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Effect of pyrophyllite incorporation in porcelain composition on mechanical properties and microstructure

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Abstract

China clay (Kaolin) and quartz in the ratio of 1:2 was progressively replaced by pyrophyllite in a conventional porcelain mix. Addition of 15% pyrophyllite as a replacement of a combination of china clay and quartz decreased the linear shrinkage by 5.03% while fired strength was improved by about 31.5% compared to that of the conventional body fired at 1300 °C. Incorporation of pyrophyllite beyond 15% resulted in early vitrification of porcelain composition, although in lower proportion the effect is not so significant. Percentage of mullite was found to increase in the fired specimens even when kaolinite was progressively replaced by pyrophyllite. However, beyond 22.5% pyrophyllite addition, there occurred large volume of glass formation. Presence of large volume of glassy phase as well as formation of large pores of various shapes resulted in deterioration in ceramic properties. The size and shape of mullite crystals is to a large extent controlled by the fluidity of the liquid matrix from which they grow and this is again a function of temperature and composition.

Keywords: Mullite; Porcelain; Pyrophyllite

1. Introduction

Triaxial porcelain bodies have been studied extensively for the past 50 years. Because of the complexities of the porcelain system, there still remain significant opportunities for research and study, particularly in the areas of raw material understanding, processing science, and phase and microstructure evolution [1]. The compositions of porcelain can contain not only conventional but also non-conventional raw materials [2,3]. Incorporation of non-conventional raw materials viz. wollastonite, talc, pyrophyllite etc. in the batch formulations is becoming popular in order to accommodate faster firing technique as well as to reduce the maturing temperature [4,5].

The firing of porcelain promotes physico-chemical reactions which are responsible for attaining the ultimate properties of the ceramic products. In this process it must be considered the kinetic limitations, development of the phases and complexities of the microstructure. This is also to be kept in mind that selection of raw materials and their proportions, processing parameters particularly firing atmosphere, maximum temperature attained and the soaking time have strong influence in the development of microstructure which in turn influence the ultimate thermo-mechanical properties of the products. Chaudhury [6] observed that thermal shock resistance of hard porcelain deteriorated with the increasing amount of quartz and glassy matrix while the same improved with increasing mullite content. Khandelwal and Cook [7] achieved 200% increase in transverse strength by adding 40% alumina to a vitreous china body. Increasing alumina and decreasing feldspar content increased the total crystallite content of the fired body. Development of higher proportion of mullite crystals and their intense interlocking was thought to increase the strength. On the other hand the same authors compared the strength of two quartz bodies with different mullite contents and opined that it was not the mullite content but the microstructure (the quantity, size, size distribution and shape of various constituent phases) contributed significantly in the development of porcelain properties. Carty and Senapati [1] also concluded that ultimate microstructure and intrinsic flaws are the principle factors which influence the ceramic properties of whitewares. Schüller [8] observed that a variation in strength occurred with a variation in quartz content and highlighted that the best

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dimension of quartz is between 15 and 30 μm. Ece and Nakagawa [9] explained that fractures initiating from flaws, were micro-cracks around quartz grains acting as links between closed pores which reduce the mechanical strength of porcelain. Braganca and Bergmann [10] opined that influence of the phases on mechanical strength of porcelain is limited with regard to firing temperature. They further opined that the difference in thermal expansion between quartz and matrix was responsible for creation of flaws which limited the porcelain strength. In our earlier communication [11] quartz was progressively replaced by pyrophyllite in a conventional porcelain mix. It was observed that up to 15% pyrophyllite incorporation there was improvement in thermo-mechanical properties. Presence of mullite in higher proportion with this composition was found to be responsible for higher mechanical strength. In another communication [12] incorporation of pyrophyllite as a progressive replacement of china clay (Kaolin) in a porcelain composition resulted in improved flexural strength as compared to that of the conventional body. Percentage of mullite was found to increase in the mixes while kaolinite was progressively replaced by pyrophyllite. However maximum flexural strength and mullite content were observed at 1300 °C with mix having 5% pyrophyllite content.

In this paper we have tried to incorporate pyrophyllite as a replacement of a combination of china clay and quartz in the ratio of 1:2. Effect of such substitution on shrinkage, bulk density, porosity, strength and phase evolution in relation to firing temperature was studied and compared with conventional whiteware mix. Some selected samples were examined for microstructural analysis.

2. Experimental

The raw materials used in this investigation were china clay (Rajmahal, Bihar, India), plastic clay (Ranchi, Jharkhand, India), quartz, feldspar and pyrophyllite (Maharashtra, India).

The chemical analysis of the raw materials conducted by standard method is given in Table 1.

