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Effect of substitution of quartz by rice husk ash and silica fume on the properties of whiteware compositions

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Abstract

Active silica from rice husk ash (RHA) and silica fume (SF) were progressively incorporated in a whiteware composition in substitution of quartz. The Influence of the addition on the thermo-mechanical properties, vitrification behavior and microstructure has been investigated. It has been found that replacement of quartz by RHA+ SF (1:1) reduced drastically both the maturing temperature (50–100 °C) and the thermal expansion (3.24–14.6%) at 600 °C with the improvement in the fired strength. Maximum improvement in the fired MOR (20.8%) was observed in a whiteware composition containing 10% (RHA + silica fume). On complete replacement of quartz (25%) by RHA + SF, the fired strength was noticed around 14.95% in whiteware body matured at lower temperature by 100 °C.The improvement in the properties is attributed to sharp changes in the microstructural features as a result of significant reduction in the content of the quartz phase and the simultaneous increase in glassy phase. The findings would be helpful to improve the properties of whiteware as well as to reduce the energy consumption during firing process.

1. Introduction

Rice husk ash (RHA) contains an active form of silica (SiO_2) and is available in large quantities in India [1]. Rice Husk contains ash from 13 to 29% by weight depending on the variety, climate and geographic location [2]. The presence of silica as SiO_2 in rice husk ash has been known [3] since 1938. The silica in rice husk is in hydrated amorphous form, either opal or silica gel [4,5]. The use of rice husk ash in the form of silica in the ceramic field was also reported by Samadani et al. [6] Borthakur et al. [7] also investigated physico-chemical properties of RHA for its application. Authors also studied the effect of RHA in white ware composition [8] and found improvements in the properties with the reduction in the maturing temperature. Silica fume [9,10], an anhydrous amorphous silica formed at high temperature is collected from the gas phase in submicron particles which has high surface area spherical shape and extremely low bulk density. Authors also studied the effect of silica fume in whiteware composition [11] and found improvements in the thermo-mechanical properties with the reduction in the maturing temperature.

Almost all triaxial whiteware compositions contain quartz as a major crystalline phase. Quartz grains embedded in the glassy matrix of a whiteware have a deleterious effect on the mechanical strength mainly because of α-β quartz transformation during cooling [12–14] resulting in the development of stress around quartz grains which initiate fracture [15].

Due to the change in volume of free silica during reconstruction and displacive transformation, the thermo-mechanical properties of white ware bodies change considerably as reviewed in detail [16]. Several investigators [17–26] observed improvements in the mechanical properties of whiteware bodies with the addition of sericitic pyrophyllite, kyanite, bauxite, sillimanite sand and alumina in substitution of quartz. On the other hand the silica rich glass favors the recrystallization of mullite at low temperature and its dissolution at high temperature [27]. It is a known fact that quartz grains in different sizes have significant effects on mechanical strength of porcelain [28]. A similar conclusion had been

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also reached that the mechanical strength of porcelain is influenced mainly by stresses set up in the glassy matrix rather than by the amount or size of mullite crystals formed [29]. In addition, the pre-stress effect increases with an increase in the amount of residual quartz [29]. Some investigators claimed that the low quartz content provides high strength [30,31].

Tomizaki et al. [32] observed that dissolution of quartz in the glassy phase and precipitation of cristobalite phase are dependent on the silica particle size. The low quantity of quartz, the high content of cristobalite and the low internal porosity have detrimental effects on mechanical strength.

Rice husk ash and Silica fume contain active silica in high proportion which have so far not been used in whiteware manufacture. Rice husk ash with minor or negligible amount of carbon and silica fume, if incorporated in the whiteware composition in substitution of quartz, could be expected to create improvements in the thermo-mechanical properties and might be beneficial for the development of technical and electro ceramics.

Keeping this in mind, the present investigation was made to study the effect of RHA + SF in whiteware compositions at different temperatures in respect to vitrification, thermo-mechanical properties and microstructure.

