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Microstructure and thermo mechanical properties of a talc doped stoneware composition containing illitic clay

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Abstract

The influence of talc on the thermo mechanical properties and microstructure of a stoneware composition containing illitic clay has been studied. It has been observed that there is an optimum addition of talc/feldspar combination to reach proper vitrification at a relatively lower temperature. The addition of 3 mass% talc resulted in increased flexural strength (69.7 MPa at 1200 °C), decreased water absorption value (0.28%) and increased relative density (94.83%). Decrease in residual quartz content with progressive addition of talc led to decrease in percent thermal expansion upto 3 mass% talc addition, beyond which reverse trend was observed due to increased proportion of the high expansion glassy phase. Addition of talc had little effect on the mullite content of the fired body. The decrease of sintering temperature of bodies containing more than 3 mass% talc led to enlargement of pores which is responsible for decrease in the fired MOR of the matured specimens.

Keywords: B. Porosity; C. Thermal expansion; D. Mullite; D. Porcelain; Talc

1. Introduction

Talc is a mineral of hydrous magnesium silicate with the chemical formula 3 MgO, 4 SiO_2, H_2O. The use of talc in the production of ceramics is not new. This valuable raw material is used for production of different ceramic bodies [1] as well as in stain, paper, cosmetics, medical products etc. Its use in electrical porcelain body in varying amounts is well recognised. Koenig [2] used a (50:50) combination of talc–nepheline syenite combination in hotel chinaware and showed the vitrifying action of talc. Lynch and Allen [3] also produced a vitrified body at a low temperature using a talc–nepheline syenite combination. Ibrahim et al. [4] used talc in the range of 5–28% in combination with nepheline syenite and concluded that small addition of talc caused the dissolution of mullite and increased the glass content. Flux containing Mg ions lowers maturing temperature, reduces porosity and increases strength but shortens the firing range of vitreous bodies [5]. Introduction of varying amounts of talc in different ceramic bodies shows widely varying results. Sallam et al. [6] studied the influence of raw and calcined talc addition in a triaxial porcelain composition and concluded that the mode of talc addition affected the form and shape of enstatite and cordierite grains developed. Several research workers [7–10] have utilised talc in the production of fast fired tiles. Talc used in the body compositions varied from 20–90 mass%. The major observations were that talc in the bodies decreased the gas separation and improved the machanical strength of the tiles. In an earthenrware tile composition introduction of talc improved the bending strength while water absorption as well fired shrinkage values were found to decrease [11]. In a single fired vitrified floor tile composition Grosjean [12] observed that the higher the chlorite content in the talc, the more gradual was the fusion. He also observed that feldspar- talc eutectic helped to improve vitification while exhibiting higher warpage values. Grosjen [13] while studying the influence of talc on kaolino-illitic clay observed that influence became apparent at 1100 °C and there was a decrease in the maturing temperature. A large and widely varying effects can be achieved by using talc, but often only within carefully defined limits of composition and firing temperature.

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The demand to save energy and use of suitable low cost fluxes is steadily increasing because of rapidly increasing costs of personnel, raw materials and particularly fuel. The technological innovation to reduce the firing time and thereby reducing the fuel consumption is steadily gaining importance. An attempt towards reduction in the maturing temperature of ceramic bodies by compositional reorientation and/or by incorporation of non conventional raw materials deserves due attention. In addition to that, technologists are giving more attention to the use of common clays due to abundance of such alumino-silicate mineral as well as its ready availability.

With this in mind the study was undertaken to utilise one red burning illitic clay in a clay–quartz–feldspar triaxial system. The effect of talc on the composition was investigated to find out the optimum addition as well as an attempt has been made to give a possible explanation of the results obtained.

2. Experimental procedure

The raw materials selected for the investigation were a common clay (Illitic in nature) from West Bengal (India), plastic kaolinitic clay, sand, potash feldspar and talc. The physico-chemical characteristics of these clays have been discussed elsewhere [14]. Chemical and mineralogical analysis of the raw materials were carried out according to standard procedure and reported in Tables 3 and 4. The common clay was subjected to simple beneficiation which comprised of dispersion in water and passing through 200 mesh sieve for removal of gritty materials as well as some portion of free silica associated with the raw clay.

All the body mixes (T_0–T_6) were formulated with 65 mass% plastic component and 35 mass% non plastic component. Progressive incorporation of talc was made as an auxilliary addition reaching a maximum value of 6 mass% (Table 1).

Various body ingredients were separately weighed and wet ground in a pot mill using porcelain pebbles as the grinding media. The grinding operation was continued upto the required fineness (0.5% residue over 200 mesh sieve) and the slurry was made into a casting slip with requisite doses of deflocculants (a mixture of soda ash and soda silicate) and left for ageing. Rectangular bars of 140 mm length and 12 mm×12 mm cross-section were made by slip casting method. The cast bars were demarcated at 100 mm intervals and then allowed to dry. The dried bars were tested for determination of properties in the unfired stage viz. linear shrinkage on drying and MOR according to the standard procedure. The dried bars were then fired in an electric furnace at seven different test temperatures between 1050 °C and 1250 °C at an average heating rate of 2.5 K/min with 2 h soaking time at the respective peak temperatures. Samples were furnace cooled for further experimentation.