Batch (Table 2) was wet ground for 16 h in porcelain jars with porcelain balls up to a fineness of around 53 μm. The ground slurry was sieved, passed through a permanent magnet, dewatered and dried in an oven at 120 °C. The dried mass was powdered and passed through a 150 μm sieve, moistened with 6% water and test specimens were pressed in a semi-automatic press of 5 MT capacity. Specimens were made in the form of rectangular bars of 100 mm × 15 mm and 5 mm thickness. The test specimens were dried and subsequently fired between 1150 and 1300 °C in an electric furnace with 2 h soaking at the respective peak temperatures.

Table 1
Chemical analysis of raw materials (wt%).

Constituency	Pyrophyllite	China clay	Plastic clay	Quartz	Feldspar
SiO_2	59.51	48.87	58.38	98.11	66.81
TiO_2	0.26	0.93	1.15	Trace	Trace
Al_2O_3	30.43	34.39	25.54	0.41	18.08
Fe_2O_3	0.86	0.87	0.49	0.22	0.24
CaO	0.37	1.42	0.95	0.68	1.03
MgO	0.78	Trace	Trace	Trace	0.23
Na_2O	0.73	0.10	0.13	0.15	1.69
K_2O	1.95	0.23	0.63	0.07	10.94
LOI	5.40	12.83	12.24	0.19	0.58

Table 2
Body composition with progressive replacement of china clay and quartz with pyrophyllite (%).

Composition	China clay	Plastic clay	Feldspar	Quartz	Pyrophyllite
PS-1	25	25	25	25	0
PS-2	22.5	25	25	20	7.5
PS-3	20	25	25	15	15
PS-4	17.5	25	25	10	22.5
PS-5	15	25	25	05	30
PS-6	12.5	25	25	0	37.5

Apparent porosity and bulk density of the specimens fired at different temperatures were measured by the water displacement method. Flexural strength in three point bending stress was measured with an electromechanical universal tester (Instron 5500R – Universal Testing Machine). The cross-head speed was 1 mm/min and a span of 80 mm was maintained throughout the experiment. Flexural strength was measured using the formulae $P = 3WL/2\,bd^2$, where P is the flexural strength, W is the breaking load, L is the span length; b and d are the width and depth of the specimens, respectively.

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 some selected sintered samples using a LEO S-430i apparatus. SEM studies were performed on fired and matured specimens, polished and etched (using 5% HF for 60 s duration).

Concentration of crystalline phases was estimated by X-ray diffractometry in a Philips make X-Pert Pro diffraction unit attached with secondary monochromator automatic divergence slit and Ni-filter was used to get monochromatic CuKα radiation. The instrument was run in step scan mode with step size (0.02) and time per step 8 s/step within the angle 5–75°. The collected data were refined using profit software. X-Pert plus and Quaser software based on Rietveld were used to calculate the percentage of mullite and quartz phases where standard mullite and quartz were used as reference materials.

3. Results and discussion

The chemical analysis (Table 1) shows that clay, quartz and feldspar used in the present study were of the standard type. In the pyrophyllite sample SiO_2 and Al_2O_3 are the major constituents with low Fe_2O_3 and alkali ($K_2O + Na_2O$) content. The XRD pattern of pyrophyllite [11] showed, in addition to the presence of pyrophyllite as the major phase, some quartz inclusions. In the batch compositions china clay and quartz in the ratio of 1:2 were progressively replaced by pyrophyllite. The oxide compositions of all the experimental bodies are given in Table 3. The results revealed that there was gradual decrease in SiO_2 and increase in Al_2O_3 content among the compositions

Table 3
Oxide composition of the different compositions (wt%).

Constituents	Composition					
	PS-1	PS-2	PS-3	PS-4	PS-5	PS-6
SiO_2	72.285	70.655	69.025	67.395	65.765	64.135
TiO_2	0.60	0.59	0.58	0.57	0.56	0.55
Al_2O_3	21.88	23.27	24.66	26.05	27.44	28.83
Fe_2O_3	0.51	0.55	0.59	0.63	0.67	0.71
CaO	1.11	1.07	1.03	0.99	0.95	0.91
MgO	0.06	0.12	0.18	0.24	0.30	0.36
Na_2O	0.53	0.58	0.63	0.69	0.74	0.79
K_2O	3.03	3.17	3.32	3.46	3.61	3.76
SiO_2:Al_2O_3	3.304	3.036	2.800	2.587	2.397	2.225
$Na_2O + K_2O$	3.56	3.75	3.95	4.15	4.35	4.55

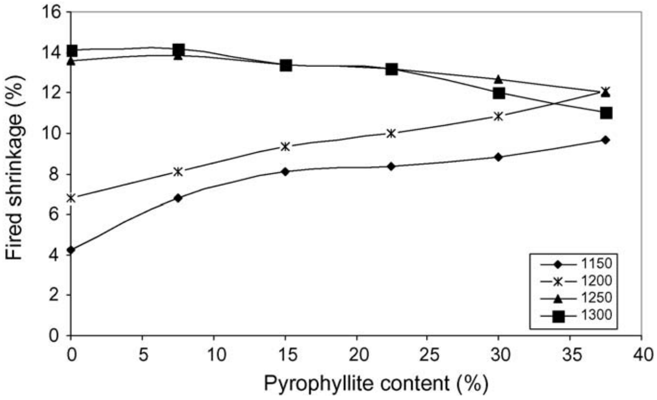

Fig. 2. Variation of fired shrinkage with pyrophyllite content.