2. Experimental procedure

The test specimens of starting composition of whiteware was prepared using Amrapali china clay (Supra grade), potash feldspar, calcined quartz. All the raw materials are available abundantly in India and are being used for the production of white ware ceramics in the country. In this investigation, Rice husk ash (RHA) and silica fume (SF) were incorporated in the whiteware composition in the substitution of quartz (Table 1). RHA incorporated in the whiteware body was prepared by controlled burning of raw rice husk in air so as to avoid the presence of carbon (<5 wt.%) as far as practicable. Rice husk ash was wet ground in a ball mill using porcelain balls as grinding media for 35 h.

Table 1
Body composition with progressive replacement of quartz by rice husk ash and silica fume in a standard whiteware composition (mass%)

Raw materials	Body		Mix		No.	
	CSR-1	RSF-1	RSF-2	RSF-3	RSF-4	RSF-5
Amrapali china clay	50	50	50	50	50	50
Potash feldspar	25	25	25	25	25	25
Calcined quartz	25	20	15	10	5	0
Rice husk ash (RHA)	0	2.5	5	7.5	10	12.5
Silica fume (SF)	0	2.5	5	7.5	10	12.5

After drying the slurry it was taken for the formulation of body where as silica fume was used as received from M/s Pooja Enterprises, Mumbai, which has a surface area 14 m^2/g. Calcined quartz and potash feldspar were ground separately in a ball mill by the wet process to the fineness of around 300 mesh. The slurry was sieved, passed through a magnetic channel and dried. The particle size distribution of ground materials as well as china clay and silica fume was determined by the Andreasen pipette method. Chemical analysis of the raw materials were carried out according to the standard procedure.

Different body compositions were prepared using ball milling, sieving and de-watering processes to get plastic masses. The test specimens were extruded in a vacuum extruder (Edward & Jones, England) in the form of cylindrical bars of 1.5 cm in diameter and 15 cm in length. These specimens were dried and then fired between 1100 and 1300 °C at temp. interval of 50 °C in an electric furnace under a heating rate of 3.5 °C/min. with 1hr. soaking. Plastic and dry properties such as water of plasticity, dry linear shrinkage, dry strength and bulk density of dried test specimens were determined as per standard procedure.

Fired properties of test specimens such as fired linear shrinkage, fired strength, water absorption, apparent porosity and bulk density were determined by standard methods. Thermal expansion of matured specimens were determined using the thermo-mechanical analyzer (TMA), Shimadzu, Japan, DT-30 model, under a heating rate of 10 °C/min.

The relative changes of major crystalline phases quartz and mullite in whiteware bodies were identified by XRD and the micro-structural features were studied by SEM on fractured etched surfaces in 10% HF for 5 min for the existence of different phases. Specimens, cleaned by ultrasonic vibration were coated with a thin layer of gold palladium by sputtering. The coated surfaces were used for the study of microstructure under a Stereo-Scane S250 scanning electron microscope.

3. Result and discussion

Different body mixes (Table 1) were prepared for the evaluation of properties in green as well as after firing. Chemical analysis of raw materials, used in the body formulation are presented in Table 2. The chemical analysis of raw material indicated the presence of constituents in required amounts for the formulation of whiteware compositions. The content of SiO_2 in quartz was observed 96.80% whereas rice husk ash silica fume contain 88.44% and 96.05% SiO_2 respectively. Rice husk ash indicated high amount of alkalies (2.31%) as a K_2O and Na_2O. Rice husk ash also indicated a loss on ignition 4.62% which may be due to the presence of

Table 2
Chemical analysis of raw materials (mass%)

Oxides	Amrapali china clay (supra grade)	Potash feldspar	Calcined quartz	Rice husk ash	Silica fume
SiO_2	46.24	65.65	96.80	88.44	96.05
Al_2O_3	35.46	17.84	1.90	1.21	1.53
Fe_2O_3	1.16	0.16	0.10	0.40	0.39
TiO_2	0.76	Trace	Trace	Trace	Trace
CaO	1.58	0.20	0.15	1.20	Trace
MgO	0.08	0.24	0.02	1.82	0.60
Na_2O	0.10	2.75	0.46	0.50	0.34
K_2O	0.42	12.29	0.14	1.81	Trace
L.O.I.	14.10	0.84	0.40	4.62	0.90

carbon in the rice husk ash. The presence of alkalies would favour early vitrification of whiteware bodies.

The results of wet sieve analysis and particle size distribution of clay and ground raw materials are presented in Table 3. The results showed that the particle size of quartz, rice husk ash and silica fume below 15 μm are 87%, 79% and 91.5% respectively. The particle size distribution of all the raw materials are identical.