Table 1
Composition of body mixes (mass%)

	T_0	T_2	T_3	T_4	T_6
Beneficiated common clay (illitic in nature)	45	45	45	45	45
Plastic kaolinitic clay	20	20	20	20	20
Sand powder (−200 mesh)	25	25	25	25	25
Feldspar powder (−200 mesh)	10	10	10	10	10
Talc	0	2	3	4	6

Table 2
Some important physical properties of the casting slip and green body

	T_0	T_2	T_3	T_4	T_6
Water of plasticity (%)	24.82	24.80	24.87	24.71	24.96
Electrolyte concentration used					
•Soda ash	0.08	0.08	0.08	0.08	0.08
•Sodium silicate	0.25	0.25	0.25	0.25	0.25
Density of the slip (gm/cm^3)	1.49	1.50	1.52	1.50	1.50
Rate of casting (mm/h)	10.0	10.4	10.6	10.6	10.6
Linear shrinkage on drying (%)	3.82	3.82	3.86	3.86	3.86
Green MOR (kg/cm^2)	22.8	22.6	22.0	22.0	22.0

Table 3
Particle size distribution of a typical body mix (T_3)

Particles	Cumulative percent
Finer than 25 μ	96.5
Finer than 20 μ	91.5
Finer than 15 μ	82.5
Finer than 10 μ	69.0
Finer than 8 μ	62.0
Finer than 5 μ	48.5
Finer than 3 μ	38.5
Finer than 2 μ	32.0
Finer than 1 μ	25.0

Average particle size = 5.2±0.2 μ of different body mixes.

Table 4
The chemical analysis of raw maerials (mass%)

Oxide constituents	Beneficiated common clay	Plastic kaolinitic clay	Sand	Feldspar	Talc
SiO_2	65.75	62.46	94.92	66.81	62.06
TiO_2	1.23	0.84	0.21	Tr.	0.10
Al_2O_3	16.94	24.88	2.22	18.08	0.84
Fe_2O_3	2.28	1.34	0.61	0.24	0.72
CaO	1.43	0.44	1.06	1.03	1.36
MgO	0.39	0.17	Tr.	0.23	30.45
Na_2O	0.50	0.46	0.18	1.69	0.07
K_2O	2.08	0.89	0.21	10.94	0.46
LOI	8.97	8.52	0.44	0.58	4.01

3. Measurement

The particle size distribution of the ground body mixes was measured using a sedigraph apparatus, model 5000 D of Micromeritics Inc. USA.

Thermo-mechanical properties of the fired samples viz. linear shrinkage, MOR, water absorption, apparent porosity and bulk density were evaluated in accordance with the standard procedure. The bending strength of the fired specimens was measured with an electro-mechanical universal tester (Instron 1195) with three point bending fixture. The cross head speed was 1 mm/min and a span of 100 mm was maintained throughout the experiment.

Linear thermal expansion of the matured specimens was determined using an Orton automatic dilatometer, Bosch & Lamb, UK, at a heating rate of 2 K/min.

Major crystalline phases present in the matured specimens were identified by X-ray diffraction (XRD) using a Philips PW-1730 X-ray diffractometer. Microstructure was studied by SEM analysis on selected sintered samples using a LEO S430I apparatus.

4. Results and discussion

The plastic and dry properties of different mixes are presented in Table 2. The results revealed that progressive incorporation of talc in different body mixes had very insignificant effect so far as water of plasticity is concerned. No significant changes were noticed in the dry linear shrinkage on drying and green MOR of the mixes with the talc addition. Shrinkage values varied marginally from 3.82% (body mix T_0) to 3.86% (body mix T_6) while MOR values decreased from 22.8 kg/cm^2 (T_0) to 22.0 kg/cm^2 (T_6).

The particle size distribution of different body mixes were almost identical, being in the range of 5.2±0.2 μ

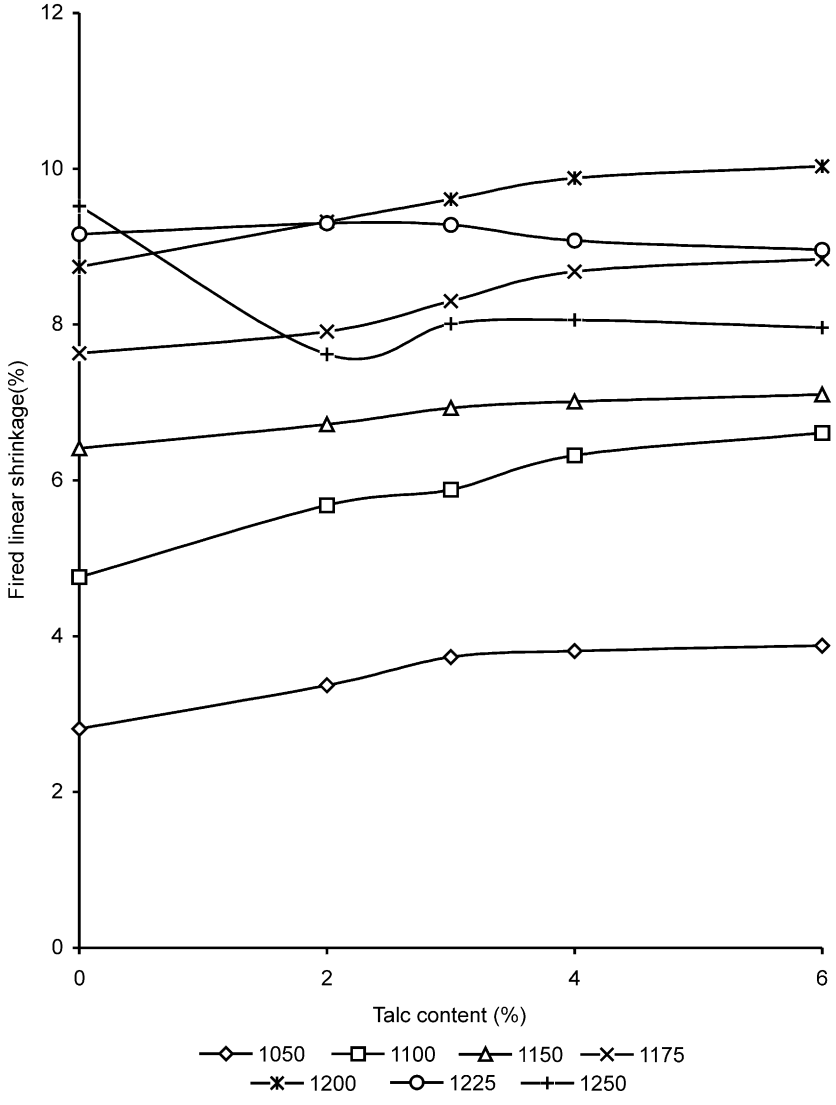

Fig. 1. Effect of talc addition on fired shrinkage.