(PS-1 to PS-6) when a combination of quartz and china clay was progressively replaced by pyrophyllite. Fig. 1(a) indicates that the SiO_2/Al_2O_3 ratio decreased gradually as pyrophyllite was progressively incorporated and the same varied from 3.30 in mix PS-1 to 2.23 in mix PS-6. Marginal increment in alkali content was observed (varying from 3.56% to 4.55%) as more and more pyrophyllite was incorporated in the mixes [Fig. 1(b)], however, there was virtually no variation in the alkaline earth oxide content among the mixes. Fig. 2 shows that up to 1200 °C fired shrinkage increased with increasing pyrophyllite content, however, at higher temperature although the fired shrinkage was more as expected, but the same was found to follow a decreasing trend with increasing pyrophyllite content. Similar trend was also observed when quartz was progressively replaced by pyrophyllite [11]. A reduction in fired shrinkage at maturity was also observed by several workers [13–16] while studying the effect of pyrophyllite in stoneware type compositions. The curve for apparent porosity values of specimens (Fig. 3) indicates a gradual decrease irrespective of firing temperatures when pyrophyllite was progressively incorporated. The specimens fired at 1250 °C indicated a sharp drop from 4.75% (PS-1) to 0.20% (PS-4). The specimens attained complete vitrification (apparent porosity value below 0.5%) with pyrophyllite content 15% or more. There was hardly any change in apparent porosity values of the specimens PS-3 and PS-4 when temperature was further raised to 1300 °C indicating a fairly wide vitrification range of these mixes. However, mixes with pyrophyllite content 30% and above (specimens PS-5 and PS-6) showed overfiring tendency at 1250 °C or above. Similar observations were made when quartz was gradually replaced by pyrophyllite [11]. Variation in flexural strength of fired specimens with increasing pyrophyllite content has been presented in Fig. 4. It is interesting to note that MOR values gradually increased as more and more pyrophyllite was incorporated in the mixes as a replacement of china clay and quartz. However, highest strength (87.43 MPa) was observed with specimen PS-3 at 1300 °C with pyrophyllite content 15%. There was significant decrease in MOR values with further increase in pyrophyllite content beyond 22.5%. However, in all the cases pyrophyllite incorporated compositions produced higher MOR in comparison to that of the

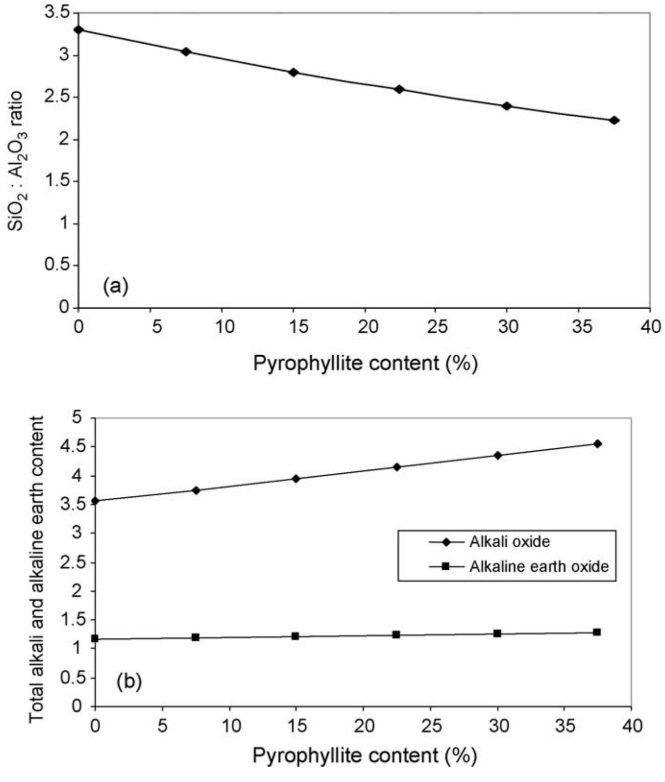

Fig. 1. (a) Variation of silica–alumina ratio with pyrophyllite. (b) Variation of alkali and alkaline earth oxide content with pyrophyllite.