Plastic and dry properties of body mixes are presented in Table 4. The water of plasticity increased gradually. This may be due to the addition of SF + RHA of more fineness in whiteware compositions. Dry shrinkage of bodies was also observed in increasing trends from 3.62% to 5.60% which may be due to finer particles as well as more water of plasticity in whiteware bodies. The dry bulk density of green body with 5% (RHA + SF) was observed with a maximum of 1.72 g cm^{-3} which may be due to the appropriate proportion of particles of different sizes in the body mix. Gradual increase of RHA and silica fume decreased the dry bulk density of test specimens which may be due to increase in fine pores during drying on removal of water from inside of the body mass. The dry strength (MOR) observed more or less same in the range of 0.99–1.11 $N.mm^{-2}$. The marginal improvement in the strength of body may be due to addition of rice husk ash and silica fume as well as particles of appropriate sizes.

Table 3
Particle size distribution of raw materials used for body formulation (mass%)

Oxides	Amrapali china clay (supra grade)	Potash feldspar	Calcined quartz	Rice husk ash	Silica fume
Sieve analysis					
+150	0.10	0.06	0.14	0.40	0.00
−150 + 106	0.30	0.55	0.32	0.52	0.00
−106 + 75	0.35	0.20	0.50	0.40	0.10
−75 + 53	2.15	0.70	0.45	0.62	0.20
−53	97.10	98.49	98.69	98.06	99.70
By Sedimentation technique					
Greater than 25 μm	7.0	2.5	5.0	9.5	2.0
Between 25 and 15 μm	6.0	10.0	8.0	11.5	6.5
Between 15 and 10 μm	4.0	5.5	14.0	8.0	7.5
Between 100 and 5 μm	10.0	11.5	9.0	16.0	7.5
Between 5 and 3 μm	11.0	22.5	12.0	13.0	5.5
Between 3 and 2 μm	8.0	11.0	14.0	8.0	3.0
Between 1 μ	46.0	22.0	20.0	23.0	62.0
Below 10 μ	83.0	82.0	82.0	71.0	84.0
Below 15 μ	87.0	87.5	87.0	79.0	9.5

Table 4
Plastic dry properties of body mixes

Properties	Body		Mix		No.	
	CSR-1	RSF-1	RSF-2	RSF-3	RSF-4	RSF-5
Water of plasticity (%)	21.4	22.2	24.2	24.8	25.0	25.6
Dry linear shrinkage (%)	3.62	4.86	5.26	5.26	5.42	5.60
Dry M.O.R. ($N.mm^{-2}$)	0.99	1.08	1.11	1.11	1.11	1.11
Bulk density (g cm^{-3}) (dry test pieces)	1.62	1.72	1.70	1.65	1.60	1.60

The effect of rice husk ash and silica fume in substitution of quartz in whiteware bodies are shown in Figs. 1–4. The standard body marked CSR-1 showed the maximum fired linear shrinkage around 11.68% in the fired specimens at 1250 °C whereas RSF-5 body containing 25% rice husk ash and silica fume showed 15.61% linear shrinkage at 1150 °C. The curves (Fig. 1) indicated gradual increase of fired shrinkage at the vitrification point and a maximum increase of 33.65% was noticed in comparison with standard body CSR-1. However, the increase was around 19% for RSF-2 at maturity (1200 °C) on partial replacement of RHA + SF (10 wt.%). This substantial increase in the firing shrinkage of mixes is attributed to the flabby nature of RHA + SF particles and SiO_2 in the form of reactive silica. The conventional whiteware composition