(average particle size). The result is very much expected as there were only marginal variations in the body compositions. Result of particle size distribution of body mix T_3 has been presented in Table 3. The result indicated that particles finer than 2 μm and 10 μm were 32.0 and 69.0 mass% respectively while particles coarser than 20 μm was 8.5 mass% only.

The chemical analyses of the raw materials are presented in Table 4. The results showed that the common clay was highly siliceous and that SiO_2 content was 65.75 mass%. Al_2O_3 content was rather low (16.94%), even after beneficiation. The common clay was kaolinitic-illitic in nature [27]. The presence of considerable amounts of impurities in the common clay (illitic in nature) is likely to affect adversely on the vitrification range of this clay. Appreciable amount of ferruginous impurities (Fe_2O_3 and TiO_2) were also present (3.51%). The plastic kaolinitic clay was also high in silica (62.46%) and low in Al_2O_3 (24.88%). Total alkali ($Na_2O + K_2O$) content in the two clay samples were 2.58 and 1.35 mass% respectively. Sample of sand contained 94.92 mass% SiO_2 as the major constituent. Talc contained approximately 62 mass% SiO_2 and 30.4 mass% MgO as major constituents.

The properties of the fired specimens fired at different test temperatures are presented in Figs. 1–7. At any temperature upto 1200 °C, percent fired shrinkage values increased gradually as talc was progressively increased in the composition upto 6%. Beyond this temperature, a decrease in the shrinkage values was observed irrespective of the amount of talc present in the compositions excepting composition T_o (containing no talc) which showed further increase in shrinkage (Fig. 1). This indicated that talc bearing compositions possibly got overfired beyond 1200 °C. Another important observation was that the shrinkage values increased rather sharply with progressive increase in talc addition (up to 3 mass%) beyond which the effect was not so significant.

The influence of talc in the composition on fired MOR and water absorption have been presented in

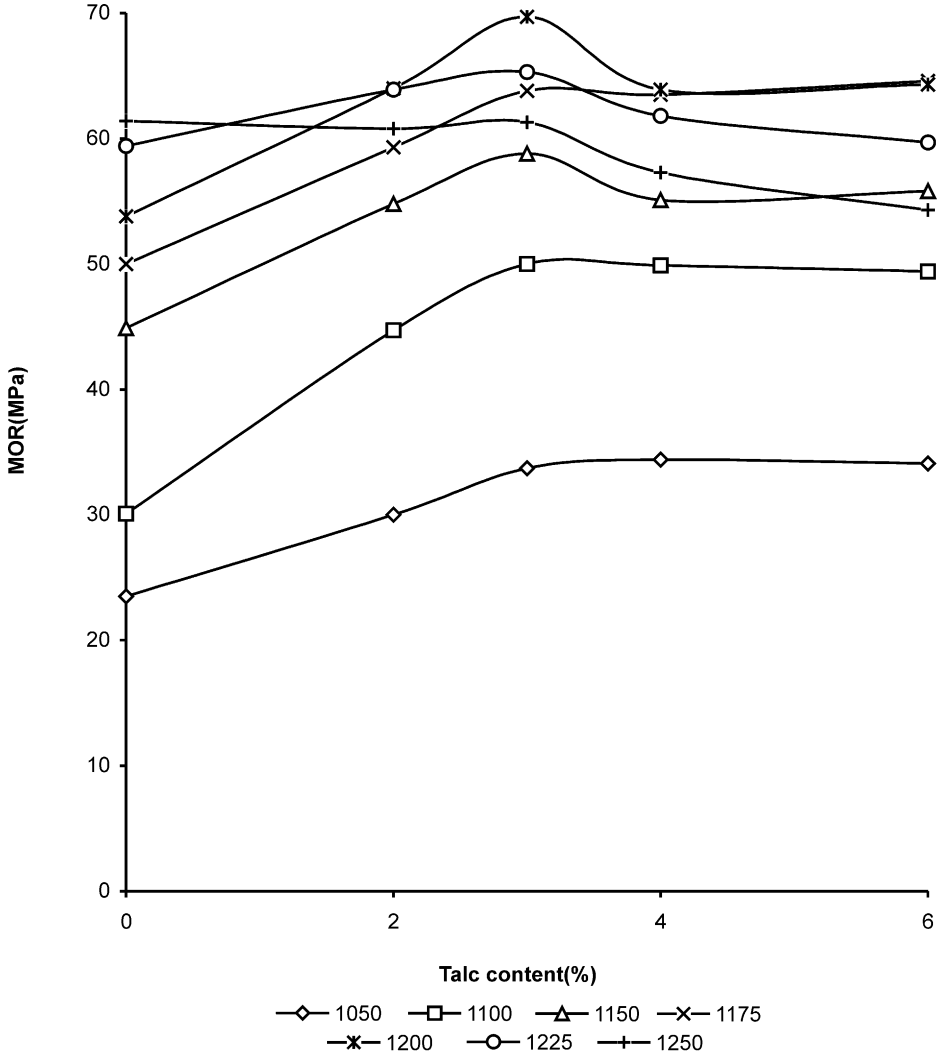

Fig. 2. Effect of talc addition on MOR.

