Phase and Micro-Structural Characterization of Sanitary-Ware Fired at Different Temperature

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ABSTRACT

The three main ingredients of sanitary-ware are clay, feldspar and quartz. This ware is being widely used and has therefore, attracted the attention of researchers from time to time. Consequently, it has been extensively investigated. The present study describes the phase and micro-structural analysis of sanitary-ware samples collected from local (Durr Ceramics Peshawar) industry. XRD (X-Ray Diffraction) of samples fired at 1100^{o}C reveals the presence of α-quartz and primary mullite only. In addition to a-quartz and primary mullite, elongated needles of secondary mullite were also present in samples fired at 1200 and 1300^{o}C. Unlike typical vitreous ceramics bodies, regions containing elongated secondary mullite originating from the clay relict and growing into the feldspar relict were few in number which is consistent with the high clay content in the starting body ingredients of the investigated samples.

Another sample investigated for comparison purposes, contained all the phases mentioned above along with some corundum grains which indicated that the composition of sanitary ware varied from manufacturer to manufacturer. EDS (Energy Dispersive Spectroscopy) detected high concentration of Fe in some regions in the bulk but the glaze did not contain any Fe.

Key Words: Clay, Flux, Mullite, Soda Glass, Filler.

1. INTRODUCTION

Sanitary-ware refers to the group of ceramics made from clay, flux and filler. The firing temperature of sanitary-ware is about 100-150^{o}C lower than that of porcelain (Gragam [1]). Sanitary-ware is in human use since centuries and has therefore, attracted the attention of many researchers from time to time.

The properties of materials are strongly dependent upon their processing conditions, constituent phases and microstructure, and the same is the case with sanitary-ware. Therefore, the processing, phase and micro-structural evolution of vitreous ceramics including sanitary ware has been investigated previously (Klein, [2], Weymouth [3], Jackson [4], Cole [5] and Lee and Iqbal [6]).

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2. MICROSTRUCTURE PROPERTY RELATIONSHIP OF SANITARY-WARE

Sanitary-ware products are required to have minimum water absorption index, high scratch resistance, resistance to chemical detergents, crazing resistance, thermal shock resistance and maximum mechanical strength (Manfredini et. al. [7]). The manufacturers aim at producing good quality products and therefore, the researchers have continuously investigated the role of initial body ingredients, processing and their effect on the properties of finished products.

In sanitary-ware bodies, primary and secondary mullite crystallize from metakaolin (Al_2O_3:$2SiO_2$) coming from the de-hydroxylation of kaolinite and grow into the less viscous feldspar relict ($K_2O.Al_2O_3.6SiO_6$). McConville et. al. [8] reported that the Kaolin transformed to Meta-kaolin at 550ºC. However, Qiu et. al. [9] reported the formation of meta-kaolin by de-hydroxylation of Kaolinite at 420-660ºC.

$$\mathrm{Al_2O_3.2SiO_2.2H_2O} \xrightarrow{\text{about 420-660}^\circ\text{C}} \mathrm{Al_2O_3.2SiO_2} + \mathrm{2H_2O}$$

At 980ºC, Mata-kaolin begins to decompose into SiO_2 and γ-Al_2O_3 via

$$\mathrm{Al_2O_3.2SiO_2} \xrightarrow{\text{about 980}^\circ\text{C}} \gamma - \mathrm{Al_2O_3} + \mathrm{2SiO}_{2(amorphous)}$$

Amorphous silica produced during the decomposition of clay, re-combines with Al_2O_3 to form mullite at temperatures above 1100ºC (Qiu et. al. [9]).

$$3\gamma\text{-}\mathrm{Al_2O_3} + \mathrm{2SiO}_{2(amorphous)} \xrightarrow{\text{about 1100}^\circ\text{C}} \mathrm{3Al_2O_3.2SiO_2}$$

The Al_2O_3:SiO_2 ratio in primary and secondary mullite has been reported to be 2:1 and 3:2 respectively (Iqbal and Lee [10]).

