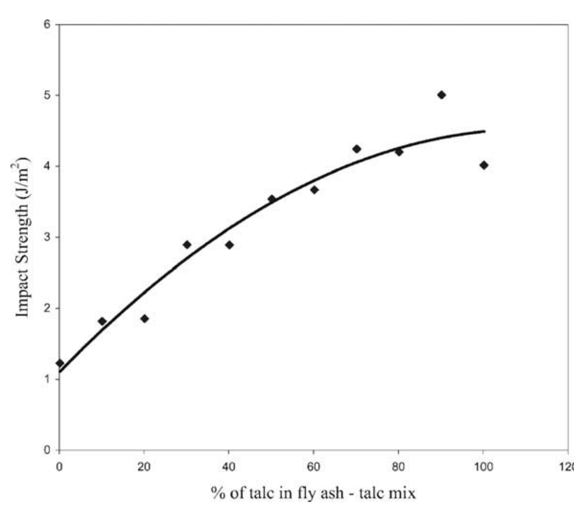

Fig. 2. Impact strength of fired ceramic tile samples prepared from fly ash (100–0% w/w) + talc (0–100% w/w) mixtures with SHMP (10% w/w).

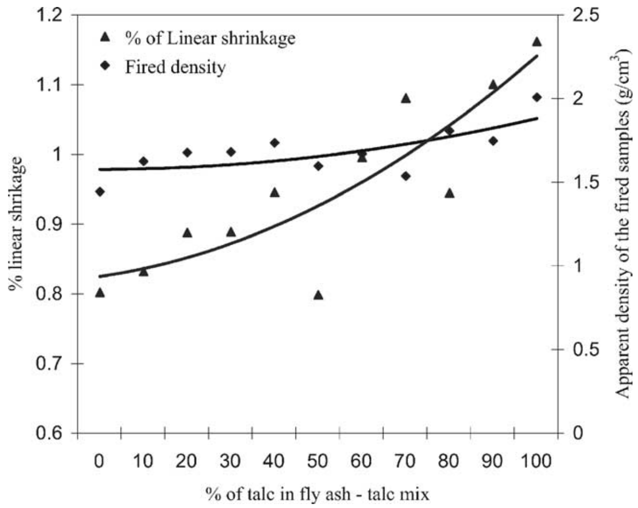

Fig. 3. Linear shrinkage and apparent density of fired ceramic tiles samples.

content (at the cost of aluminum phosphate) and decrease in sillimanite content—the facts supported by X-ray diffraction studies.

3.6. The % linear shrinkage and density of fired tiles

The effect of addition of talc in the fly ash on the % linear shrinkage and apparent density of fired tiles is shown in Fig. 3. Both, the % linear shrinkage and apparent density are observed to increase with increase in talc content in the tile. The increase in % linear shrinkage indicates enhanced densification of tiles due to increase in talc content. The increase in density with increase in talc content is attributable to higher density of talc (2.7) as compared to that of fly ash (2.07), in addition to the effect of enhanced densification.

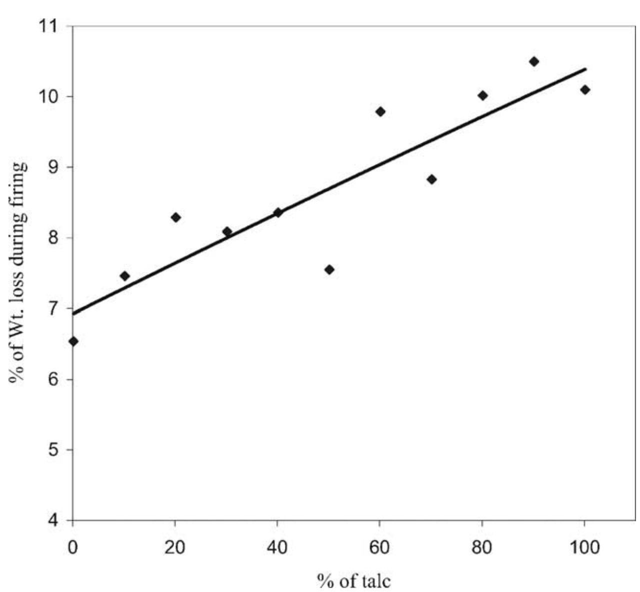

Fig. 4. Weight loss of the ceramic tiles fired at 950 °C temperatures.