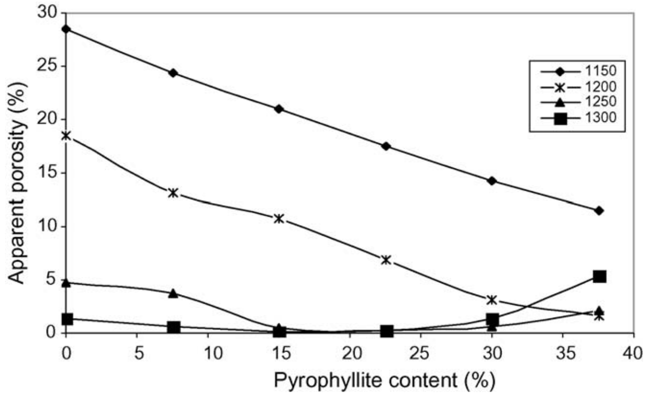

Fig. 3. Variation of apparent porosity with pyrophyllite content.

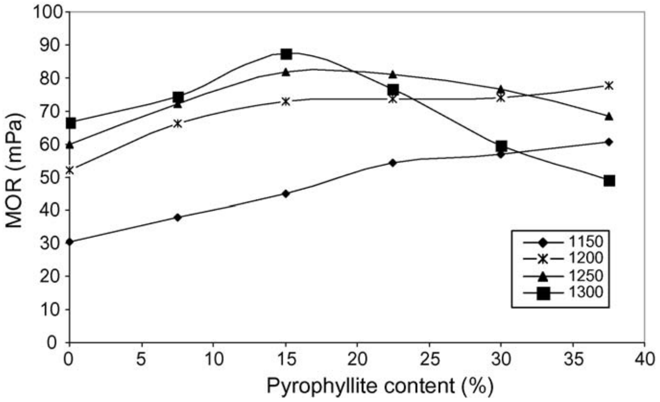

Fig. 4. Variation of MOR with pyrophyllite content.

conventional one, i.e. mix PS-1. The results are quite obvious as bending strength in ceramics increases exponentially with decreasing porosity, very much dependent on the amount of liquid phase present at the firing temperature, and that, the denser the fired bodies, the higher the bending strength [9,10,17]. Variation in bulk density of specimens fired up to 1250 °C (Fig. 5) also showed the similar trend and increased with increasing pyrophyllite content. However, mixes PS-5 and PS-6 containing 30% and 37.5% pyrophyllite, respectively showed substantial decrease in bulk density values even at 1250 °C indicating distinct overfiring of the specimens. The highest bulk density value (2.46 g/cm^3) was observed with specimen PS-3 (containing 15% pyrophyllite) matured at 1300 °C. The maximum bulk density was recorded at the same temperature at which MOR was also highest, as was also observed by others [10]. There was hardly any change in the bulk density value with mix PS-4 (containing 22.5% pyrophyllite) when temperature was raised from 1250 to 1300 °C. It can be inferred that pyrophyllite incorporated porcelain composition (at least up to 22.5% pyrophyllite) has fairly wide vitrification range which is very essential for development of commercial products.

XRD analysis indicated two major phases viz. quartz and mullite in all the mixes. Interestingly no cristobalite phase was observed even at 1300 °C. Analysis of phases of all the specimens (Fig. 6(a) and (b)) indicates higher proportions of mullite in the mixes with pyrophyllite content up to 22.5% in Comparison to that of the conventional one (specimen PS-1 containing no pyrophyllite) irrespective of the firing temperature. Maximum mullite content (18.8%) was observed with specimen PS-3 having pyrophyllite content 15% (Fig. 6(a)) and the same decreased as pyrophyllite was further incorporated as a replacement of china clay (Kaolin) and quartz. Similar types of observations were also observed when firing temperature was further increased to 1300 °C. The presence of pyrophyllite helped in producing a composition which facilitated mullite crystallization [12]. Thus in the present system, when pyrophyllite is gradually incorporated at the expense of china clay, nucleation of mullite and its crystallization to form an interlocking dense microstructure is facilitated. This possibly resulted in higher percentage of mullite in specimen PS-3 as well as improved mechanical properties. Similar observations were made by other research workers [18,19]. It may be noted that simultaneously with mullite formation from dehydroxylated pyrophyllite, large amount of amorphous SiO_2 is released which will definitely go into solution forming SiO_2-rich glassy phase.

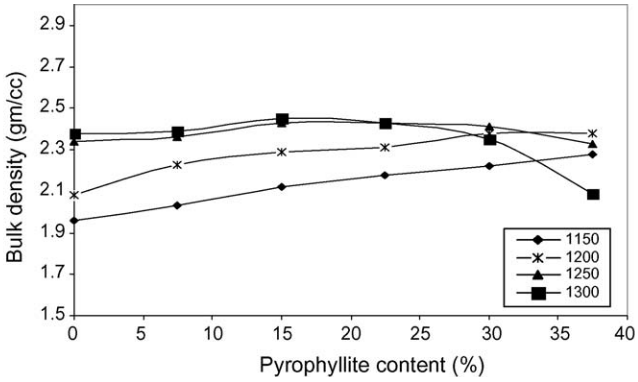

Fig. 5. Variation of bulk density with pyrophyllite content.