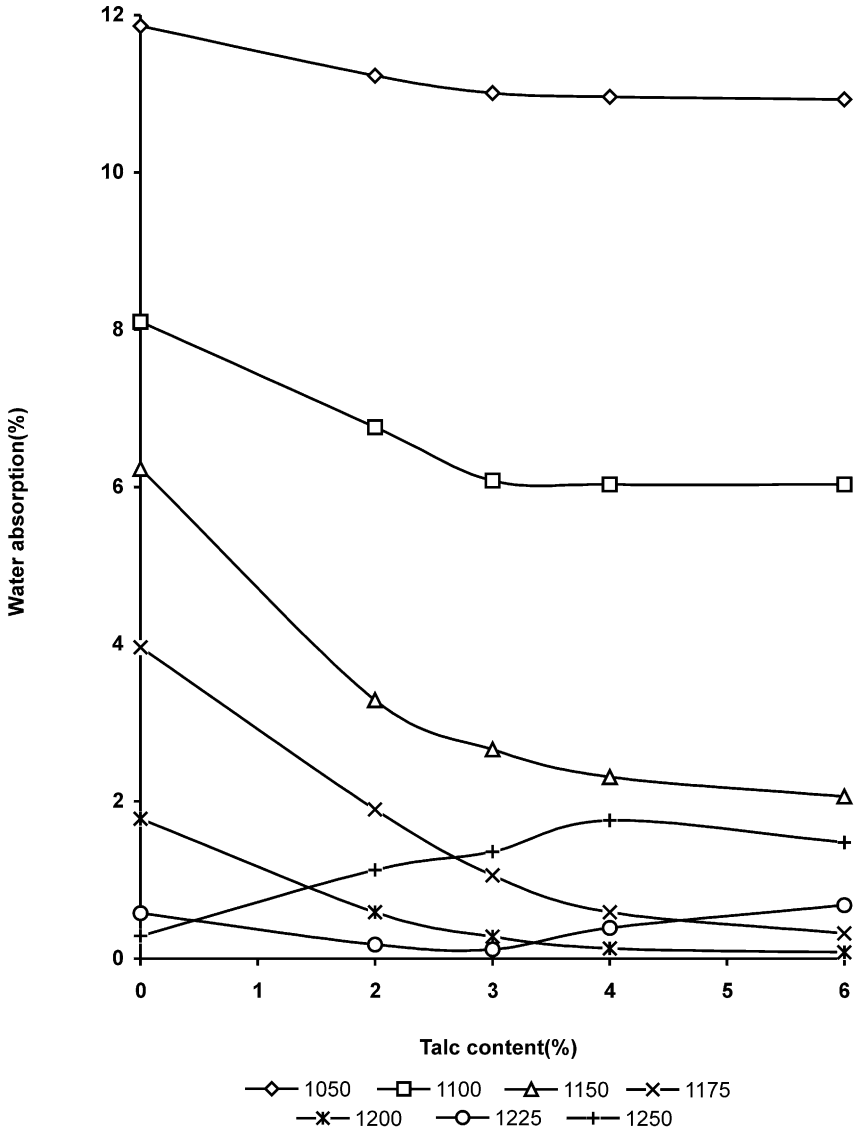

Fig. 3. Effect of talc addition on water absorption.

Figs. 2 and 3. The effect of talc was less prominent below 1100 °C and became apparent from 1100 °C and onwards which supports the observations of Grosjean [13]. It is evident from the results that with progessive incorporation of talc up to 6 mass% there was a beneficial effect on the flexural strength of the specimens irrespective of the temperature of firing up to 1200 °C. However, the MOR values increased very sharply and reached a maximum with 3 mass% talc addition, beyond which a decrease in the MOR values was observed. The flexural strength of vitrified bodies reached a maximum value of 69.7 MPa with 3 mass% addition of talc. Water absorption values gradually decreased with progressive incorporation of talc and reached minimum values at 1200 °C and beyond this temperature reverse trend was observed possibly due to overfiring of the specimens at 1250 °C excepting composition T_o with no talc addition. It is therefore, evident that in combination with feldspar, even 3 mass% talc is sufficient to effect vitrification (water absorption value below 0.5%) at lower temperature (at least by 50 °C) while improving the fired strength. This supports the observations of both Sallam et al. [6] and Prasad et al. [15].

The mechanical strength of a conventional ceramic body made of crystalline aggregate embedded in a glassy matrix is greatly influenced by the stresses developed in the glassy phase as well as closed pores, shape, size and distribution of residual quartz grains and mullite crystals. Multiphase ceramics with phases having different coefficient of expansion tend to develop griffiths flaws resulting in the lowering of mechanical properties. It has been reported [16–18] that increased strength in clay–quartz–feldspar triaxial compositions was due to increased mullite content and its morphology.