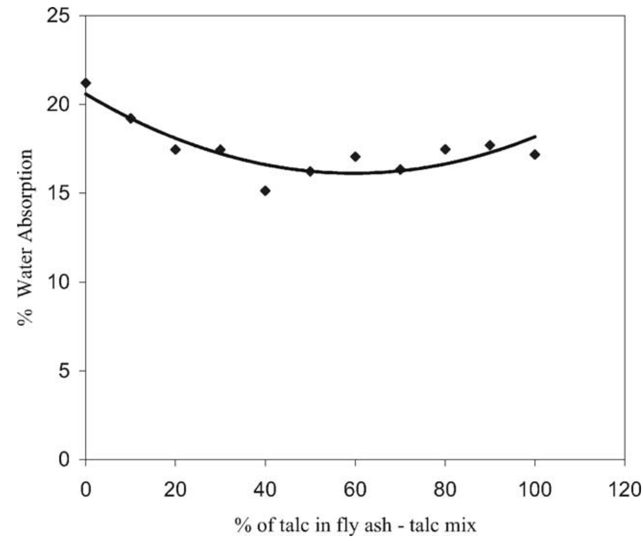

Fig. 5. Water absorption of fired ceramic tiles prepared from fly ash–talc mixtures.

3.7. Weight loss in the ceramic tiles due to sintering

With increase in the talc content in the mix, the % weight loss in the sintered tiles is observed to increase (cf. Fig. 4). The loss in weight due to sintering is caused by removal of adsorbed moisture of all raw materials and structural water of SHMP and talc. As the SHMP content is constant in all the compositions, the observed increase in the % weight loss is attributable to the increase in the quantity of water loss by removal of structural water of talc.

3.8. Effect of talc addition on % water absorption by sintered tiles

The plot of % water absorption versus percentage of talc in the mix is shown in Fig. 5. It is observed that initially, the % water absorption values decrease with increase in talc content up to about 50% and again increase thereafter. The increase in % water absorption beyond 50% talc may

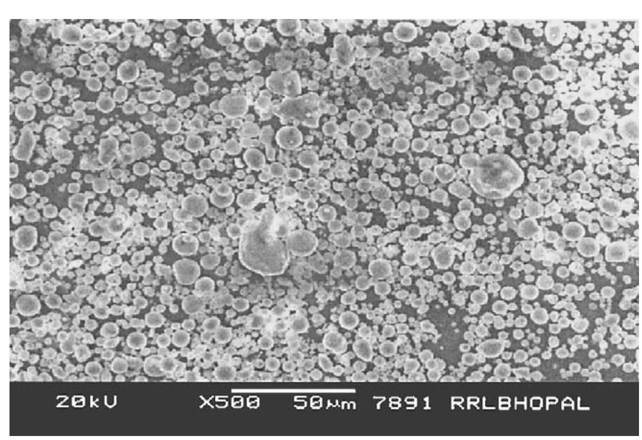

Fig. 6. SEM micrograph of fly ash sample.

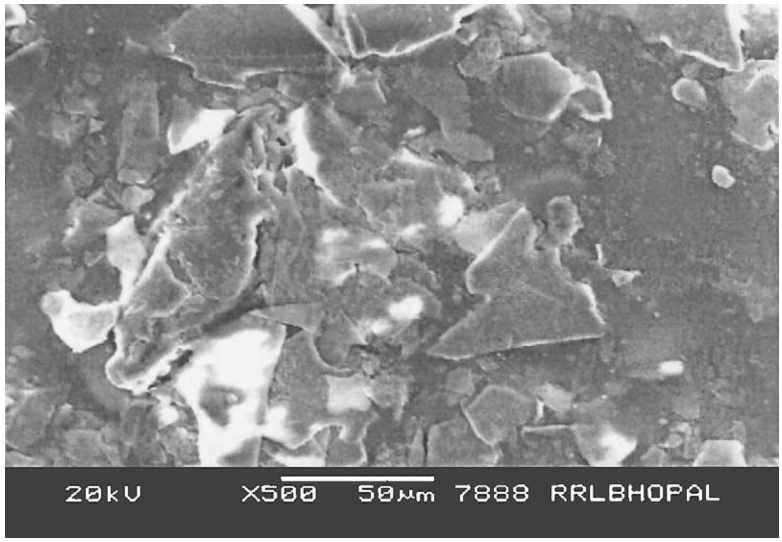

Fig. 7. SEM micrograph of talc sample.