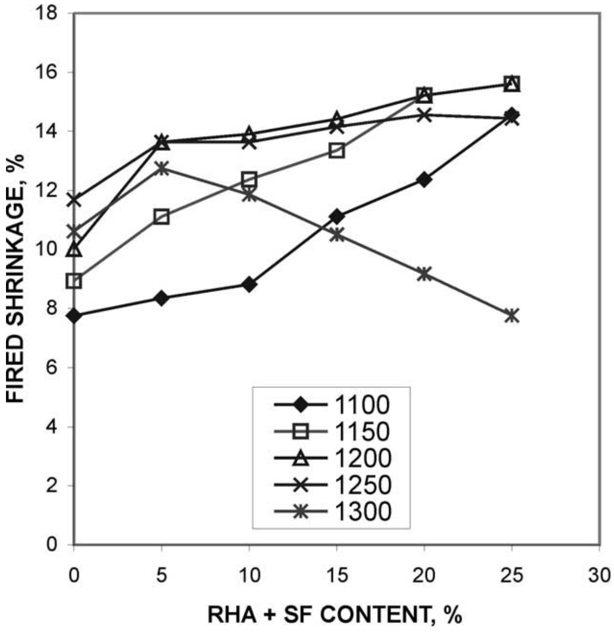

Fig. 1. Effect of RHA + SF on the fired shrinkage of body mixes fired at different test temperature.

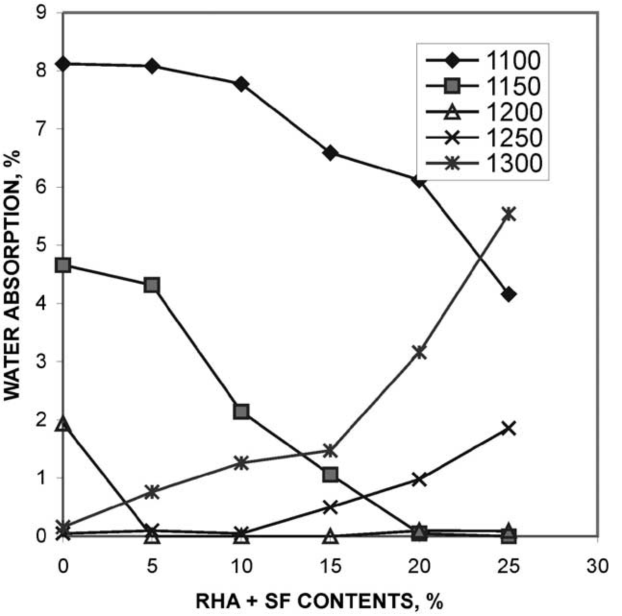

Fig. 2. Effect of RHA + SF on the water absorption of body mixes fired at different test temperature.

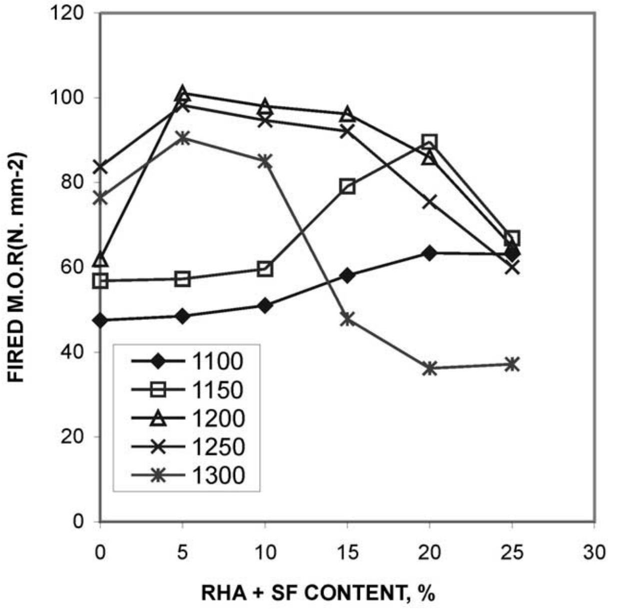

Fig. 3. Effect of RHA + SF on the fired mor of body mixes fired at different test temperature.

(CSR-1) indicated zero water absorption (Fig. 2) at a temperature of 1250 °C with a bulk density (Table 5) of 2.38 g cm^{-3}. With progressive incorporation of RHA + SF, the maturing temperature decreased by about 50 °C in RSF-1, RSF-2, RSF-3 containing 5, 10 and 15 wt.% respectively. These mixes showed a wide vitrification range (~50 °C). However, the body mix RSF-4 and RSF-5 on replacement of quartz by RHA + SF 20 and 25 wt.% respectively, attained early vitrification (Fig. 2) at comparatively low temp. of 1150 °C only with bulk density of 2.37 and 2.36 g cm^{-3} (Table 5) respectively. The maximum bulk density 2.41 g cm^{-3} was observed in the sample containing 5, 10, 15 wt.% RHA + SF and fired at 1200 °C. The fired strength also confirmed the maximum bulk density of the test specimens fired at 1200 °C.This body also showed a wide vitrification range of about 100 °C. The reduction in the vitrification temp. of test specimens is attributed to the finer particle size [33] of the starting materials coupled with intimate mixing as well as the presence of RHA + SF in the form of active silica. The alkalies present in the RHA + SF also took part for the early vitrification of the test specimens of whiteware compositions.