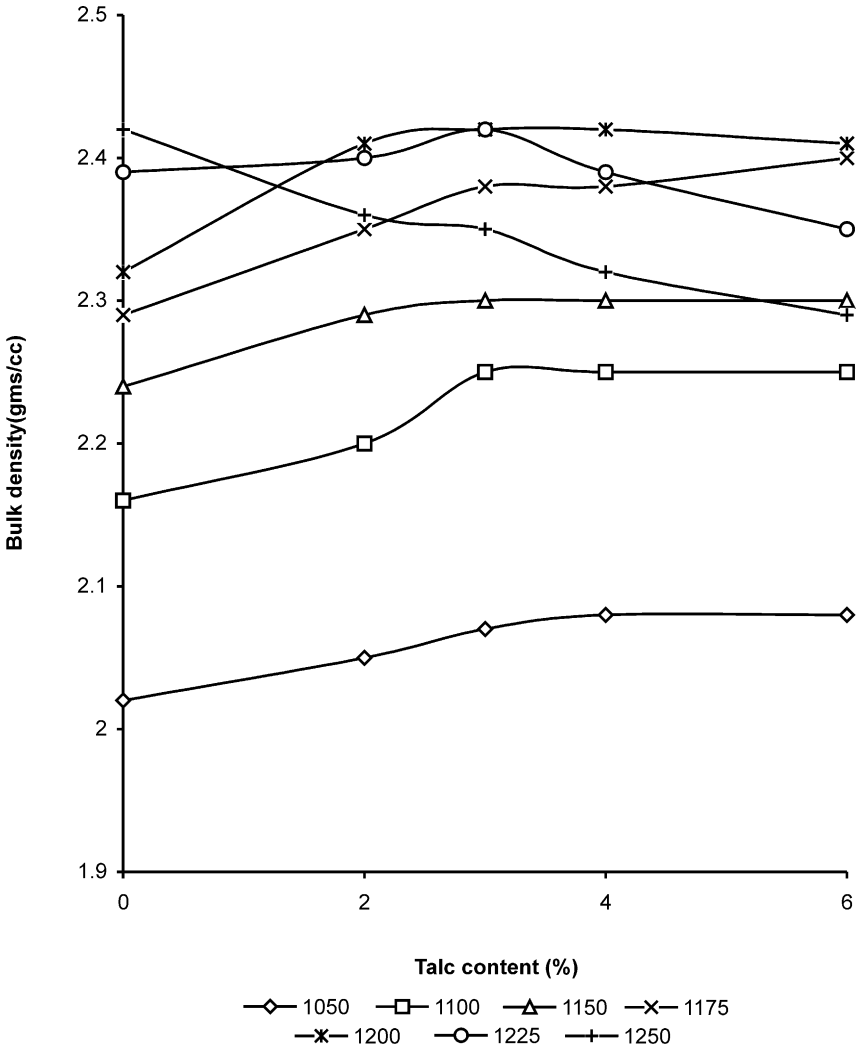

Fig. 4. Effect of talc addition on bulk density.

Fig. 8 shows the powder X-ray diffraction pattern of the matured samples. Two major phases were observed in all the specimens, namely mullite and quartz. One interesting observation is that with gradual increment of talc, there is a progressive decrease in the peak heights (lines at 4.367 and 3.423 Å) for quartz showing a significant reduction in the content of free quartz in the fired specimens. However, the heights of the peaks for mullite (lines at 2.221, 2.543 and 5.367 Å) remained almost identical to that of the reference body (T_0). It may, therefore, be inferred that with progressive addition of talc in the composition, the content of free or residual quartz is substantially decreased. However, no evidence of increment of mullite was observed in contrary to the findings of Sane and Cook [19] and Tkalcec and Falz et al. [20]. In the present investigation, total Al_2O_3 content in the body mixes was rather on the lower side due to the use of high silica kaolinitic clay and kaolinitic–illitic clay as against high alumina porcelain body by other research workers [19, 20]. This might be the cause of the observed result that mullite content was not affected by talc addition.

Presence of talc in the composition led to early glass formation. By dissolving talc, the glassy phase becomes richer in Mg^{+2}, Ca^{+2} and Fe^{+3} ions and attacked the quartz grains and dissolved them more intensively. The melt was thus enriched with silica, and it became more viscous and crystallisation of mullite is inhibited [21, 22]. However, decrease in the relative proportion of residual quartz grains would decrease stresses around the glass crystal interface caused by the difference in the thermal expansion coefficients of quartz and the glassy phase. This will result in the reduction in the number of griffith flaws in the matrix thereby causing a favourable effect on the body's mechanical strength [23]. Thus maximum value of flexural strength (69.7 MPa) was obtained with 3 mass% talc addition. Talc contributes greatly to the increased formation of a glassy matrix. Brittleness of the glassy phase and its poor fracture resistance contributed to the decrease in mechanical strength [24] with higher talc addition (beyond 3%).

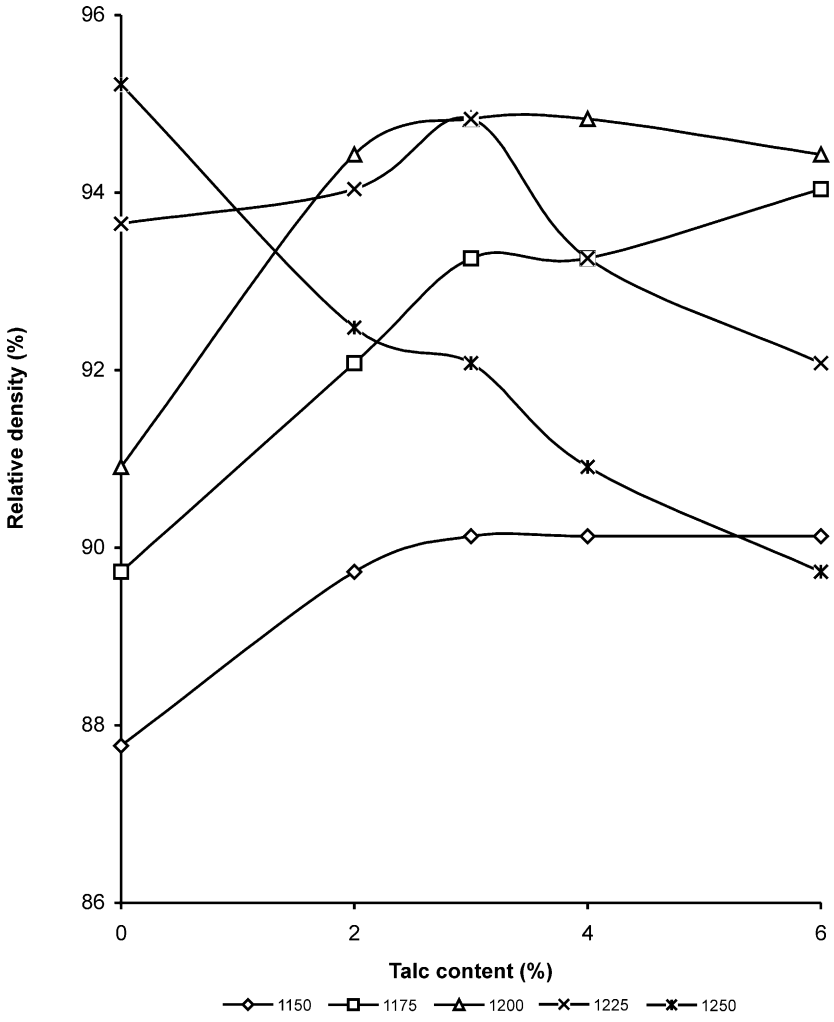

Fig. 5. Effect of talc addition on relative density.