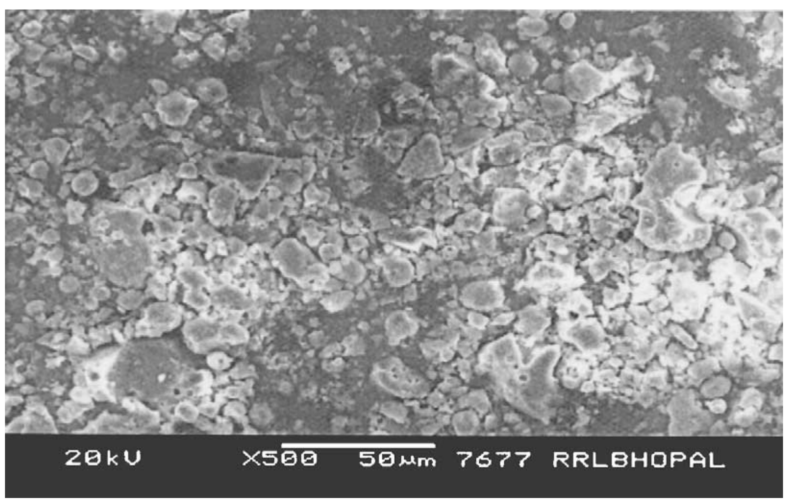

Fig. 8. SEM micrograph of powdered tile sample made by sintering of fly ash with 10% (w/w) SHMP at 950 °C.

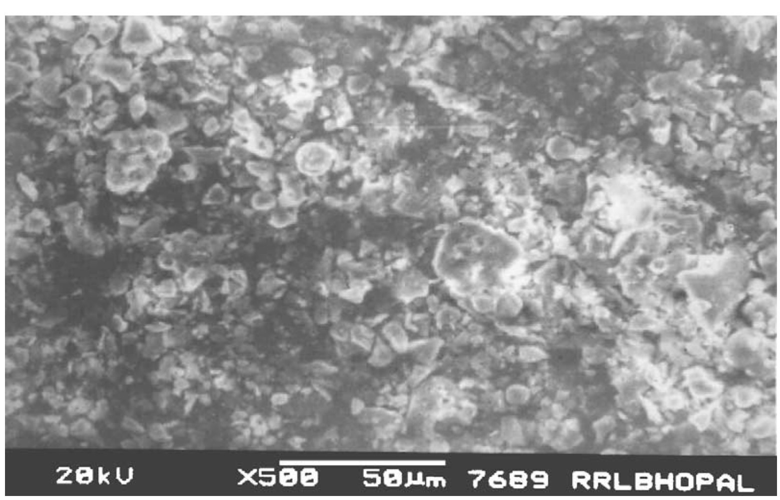

Fig. 9. SEM micrograph of powdered tile sample made by sintering of 90% mix (containing 80% fly ash + 20% talc) with 10% (w/w) SHMP at 950 °C.

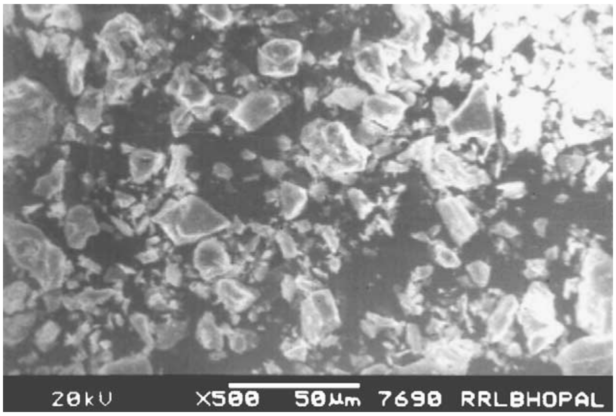

Fig. 10. SEM micrograph of powdered tile sample made by sintering of 90% mix (containing 50% fly ash + 50% talc) with 10% (w/w) SHMP at 950 °C.

possibly be due to the formation of some pores by escaping water vapours caused by dehydroxylation of talc while the initial decrease in % water absorption may be attributed to an increased densification on addition of talc, as also indicated by the trends of increase in the fired density of tiles. Since the percent water absorption is high (>15%), the tiles may be suitable as wall tiles after applying glaze.