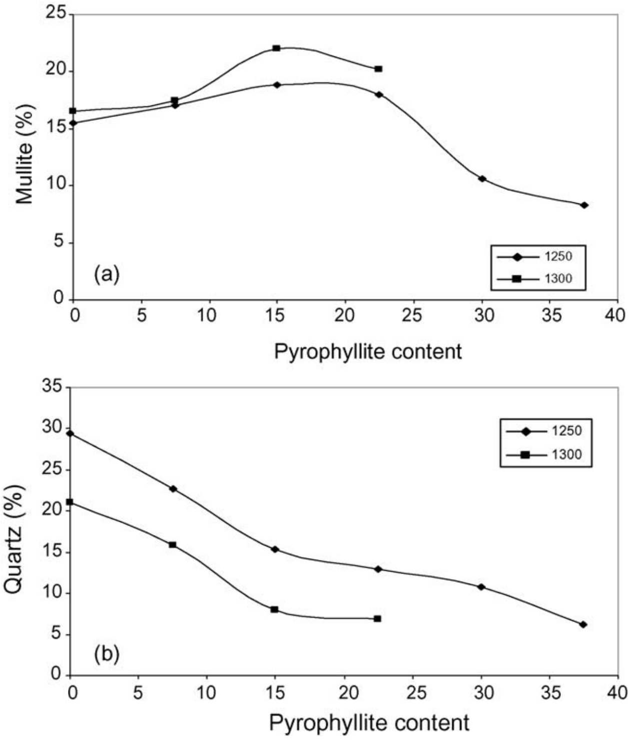

Fig. 6. (a) Percent mullite phase present in the sintered specimens with varying pyrophyllite content. (b) Percent quartz phase present in the sintered specimens with varying pyrophyllite content.

$$\underset{\text{dehydroxylated pyrophyllite}}{3(Al_4Si_8O_{22})} \rightarrow 2\underset{\text{mullite}}{(3Al_2O_3,\ 2SiO_2)} + 20\,SiO_2$$

Beyond optimum limit, addition of pyrophyllite will result in the formation of large volume of SiO_2-rich glassy phase inhibiting the recrystallization of secondary mullite phase. At the same time this glassy phase deteriorates the mechanical properties of the specimens as is observed with specimens containing more than 15% pyrophyllite. The XRD study also revealed that specimen PS-6 (mix with no extra addition of

Table 4
Phase analysis (wt%) of specimens of the different compositions fired at 1250 and 1300 °C.

Composition	Mullite		Quartz		Glass	
	1250 °C	1300 °C	1250 °C	1300 °C	1250 °C	1300 °C
PS-1	15.5	16.5	29.4	21.0	55.1	62.5
PS-2	17.0	17.5	22.7	15.8	60.3	66.7
PS-3	18.8	22.0	15.3	8.0	65.9	70.0
PS-4	18.0	20.2	12.9	6.9	69.1	72.9
PS-5	10.6	nd	10.8	nd	78.6	nd
PS-6	8.3	nd	6.2	nd	85.5	nd

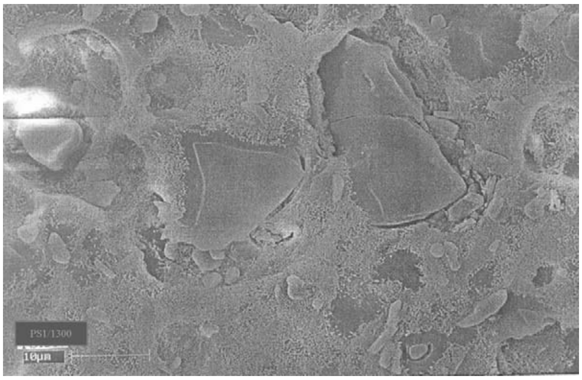

Fig. 8. SEM of composition PS-1 at 1300 °C.

quartz) contained 6.2% quartz phase at 1250 °C. Presence of free quartz in the other raw materials contributed to the increased values. The quartz phase decreased substantially at 1300 °C irrespective of the specimens analyzed [17].