When the talc concentration increases beyond 3 mass%, more residual quartz goes into solution due to enhanced fluxy action of MgO in combination with feldspar. This results in the formation of more liquid of lower viscosity, which, in turn, favours early vitrification of the fired specimens. The increase in the proportion of liquid phase is expected to fill the interparticle body pores. This possibly resulted in specimens having fewer stress concentrators [25] thus increasing the possibility of crack propagation through the matrix [23] and causing deterioration in the mechanical properties.

The progressive increase of bulk density with progressive increase in talc content has been presented in Fig. 4. However, the bulk density of the specimens containing 3 mass% talc attained the highest values at almost all the test temperatures and little effect was observed when percentage of talc was further increased beyond 3%. Excepting composition T_0 (0% talc addition) which attained maximum bulk density at 1250 °C, all the other compositions attained maximum bulk density at 1200 °C and compositions T_3 (3 mass% talc) and T_4 (4 mass% talc) showed the highest value 2.42 g/cc. Beyond 1200 °C all these specimens showed marked decrease in the bulk density values indicating overfiring. Fig. 5 shows the relative densities of the compositions fired. Talc bodies achieved 90% relative density even at 1150 °C and around 95% at 1200 °C. MgO in talc thus effected early densification of the body mixes, increasing the mobility of atoms at grain boundaries and therefore, facilitating the movement of pores out of the material. This supports the observation of Schroeder [16]. Generally after the apparent porosity reaches zero, the closed porosity tends to increase with the relative density abruptly decreasing. Since the specific gravity (G) and the mass of the fired body remain virtually constant in the range from vitrification to overfiring stage or bloating, the solid volume will also remain constant in that range. For such conditions, the change in closed porosity (Pc) directly affects the estimated bulk density (D) according to the relation.

$$\mathrm{Pc} = 100\ (1 - D/G)$$

Fig. 6 shows the variation in closed perosity with progressive increment of talc content. values of calculated

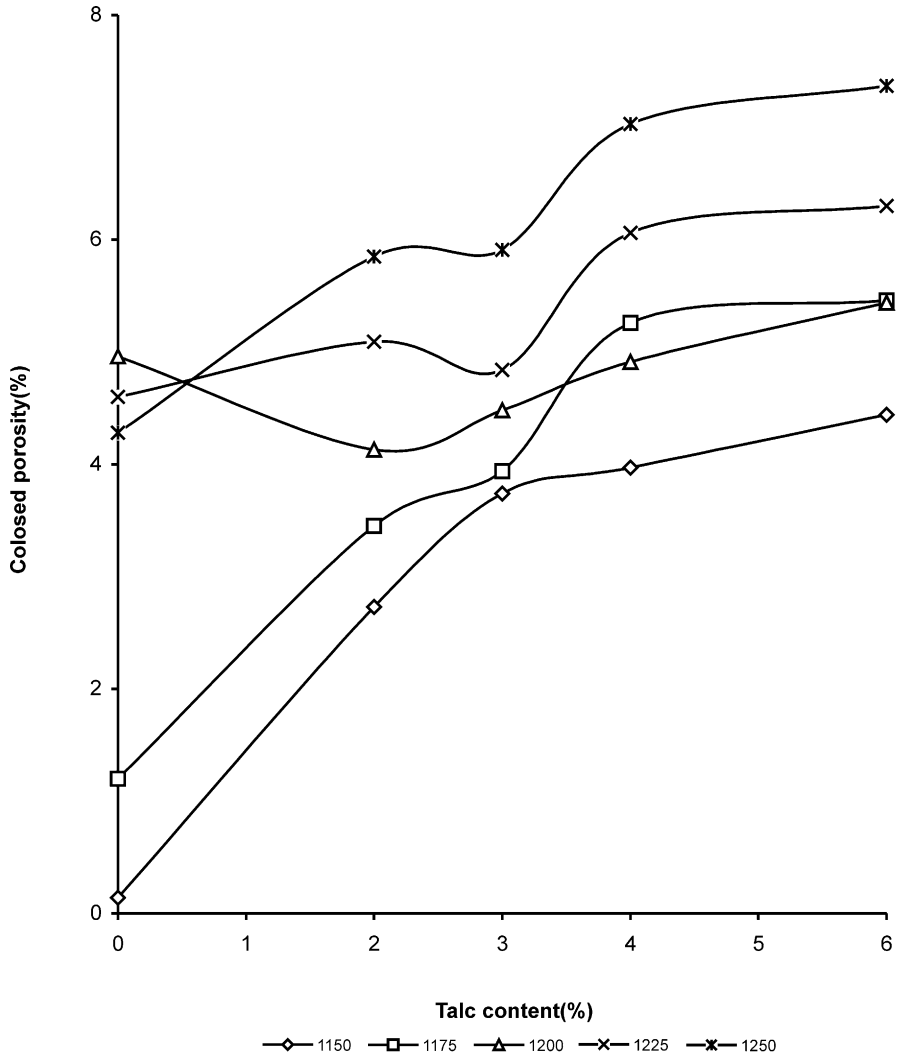

Fig. 6. Effect of talc addition on closed porosity.

closed porosity for the vitrified samples ranged between 4.48 and 6.06%. One interesting observation is that with progressive incorporation of talc in the compositions, there was a gradual decrease in apparent porosity values in the vitrified specimens (at 1200° and 1225 °C), while an opposite trend was observed so far as closed porosity was concerned. In the present investigation the flexural strength was found to be proportional to relative density and inversely proportional to closed porosity. Generally, pores must be eliminated as the bending strength of ceramics decreases exponentially with the increase in porosity. Norris et al. [26] reported that, for siliceous percelains, a maximum bending strength developed when the apparent porosity decreased to zero and it was 20–30 °C below the temperature at which maximum relative density could be achieved. However, in the present investigation, maximum flexural strength as well as maximum relative density were obtained at an apparent porosity value of 0.69% (composition T_3 at 1200 °C) and both the values decreased with further decrease in apparent porosity values. The relative increment in the closed porosity values may be correlated to this observation. Tkalcec et al. [20] observed that in an aluminous porcelain body, addition of more than 1 wt.% talc leads to a decrease of strength which can be attributed to the appearence of greater quantities of large pores. This may be correlated with the observations made in the present investigation that flexural strength decreased with addition of more than 3 mass% talc which resulted in attaining vitrification of the specimens before completion of reactions among different constituents. This confirms the observations of Tkalcec et al. [20] and Gamlem and Lyng [30] who observed that talc added to ball clay led to densification of body before the gaseous phase was completely released from raw materials.