3.9. Morphology of powdered tile samples

Scanning electron microscope (JEOL Model: JSM 5600) was used to study the morphology of the powdered tile samples made by sintering of fly ash–talc-SHMP compositions at 950 °C. For a comparison, the SEM of the fly ash and talc samples has also been included. The SEM micrographs are shown in Figs. 6–12. The fly ash particles are characterized by typical spherical shape where as the talc sample has a flaky morphology (cf. Figs. 6 and 7). On sintering of fly ash with 10% SHMP, the presence of needle like crystals of mullite phase and formation of irregular rhombohedral shaped[31] T-$AlPO_4$ crystals is clearly seen (cf. Fig. 8). In the powders of the sintered tiles made from 90% mix (80% fly ash + 20% talc) + 10% SHMP, in addition to the crystals of mullite and aluminum phosphate, the presence of tiny crystals of magnesium silicate is also observed (cf. Fig. 9). In the SEM micrograph of the powdered tile sample made from 10% SHMP (w/w) + 90% mix (containing 50% fly ash and 50% talc), the presence of bigger size crystals of an additional phase—sodium magnesium phosphate is clearly seen (cf. Fig. 10). The SEM micrographs of powder tile samples made from 10% SHMP + 90% mix (containing 10% fly ash + 90% talc) and from 10% SHMP + 90% talc show the presence of irregular, large size crystals corresponding to sodium magnesium phosphate and very small crystals (corresponding to magnesium silicate) and the crystals of T-$AlPO_4$ are practically absent (cf. Figs. 11 and 12).

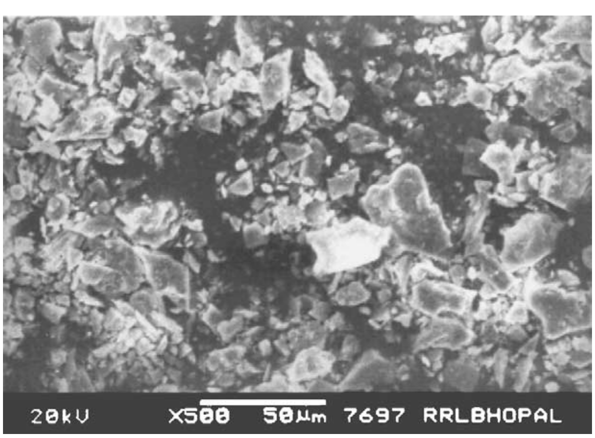

Fig. 11. SEM micrograph of powdered tile sample made by sintering of 90% mix (containing 10% fly ash + 90% talc) with 10% (w/w) SHMP at 950 °C.

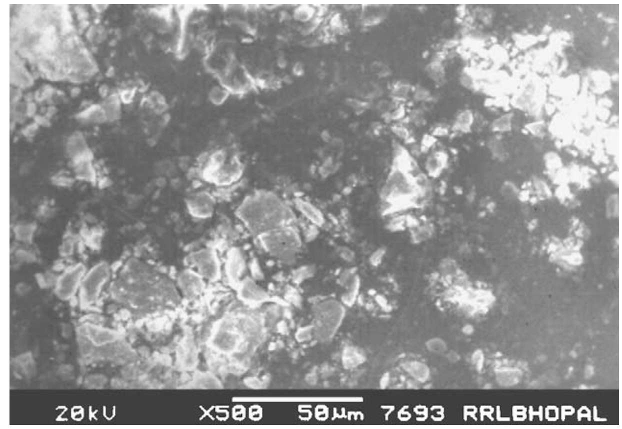

Fig. 12. SEM micrograph of powdered tile sample made by sintering of 90% talc with 10% (w/w) SHMP at 950 °C.

4. Conclusions

Based on the results of the present studies, following conclusions can be drawn:

1. The increase in the content of talc in fly ash–talc mix sintered with 10% SHMP at 950 °C leads to an improvement in the impact strength.
2. The apparent density is also observed to increase with increasing talc content whereas the water absorption passes through a minimum when the talc content is 60% (w/w) in the mix.
3. Sodium magnesium phosphate is a better reinforcing phase as compared to T-$AlPO_4$ in phosphate bonded ceramic bodies.
4. In view of higher percent water absorption, the tile samples made can be useful for wall tiles after glazing and may not be useful as floor tiles.

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