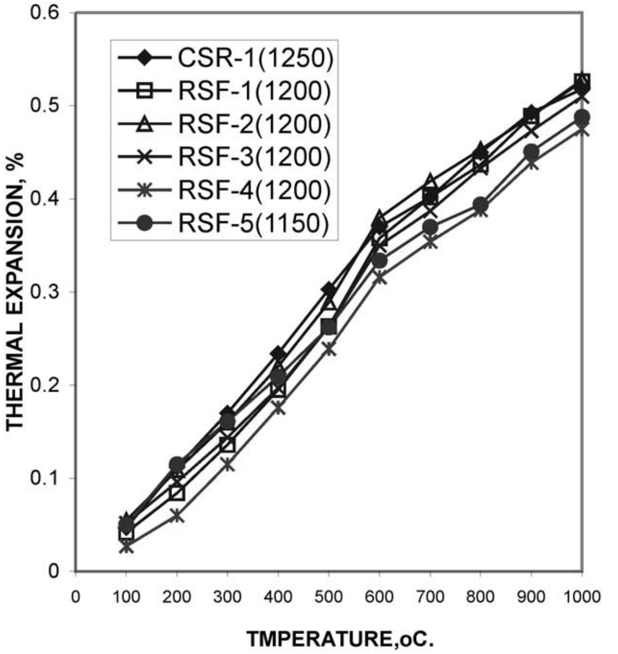

Fig. 4. Percentage of linear thermal expansion of matured test specimens as a function of temperature.

Table 5
Bulk density of different body mixes fired at different test temperatures

Mix no.	Bulk		Density		
	1100 °C	1150 °C	1200 °C	1250 °C	1300 °C
CSR-1	2.03	2.18	2.28	2.38	2.36
RSF-1	1.82	2.20	2.40	2.39	2.28
RSF-2	2.06	2.31	2.41	2.38	2.18
RSF-3	2.10	2.32	2.41	2.34	1.97
RSF-4	2.12	2.37	3.36	2.21	1.73
RSF-5	2.15	2.36	2.33	2.12	1.69

Fired strength of test specimens is shown in Fig. 3. Fired strength (MOR) of test specimens of different whiteware compositions increased with the increase in the test temperatures up to the point of respective maturing temperatures and then decreased due to over firing. Maximum strength 99.08 N.mm^{-2} was observed