The results of thermal expansion values of matured specimens (at 1200 °C) are presented in Fig. 7. Significant decrease in the percent thermal expansion values of

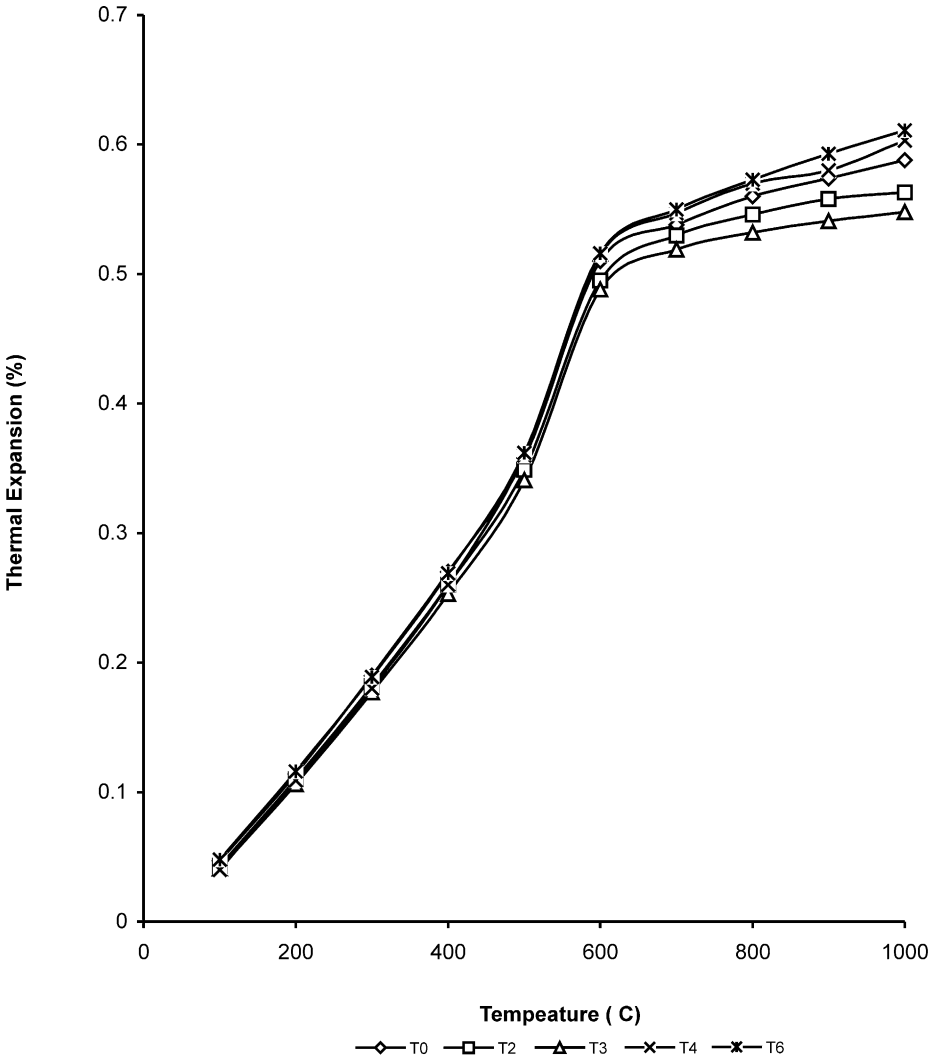

Fig. 7. Effect of talc on thermal expansion with temperature.

matured specimens were observed on progressive increase in the addition of talc at different temperatures up to 1000 °C. Minimum thermal expension value was observed with composition T_3 containing 3 mass% talc and the values showed the reverse trend with further addition of talc up to 6 mass%. This may be due to the progressive lowering of free quartz, higher thermal expansion contributing component, in the specimens (confirmed by XRD analysis) with gradual increase of talc in the compositions up to 3 mass%. However, talc contributes greatly to the increased formation of a glassy matrix. At higher temperature, the impurities present in the illitic clay particularly Fe_2O_3, CaO, Na_2O and K_2O would get dissolved in the glassy matrix resulting in the lowering of the viscosity and increasing the thermal expansion. This phenomenon possibly weighed out the positive effect of reduced quartz factor with compositions having talc content beyond 3 mass% and an overall increase in percent thermal expansion values were obtained.

Scanning electron micrographs of polished and etched section of samples T_0, T_3 and T_6 are shown in Figs. 9–12. The micrograph (Fig. 9) shows unreacted quartz grains which are possibly responsible for early failure of the referred body (T_0) due to mechanical stress [28,29]. There was a gradual reduction in the content of residual quartz in the matured specimens (Figs. 10 and 11) with progressive addition of talc. Secondary mullite needles in the glassy matrix are clearly visible. In sample (T_3) mullite crystals were rather smaller in size (Fig. 10) than those in sample T_6 (Figs. 11 and 12). Talc lower the viscosity and surface tension of the glassy phase. The glassy matrix with relatively lower viscosity in composition T_6 possibly helped the growth of secondary mullite in this specimen. In both the cases they were found to be randomly oriented. Enlargement of pores as well as increase in its quantity with the increase of talc addition is clearly visible which affected adversely on the mechanical properties of the specimens with talc addition beyond 3%.