Various proportions of two types of feldspar are commonly used in the production of commercial white-ware bodies namely potash feldspar and soda feldspar (Schramm and Hall [11]). The viscosity of the glassy phase in soda-feldspar containing porcelain bodies is less than that containing potash feldspar. The consequent increase in the fluidity of the melt accelerates the transformation of primary mullite into secondary mullite at high temperatures ($\geq$ 1200ºC). Therefore, the type and proportion of initial body ingredients can be altered to improve the properties of the product (Schuller [12]).

At 1200ºC, the dissolution of α-quartz speeds up and increases the quantity of liquid phase in the body. Pure clay relicts contain primary mullite (~7nm) and occasionally secondary mullite (~30nm) in clay relicts adjacent to large amount of feldspar relict at 1000ºC. Secondary mullite forms as a consequence of the diffusion process between feldspar and clay. It depends upon the diffusion of K_2O and Al_2O_3. Feldspar melting point is lower than the other body ingredients so that the clay with high contents of feldspar possesses low viscosity. The clay relict agglomerates containing feldspar are more fluid than those without feldspar, so secondary mullites crystals grow larger in less viscous clay-feldspar mixture as compared to primary mullite in non-viscous feldspar-free-clay agglomerates (Iqbal and Lee [13]).

Systematic micro-structural investigation of tri-axial bodies of kaolin {$Al_2Si_2O_5(OH)_4$}, feldspar ($K_2O.Al_2O_3.0.6SiO_2$, $Na_2O.Al_2O_3.0.6SiO_2$, $CaO.Al_2O_3.0.2SiO_2$), and quartz (SiO_2) reveals that Kaolin transforms to metakaolin at 550ºC, feldspar decomposes to meta-stable sanidine at temp > 600ºC and begins to melts at e” 1000ºC (Iqbal and Lee [13]).

Upon heating potash feldspar above 1200ºC, its melting point increases due to decomposition of orthoclase ($KAlSi_3O_8$) into lucite ($KAlSi_2O_6$) and silica-rich glass (Morey and Kumar, [14] cited in Schramm and Hall [11]).

Secondary mullite needles exist in a solution of cordierite crystals, rutile and α-quartz instead of glassy phase in a mixture of crystallizing glass, clay and α-quartz which increases the mechanical strength of porcelain bodies and improves their elasticity (Matti and Kumar [15]). Similarly glazed porcelains are stronger than unglazed bodies because glazing prevents propagation of micro-cracks on the surface of the body (Kobyashi et. al. [16]).

Mechanical properties of ceramics-ware can be improved by adding short fibers of alumina without any reaction with clay matrix (Palmonari et. al. [17]). The mechanical strength of sanitary-ware increases by addition of suitable ratio of soda glass (cullet) SG. SG melts at 720°C and results in the formation of maximum glassy phase at about 1100°C, which increases verification, bulk density, and consequently decreases porosity and water absorption (Youssef et. al. [18]). Thus the moisture expansion of fired clay can be best controlled (Cole [5]).

Low porosity is necessary for good mechanical strength and resistance to moisture expansion. Porosity can cause glaze crazing and loss of strength. Long life of sanitary-ware depends on porosity (Manfredini et. al. [7]).

According to Vaughan and Dinsdale[19].

$$W=k\sqrt{t}$$

Where, W is volume expansion in time "t", and $k = 10^{-8}$ $g.cm^{-2}\ day^{1/2}$ (sorption rate). For example, the recommended value of moisture expansion during one year for a sanitary-ware product of internal area $0.5m^2 (5x10^3 cm^2)$ is 0.1%0.

The mixtures of mullite, silica and potash feldspar begins to melt at 985°C. So the porosity of the white-ware bodies can be decreased by suitable ratio of the liquid (9.5% K_2O, 10.9% Al_2O_3, 79.8% SiO_2) and silica contents (Sallam et. al. [20]).