Table 3 indicates that as pyrophyllite gradually replaced quartz and china clay in the mixes, total alkali ($Na_2O + K_2O$) and Al_2O_3 content increased progressively while there was a gradual decrease in the SiO_2 content. Again, as more and more pyrophyllite is incorporated in the compositions, the pyrophyllite gets dissolved in the feldspathic melt in preference to quartz [11]. Formation and recrystallization of secondary mullite are therefore facilitated in the compositions containing higher proportion of pyrophyllite and thus supports the results of Table 4. However, incorporation of pyrophyllite beyond 22.5% resulted in the formation of large volume of glassy phase at higher firing temperature as discussed above and thereby deteriorating the ceramic properties. Formation of mullite phase also reduced drastically and the same was found to be only 10.6% with mix PS-5 at 1250 °C. Interestingly it was observed in our earlier study [11] that beyond 15% pyrophyllite there was significant deterioration in the ceramic properties when quartz was progressively replaced by pyrophyllite.

SEM analysis of specimen PS-1 fired at 1250 °C (Fig. 7) revealed two different types of mullite; clay agglomerate relicts containing primary granular type mullite and a matrix of clay-feldspar and quartz containing elongated and acicular type mullite in agreement with the observations of Lee and Iqbal [20]. The general microstructural features of commercial porcelain have been observed i.e. coarse quartz grains surrounded by solution rims of almost pure silica glass embedded in a much finer matrix system. Distinct cracks and fissures have been observed in the large quartz grains indicating severe strains around the grains. Quartz grains were rather large and angular in nature. These flaws were formed as a result of quartz and matrix expansion mismatch and the same determine the maximum possible strength of the porcelain as observed by Kobayashi et al. [17]. The matrix is predominantly glass of varying composition being alkali (Na, K) aluminosilicate in flux penetrated regions. At 1300 °C (Fig. 8) their was extensive dissolution of quartz as is evident from wider solution rim around quartz grains as well as the presence of small round shaped quartz grains. Presence of quartz grain larger than 15 μ with cracks was also observed. Dissolution of quartz depends on its size i.e. dissolution of coarser quartz grains is slower than that of fine grains.

The proportion of acicular mullite increased as more and more pyrophyllite was incorporated. Figs. 9 and 10 (SEM of PS-3 and PS-4, respectively fired at 1250 °C) showed well formed needle shaped secondary mullite forming a network throughout the microstructure. In concurrence with the observations of Lee and Iqbal [20,22] two different types of secondary mullite crystals were observed. One variety (Type III) with very high aspect ratio (30–40/1) and the second one of Type II with lower aspect ratio (3–10/1) in feldspar penetrated clay relicts. The elongated Type III mullite crystals occur in regions also containing Type II and were associated with a fluid matrix. The elongated type mullite crystals were very prominent in the microstructure of specimens PS-3 and PS-4

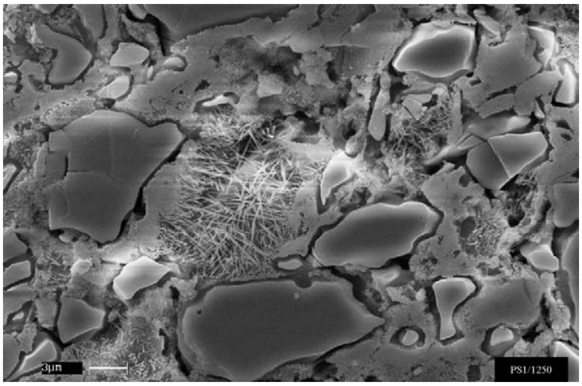

Fig. 7. SEM of composition PS-1 at 1250 °C.

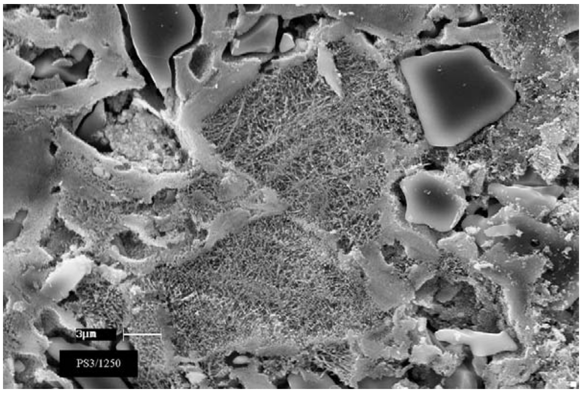

Fig. 9. SEM of composition PS-3 at 1250 °C.

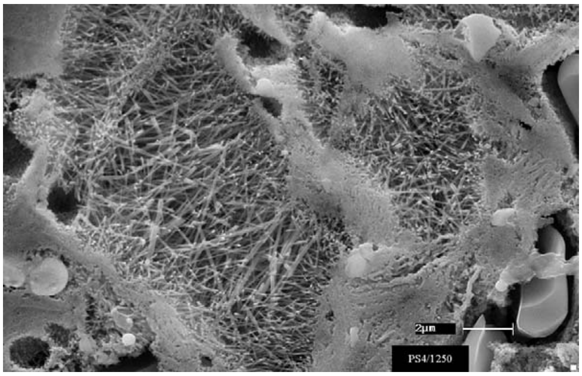

Fig. 10. SEM of composition PS-4 at 1250 °C.