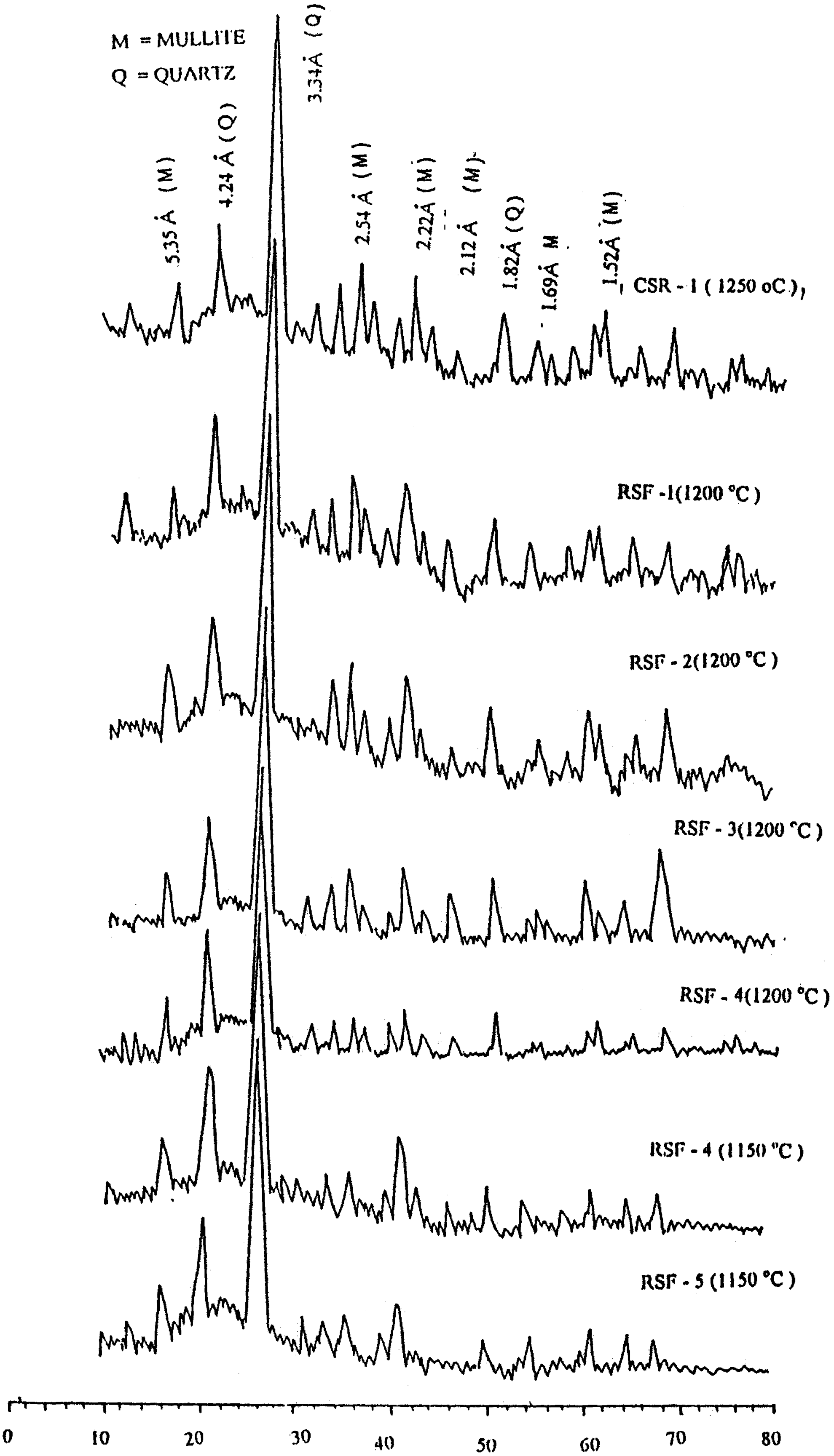

Fig. 5. XRD curves of fired body mixes containing risk husk ash and silica fume.

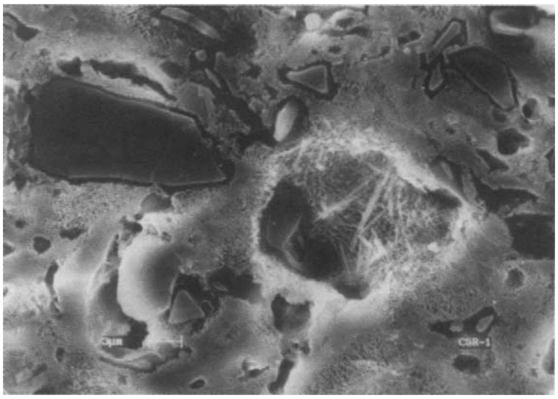

Fig. 6. Short and elongated mullite needles surrounded by other crystalline phases in the matrix of standard body (CSR-1) fired at 1250 °C for 1 h.

with the body containing 10% RHA + SF (in equal parts) fired at 1200 °C which is around 20.8% higher than the standard body marked CSR-1 and fired at 1250 °C. The bodies RSF-4 and RSF-5 containing 20 and 25 wt.% RHA + SF showed the fired strength 87.81 and 66.54 N.mm^{-2} in the specimens fired at 1150 °C. The decrease in the strength in matured specimens of RSF-5 (25% RHA + SF) was noticed to around 20% in comparison with CSR-1 which may be on increase in the content of glassy phase in the microstructure due to higher amount of active silica. It is evident from the results that, whiteware body containing 5–10% RHA + SF did not only lead to the reduction in the maturing temperature compared to the reference body (CSR-1) but also increased the fired strength of the test