Important factor that influence the mechanical strength of the sanitary-ware, is the grain size of the filler such as α-quartz. Cracks commonly observed in and around large quartz grains occur because of the large thermal expansion mismatch between the crystalline quartz ($\alpha \approx 23x10^{-6}\ K^{-1}$) and the glassy phase ($\alpha \approx 3x10^{-6}\ K^{-1}$) in the temperature range 20-750°C (Iqbal and Lee [13] cited in Lundin [21]). According to Knudsen relation (Knudsen [22] cited in Dinsdale and Wilkinson, [23]) quantitatively strength of the body is:

$$\log S=KD^{-a}$$

Here S and D are strength and particle diameter and "K" and "a" are constants.This shows that the strength increases as the size decreases. However, as the grain size is reduced, a maximum strength is reached, beyond which further reduction in size is accompanied by a reduction in strength.

Coarse filler forms an unconnected dispersion with intervening smaller clay and flux particles. When flux particles melt, the area of contact between the glassy matrix and alumina particles increases, and the strength increases. When the filler size becomes very small, this situation no longer holds, as the filler may now be more in number to be connected (Dinsdale and Wilkinson, [23]).

The bending strength of porcelain can be increased by decreasing the size of α-quartz particles to 10-30 μm (Bradi [24]).

3. EXPERIMENTAL PROCEDURE

A local factory provided sanitary-ware samples. For XRD, powder (sieved < 325 mesh) were scanned from $2\theta = 10^o \rightarrow 60^o$ at a scanning speed of 0.02/min, using Geol diffractometer (with CuKα radiation, $\lambda = 0.154838$nm) at 40 KV and 20mA. JCPDS Cards 15-775 (mullite), 33-1161 (quartz), 09-0466 and 20-0554 (feldspar (albite)) and 19-1227 (sanidine) were used to identify crystal phases.

Scanning electron microscopy specimens were polished using 15 and ¼ μm diamond pastes after grinding with silicon carbide powder and water .The polished surfaces were etched for 3 min in 5% HF solution and then gold/carbon coated. A JEOL (JSM-5910) SEM equipped with X-Ray Energy Depressive Spectroscopy (EDX-INCA 200, Oxford Instruments, UK) of analysis range from Boron to Uranium and resolution of 130eV, and SEI (Secondary Electron Images) detector, (Operating at 0.1-30 KV, resolution of 2.3 nm and magnification up to 300,000X) was usedfor chemical analysis and morphology.

4. RESULTS AND DISCUSSION

4.1 X-Ray Diffraction of Fired Samples

XRD data for three samples fired at 1100and 1300^{o}C, and one fired commercially, matched with standardized calculated data of the JCPDS cards revealed mainly mullite and α-quartz phases for specified firing range. Additionally, XRD results for a sample collected from the industry (fired commercially) showed corundum phase.

The inter-planar spacings "d" corresponding to the major XRD peaks recorded for the sanitary-ware sample fired at 1100^{o}C **(Fig. 1)** matched with the d-values for 15-775 (mullite), 33-1161 (quartz) and 09-0466 and 20-0554 (feldspar (albite)), however some of the minor peaks matched with the JCPDS cards for 19-1227 sanidine. Iqbal and Lee [13] reported formation of meta-stable sanidine in porcelain in temperature range 600-900^{o}C beyond which it disappears, while in the mixed alkali feldspars, this temperature range extends from 900-1050^{o}C (Mackenzie and Smith, [25]). So at 1100^{o}C, the presence of sanidine can be expected.

The X-ray peak positions and intensities observed for samples fired commercially and for the one fired at 1100^{o}C (Fig. 1) are nearly the same, indicating that the firing temperature of the commercially fired sample lies in the range of 1050-1150^{o}C. Estimating on the basis of peak intensities for mullite, both the samples appeared to contain approximately similar amount of mullite. This suggests that the raw materials, composition and firing temperatures of the two bodies were nearly similar.