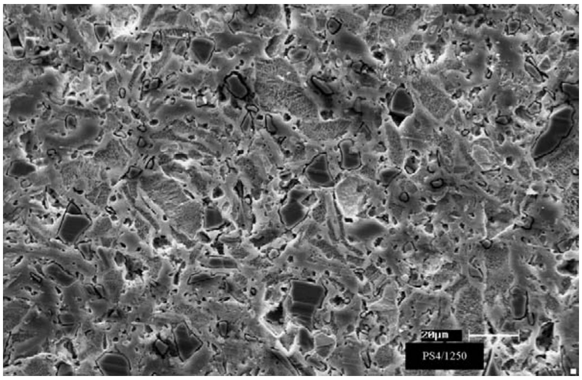

Fig. 12. SEM of composition PS-4 at 1250 °C describing the nature of pores.

containing 15% and 22.5% pyrophyllite, respectively. Incorporation of pyrophyllite as a progressive replacement of quartz not only contributes to a lower amount of quartz filler in the final body but also is capable of generating substantially higher amounts of glassy phase (Table 4). The viscosity of the liquid at least partly dictates the growth rate of crystalline phases [21]. The more viscous the liquid, the lower is the growth rate. In the pyrophyllite containing mixes, the Si present in the mineral pyrophyllite is more accessible for the high temperature liquid compared with Si in the quartz particles which undergo only partial dissolution. Along with Si and Al, alkali (Na and K) and alkaline earth (Ca and Mg) ions present in pyrophyllite also go into the feldspathic melt and thereby progressively reducing the viscosity of the liquid phase at high temperature. The observed variation in the morphology and the size of mullite crystals may be explained by the gradient in viscosity of the relevant matrix and subsequent more rapid mass transport [1]. The increase in fluidity of the matrix resulted in enhancing the size of secondary mullite (Type III) and thus supports the observations of Lee and Iqbal [20,23]. The relatively higher proportion of mullite and its well developed interlocking network structure distributed throughout the matrix in the specimens of PS-3 and PS-4 contributed significantly to the development of higher flexural strength (Fig. 4). As the relative proportion of pyrophyllite was increased in the mixes the quartz grains tend to become round and smaller in sizes. The wide solution rims around quartz grains showed their extensive dissolution. Large numbers of small and irregular shaped pores were found to be distributed throughout the microstructure of specimen PS-3 (Fig. 11). In comparison, the microstructure of specimen PS-4 (Fig. 12) revealed that the size of the pores was rather large and some were interconnected in nature. Presence of interconnected pores is believed to initiate flaws which will reduce the strength of the porcelain. It may be revealed from Table 4 that mullite content was very much comparable for the specimens PS-3 and PS-4, however, proportion of glassy phase varied from 65.9% (specimen PS-3) to 69.1% (specimen PS-4) at 1250 °C and the same increased significantly when pyrophyllite was further incorporated beyond 22.5%. Increase in the number of interconnecting pores as well as volume of glassy phase contributed significantly in reducing the bending strength of porcelain with pyrophyllite content more than 15%. Increased strength and toughness have also been reported in porcelains containing increased amounts of high aspect ratio mullite [24]. This also supports the observed results presented in Fig. 4. As temperature went beyond 1250 °C, the mullite needles possibly coarsen, leading to a smaller number of large needles (Fig. 13). Quartz grains were much smaller due to extensive dissolution. Fig. 14 shows the micrograph of PS-4 at 1300 °C. Large volume of glass formation with bigger voids of spherical and elliptical shapes is quite conspicuous. With increase in temperature there will be further decrease in the viscosity of the melt and this resulted in the transformation of the shape and size of the pores. SEM analysis of specimens PS-5 and PS-6 fired at 1250 °C (Figs. 15 and 16) revealed larger volume of glassy phase formation with needle shaped secondary mullite crystals. Although there was no externally added quartz in PS-6 mix,

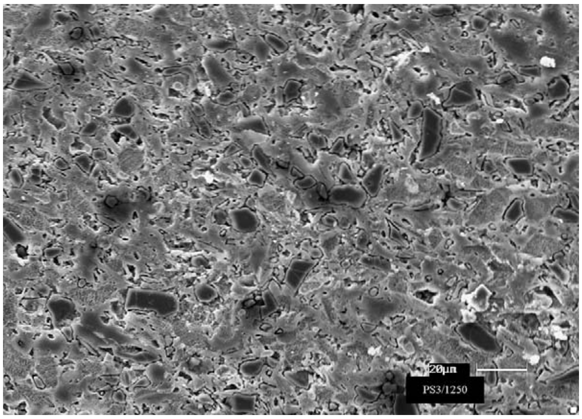

Fig. 11. SEM of composition PS-3 at 1250 °C describing the nature of pores.