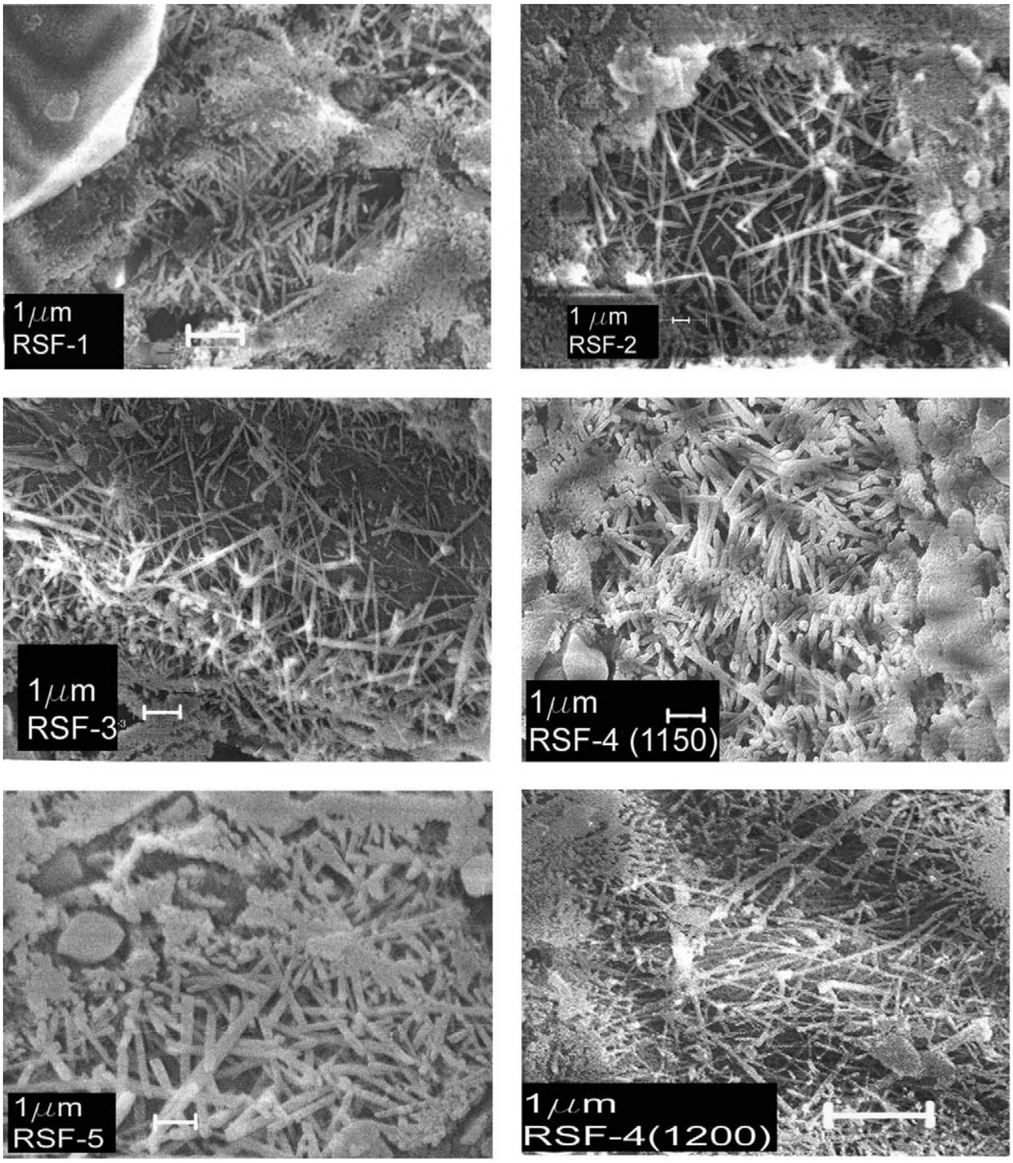

Fig. 7. Micrographs of matured and etched specimens RSF-1 (1200 °C), RSF-2 (1200 °C), RSF-3 (1200 °C), RSF-4 (1200 °C), RSF-4 (1150 °C), RSF-5 (1150 °C) showing large amount of well interlocked mullite crystals and quartz reics embedded in the matrix. The size of mullite crystals and quartz relics varying with the addition of RHA + SF in different proportions.

specimens of whiteware composition (RSF-1 and RSF-2) at 1200 °C.