Fig. 2 shows a comparison of XRD traces observed for sanitary-ware samples fired at 1100 and 1300^{o}C for two hours. Amorphous humps, although small, in the XRD

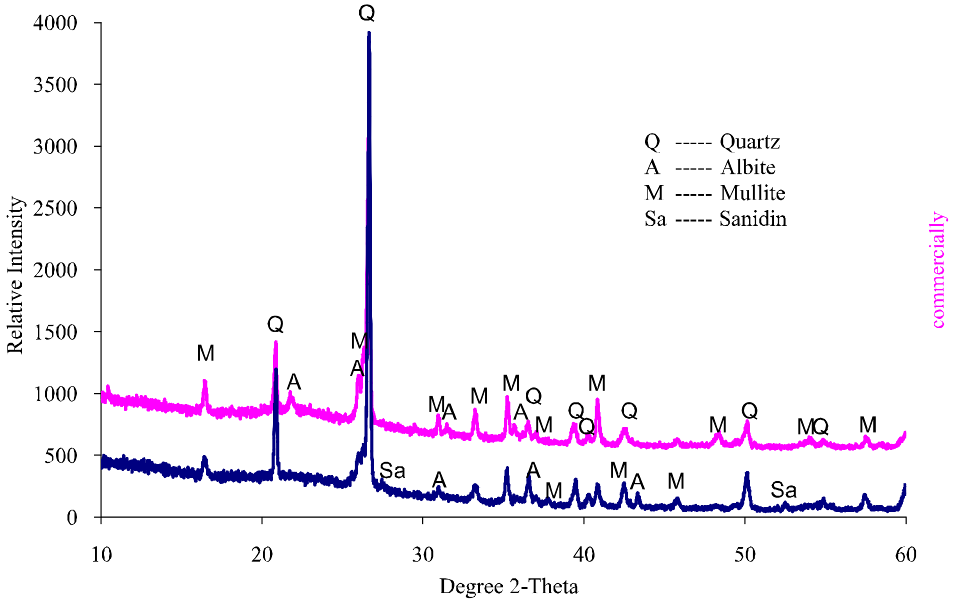


FIG. 1. COMPARISON OF XRD PATTERNS OF SANITARY-WARE SAMPLES FIRED COMMERCIALLY AND AT 1100^{O}C

pattern indicate the presence of amorphous material in the body. The decrease in the intensities of XRD peaks due to α-quartz in the sample fired at 1300°C in comparison to the one fired at 1100°C indicates the beginning of dissolution of α-quartz at temperatures >1200°C. At 1300°C, the intensities of XRD peaks due to Mullite have increased, indicating an increase in the quantity of Mullite. Furthermore, peaks due to mullite are more pronounced and sharp in comparison to those observed for the sample fired at 1100°C. This indicates that the mullite crystals have also grown in size consistent with the previous studies (Iqbal and Lee [13] and Dinsdale and Wilkinson, [23]).

Albite ($NaAl\ Si_3O_8$) phase is present due to the sodium feldspar present in the raw materials used and sanidine $(K,Na)(Si_3Al)O_8$ results from combination of Albite with potassium present in clay/feldspar.

Feldspar melts at 1100°C (Schramm and Hall, [11]) i.e. nepheline and sanidine have been reported to dissolve at temperature <1100°C (Iqbal and Lee[13]), however, albite partially dissolves at temperature slightly above 1100°C (McConvile and Caspar [26]).

The current investigation of the microstructure has consistency with previous results in case of sanidine and albite. These two phases were present at 1100°C. XRD at 1300°C (Fig. 2) also detected peaks of albite that indicated the presence of feldspar at high temperature.

4.2 SEM and EDS Analysis

SEM (Scanning Electron Microscopy) was used to investigate the microstructure of samples fired at 1100, 1200 and 1300°C. A randomly collected sample fired commercially was taken for comparison purpose. Samples for SEM were etched with 5% HF aqueous solution for various lengths of time, however, the resolution of micro-structural features in samples etched for 3 minutes was good and were therefore, included in this thesis. Secondary electron imaging and x-ray electron EDS was used for micro-structural and chemical analyses respectively.