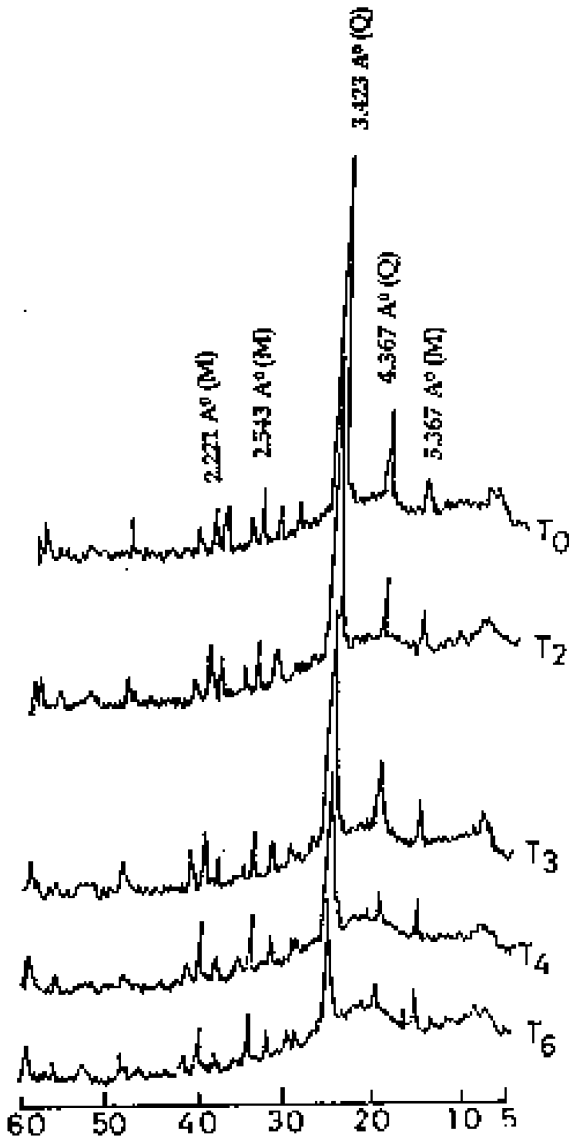

Fig. 8. XRD curves of fired and matured specimens containing different proportions of talc.

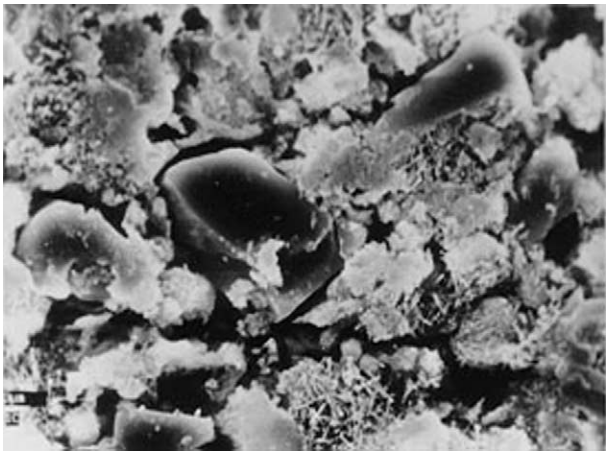

Fig. 9. Microphotograph of SEM of matured specimen of T_o.

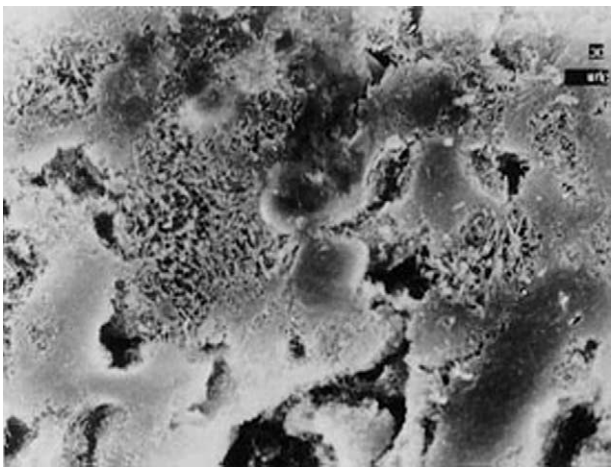

Fig. 10. Microphotograph of SEM of matured specimen of T_3.

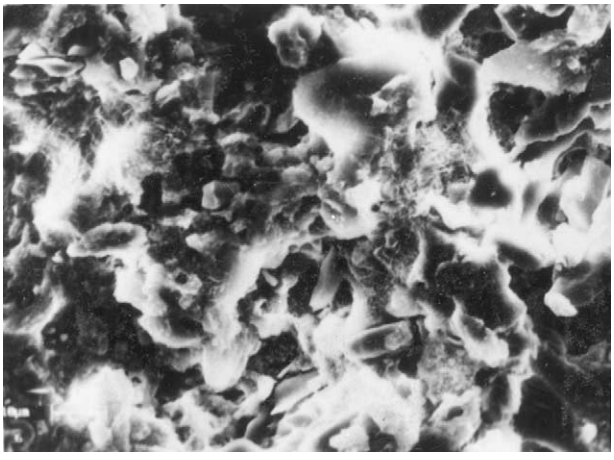

Fig. 11. Microphotograph of SEM of matured specimen of T_6.

Fig. 12. Microphotograph of SEM of matured specimen of T_6.

5. Conclusion

The study shows that

(i) There is an optimum addition of talc/feldspar combination to reach proper densification at a relatively lower temperature (at least 50 °C reduction in vitification temperature) in the system studied.
(ii) Talc added in quantities upto 3 mass% increases the body strength significantly, decreases the water absorption value, increases the relative density and decreases the percent thermal expansion. But in higher quantities it produces the opposite effect.
(iii) X-ray analysis indicated that no significant effect on mullitisation was observed as a result of talc adition. However, a gradual decrease in the residual quartz content was observed with progressive increase in talc addition.
(iv) Decrease in residual quartz content led to decrease in percent thermal expansion up to 3 mass% addition of talc, beyond which reverse trend was observed possibly due to the increased proportion of the high expansion glassy phase.
(v) The decrease of sintering temperature of bodies containing more than 3 mass% talc led to increased closed porosity as well as enlargement of pores which is possibly responsible for decrease in the mechanical strength of the body.

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