Thermal expansion of matured test specimens (Fig. 4) decreased gradually with the addition of RHA + SF by replacement of quartz in the whiteware compositions. The removal of quartz from the mixes leads to the disappearance of boundary stresses and hystersis at the quartz transformation temperature of 573 °C. The maximum decrease in the expansion in matured specimen of RSF-4 (1200 °C) was about 14.6% at 600 °C in comparison to the reference body (CSR-1). In RSF-2 body, the decrease in thermal expansion was observed around 5.4%.

The XRD pattern of the matured specimens (Fig. 5) show the presence of two major crystalline phases namely, mullite and quartz in all the body mixes. On the complete replacement of quartz by RHA + SF (RSF-5), the peak intensity of quartz decreased significantly and peak heights for mullite remained almost constant. XRD curves indicated that the addition of RHA + SF substantially decreased the content of free quartz in the composition, which confirmed from the decrease in the percent thermal expansion. Scanning electron micrographs of fracture etched surfaces of specimens of standard body (CSR-1) and RSF-1 (5 wt.% RHA + SF), RSF-2 (10 wt.% RHA + SF), RSF-3 (15 wt.% RHA + SF) RSF-4 (20 wt.% RHA + SF) and RSF-5 (25 wt.% RHA + SF) are shown in Figs. 6 and 7. Fig. 6 for CSR-1 (0 wt.% RHA + SF) shows a large amount of free quartz remained undissolved in the final microstructure along with the occurrence of well interlocked secondary mullite needless, which crystallized from the melt at the maturing temperature 1250 °C. The unreacted quartz grains as evident in the microstructure are responsible for the early failure of the standard white ware mix as a result of mechanical and thermal loading [19,34].

The micrographs Fig. 7 (RSF-1, RSF-2, RSF-3) show the change in the size of mullite crystals and quartz relics in the fired test specimens (at 1200 °C), which is due to the gradual addition of RHA + SF in the white ware composition. Test specimens fired at 1200 °C containing 5 wt.%, 10 wt.% and 15 wt.% RHA + SF (Fig. 7 showed a large amount of well interlocked mullite crystals embeded in the glassy matrix. The fired strength of these matured specimens were also observed at the higher side. The improvement in the strength may be due to development of large amount of well interlocked mullite crystals in the microstructure. These are also observed in the specimen containing 20 and 25% RHA + SF but fired at 1150 °C. The change in the size of mullite crystals due to the addition of 20 and 25 wt.% of RHA + SF in whiteware compositions might be due to well reaction at low temperature and due to formation of more glassy phase in the microstructure.

The major phases present in the micrographs (Figs. 6 and 7) were quartz and mullite apart from the glassy phase and the pores. However, the presence of pores and quartz relics appeared to be less in the micrographs especially in the sample containing RHA + SF. The presence of quartz relics decreased on replacement of quartz by RHA + SF. It has thus been possible to eliminate to a large extent, the inherent defects in the microstructure of a conventional whiteware, and improvement in the mechanical and thermal properties of body with the use of RHA + SF. It has been achieved with the use of appropriate proportion of rice husk ash and silica fume.

4. Conclusion

From the investigation, it appeared that whiteware compositions containing 5–15% RHA + SF in substitution of quartz, lower the vitrification temperature by 50 °C with the improvement in the fired strength as well as improvement in the high amount of interlocked mullite crystals in the microstructure compareds to that of the reference body. The decreases in the thermal expansion are attributed to the sharp decrease in the content of quartz. However, the content of mullite appeared to be unaffected due to addition of RHA + SF in the compositions but with a change in the size of mullite crystals and its orientation as observed in the micrograph. With the use of 20–25% RHA + SF was reduced the vitrification temperature by 100 °C but increased the fired shrinkage which may be due to formation of more glassy phase in the body. Overall, the addition of 10% RHA + SF indicated better properties with the reduction in the vitrification temperature which would contribute significantly to the economical production of whiteware ceramics due to reduction in energy consumption.

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