4.3 Samples Fired at 1100°C and Commercially Fired

Micro-structural and elemental analyses were carried out using EDS and SEM. The compositions of sample fired at

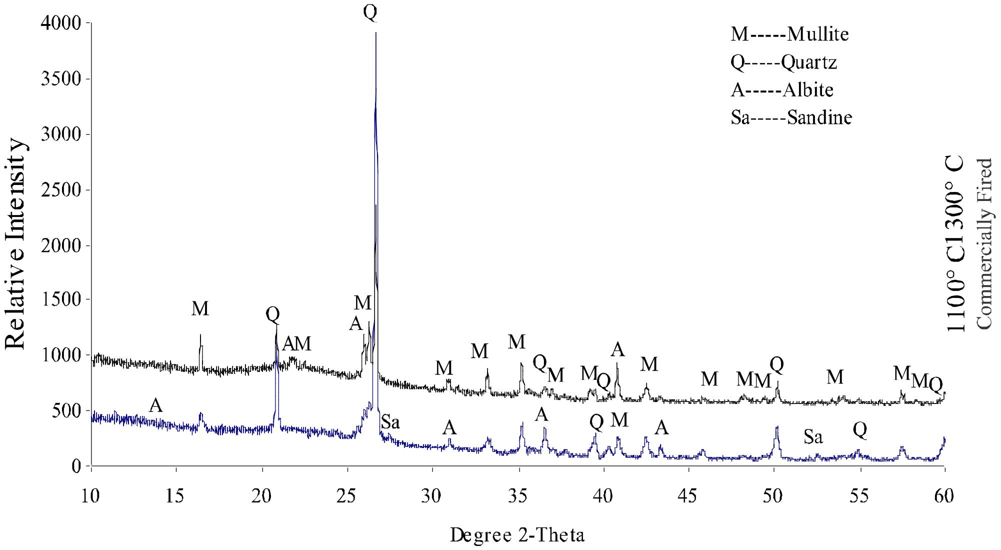


FIG. 2. XRD PATTERN OF SANITARY-WARE SAMPLES FIRED AT 1100 AND 1300°C

1100^{o}C and one fired commercially as confirmed by EDS are given in Tables 1-2.

The commercial sample provided by the local ceramic factory contained 7 Weight (%) quartz, 27 Weight (%) feldspar and 66 Weight (%) clay [Sample provider, 2004]. Soda feldspar is used to lower the firing temperature (Iqbal and Lee [13]). Fig. 3 is a SEIwith marked EDS result from a general area of the sample fired at 1100°C. α-Quartz grains are not clearly visible in this sample because its melting begins at high temperatures i.e. >1200°C (Kobyashiet. al. [27]). In samples fired at 1100°C, the solution rims around α-quartz grains coming from their partial dissolution are absent and therefore, the grains cannot be resolved from the matrix and will require compositional analysis to establish their identity.