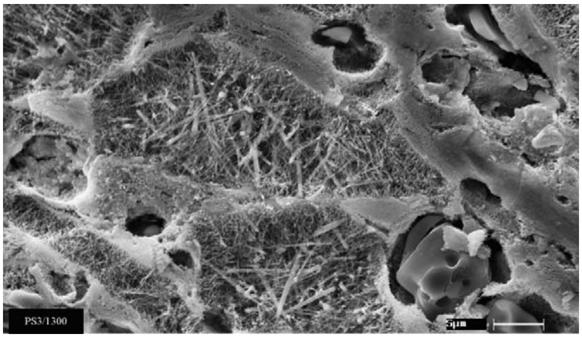

Fig. 13. SEM of composition PS-3 at 1300 °C.

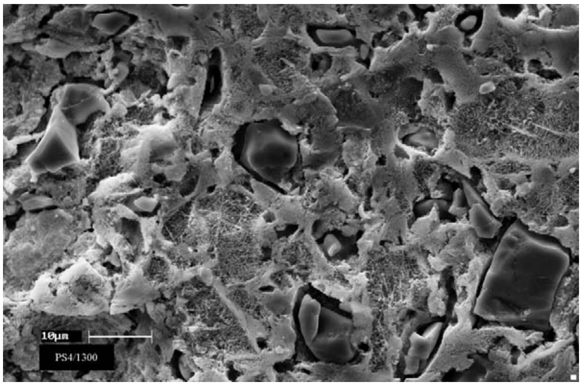

Fig. 14. SEM of composition PS-4 at 1300 °C.

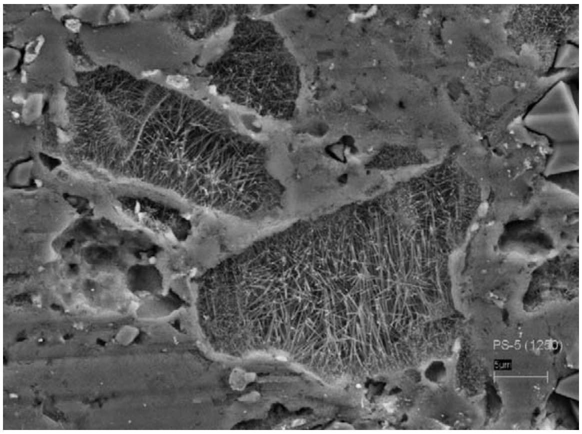

Fig. 15. SEM of composition PS-5 at 1250 °C.

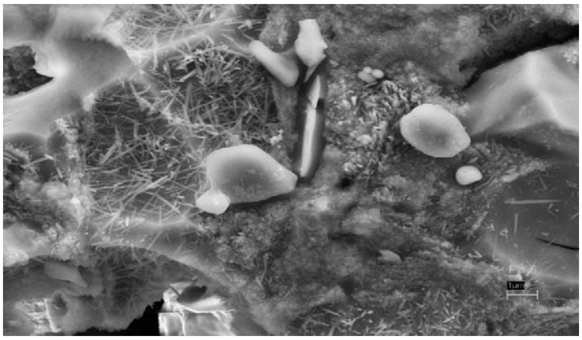

Fig. 16. SEM of composition PS-6 at 1250 °C.

Fig. 16 clearly indicated the presence of quartz grains. The quartz grains reacted extensively with the glassy phase and transformed into practically spherical shapes. Elongated mullite needles are quite conspicuous. Presence of large volume of glassy phase (Table 4) affected the ultimate ceramic properties of the specimens PS-5 and PS-6.

4. Conclusions

Incorporation of pyrophyllite as a progressive replacement of china clay (Kaolin) and quartz in a porcelain composition resulted in early vitrification. Addition of pyrophyllite reduced fired shrinkage by 5.03% and improved the flexural strength (around 31.5%) as compared to those of the conventional body due to development of interlocking mullite needles. Although there was progressive reduction in kaolinite content, percentage of mullite was found to increase in the mixes with incorporation of pyrophyllite. However, maximum flexural strength and mullite content were observed with mix having 15% pyrophyllite content at 1300 °C. Beyond some optimum proportion of pyrophyllite (in this study higher than 22.5%) there occurred a large volume of glass formation as well as large pores of various shapes non-uniformly distributed in the microstructure which resulted in deterioration in ceramic properties as well as mullite content. The size and shape of mullite crystals is to a large extent controlled by the fluidity of the liquid matrix and this is again a function of temperature and composition. Significant decrease in quartz phase was observed when temperature was raised from 1250 to 1300 °C however there was marginal increase in mullite phase.

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