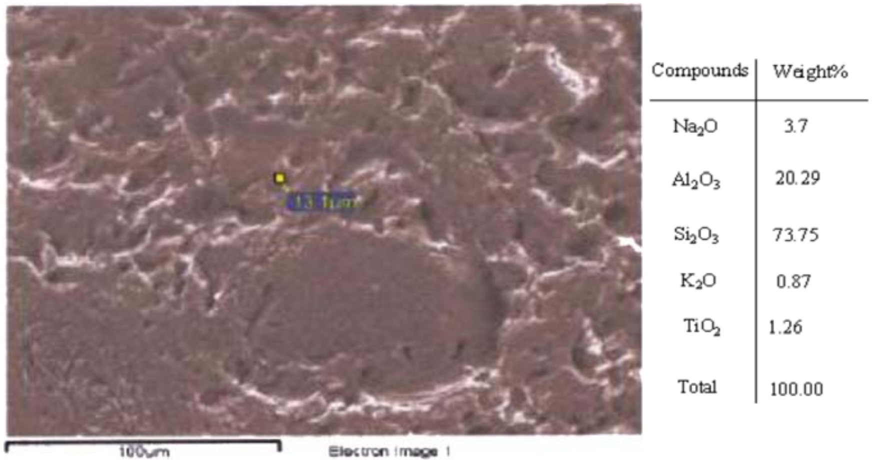
TABLE 1. OXIDES OF SAMPLE HEATED COMMERCIALLY

Compounds	Weight (%)
MgO	2.03
Al_2O_3	22.42
SiO_2	42.47
K_2O	0.96
TiO_2	0.97
Fe_2O_3	31.12
Total	100.00

TABLE 2. OXIDES OF SAMPLE HEATED AT 1100^{O}C

Compounds	Weight (%)
Na_2O	2.06
Al_2O_3	22.41
SiO_2	42.46
K_2O	0.965
TiO_2	0.977
Fe_2O_3	31.11
Total	100.00

Fig. 4 with EDS spectra show the micro-structural features and the compositions of labeled regions/grains confirming the presence of α-quartz grain. At higher temperatures (>1200°C), amorphous silica rich solution rims surround ǻ-quartz due to partial dissolution of α-quartz ([Iqbal and Lee [13]). Silica surrounding the α-quartz grains is removed by etching and therefore, α-quartz grain can be resolved from the surrounding matrix. Secondary mullite is not observable at 1100°C because formation of secondary mullite starts when the matrix gets fluid enough (>1100^{o}C) to allow mullite growth into feldspar relicts. So at 1100^{o}C body's mechanical strength is lower than at higher temperature ([Maiti and Kumar [15]).



Compounds	Weight%
Na_2O	3.7
Al_2O_3	20.29
Si_2O_3	73.75
K_2O	0.87
TiO_2	1.26
Total	100.00

FIG. 3. SECONDARY ELECTRON IMAGEFROM A GENERAL AREA OF THE SAMPLE FIRED AT 1100°C. AREA SCANNED OVER 13.1μm COMPRISES SILICA AND ALUMINA IN LARGE AMOUNT AND LESS AMOUNT OF SODIUM AND POTASSIUM WITH SOME IMPURITIES OF TITANIUM

Regions containing Primary mullite of scaly appearance are shown in Fig. 5. This is consistent with previous studies performed at temperatures close to or at 1100°C ([Lee and Iqbal [6] Iqbal and Lee [10,13]). Scaly primary mullite forms in clay relicts at temperatures ranging from 1100-1200°C ([Lee and Iqbal [6]). As mentioned earlier, needles like large secondary mullite crystals are not expected to form at this temperature. Another reason for not observing the secondary mullite may be due to the short interval of firing time. Reasonable amount (31.11 Weight (%)) of iron has also been detected by EDS (Fig. 5).

4.4 Comparison of α-Quartz and Mullite Grains

Partial dissolution of α-quartz begins at temperatures above 1200°C, therefore, α-quartz grain cannot be resolved from the matrix at low temperatures (1100°C).

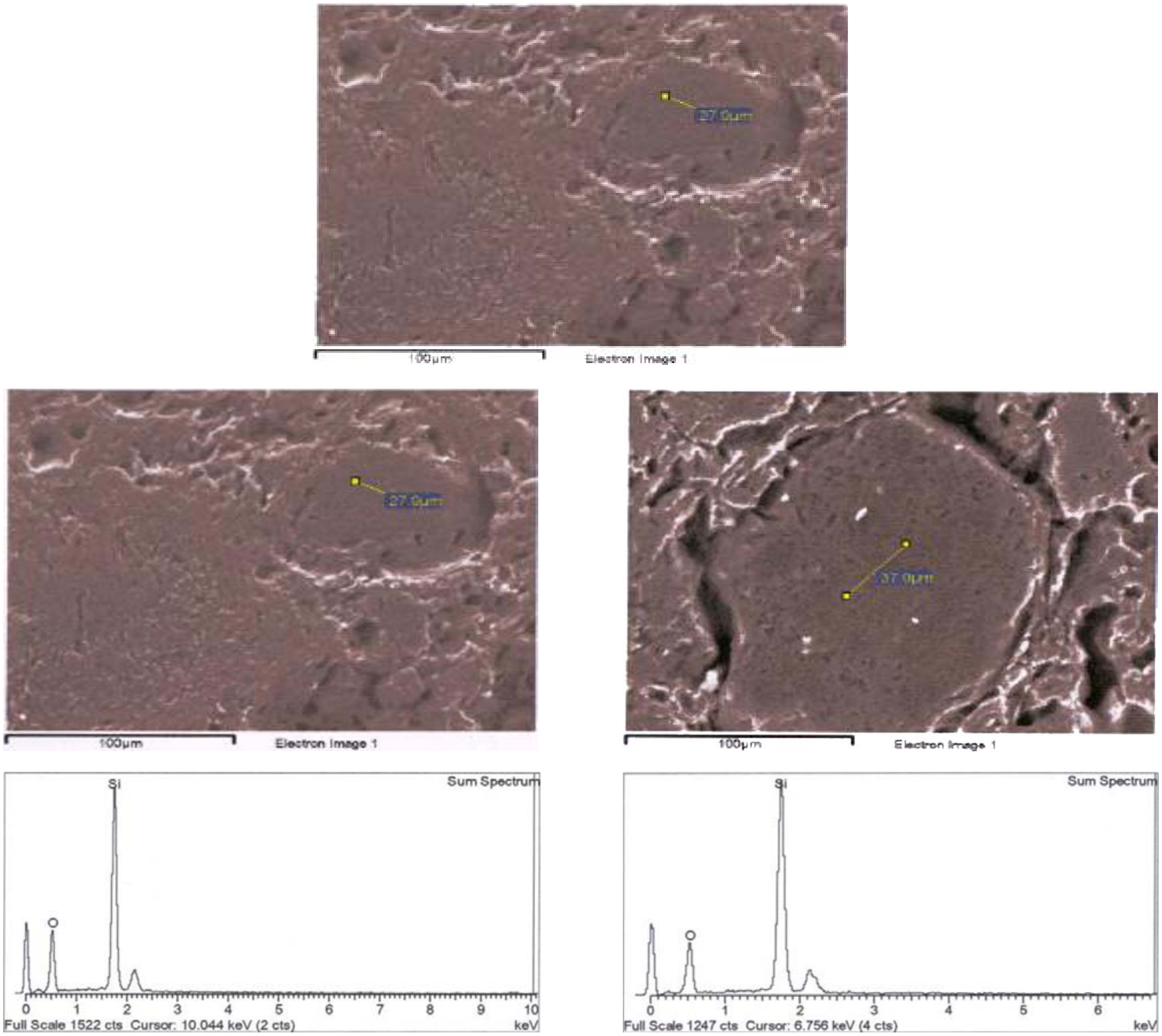


FIG. 4. AREAS SCANNED OVER 27.9 AND 37.0μm COMPRISES 100% SILICAAS CONFIRMED BY IN SET EDS SPECTRA

However, some large sharp-edged α-quartz grains can be identified via EDS at 1100°C (Fig. 6). Corundum grains (Fig. 7) are located adjacent to clay or feldspar relicts, with no solution rim surrounding them. Therefore, at temperatures below 1200°C, EDS is the only technique used in this study to confirm the identification of both quartz and corundum grains, however, at high temperatures (>1200°C), the corundum grains have no silica-rich solution rims around them and quartz grains have a silica-rich solution rim around them ([Iqbal and Lee [13]).

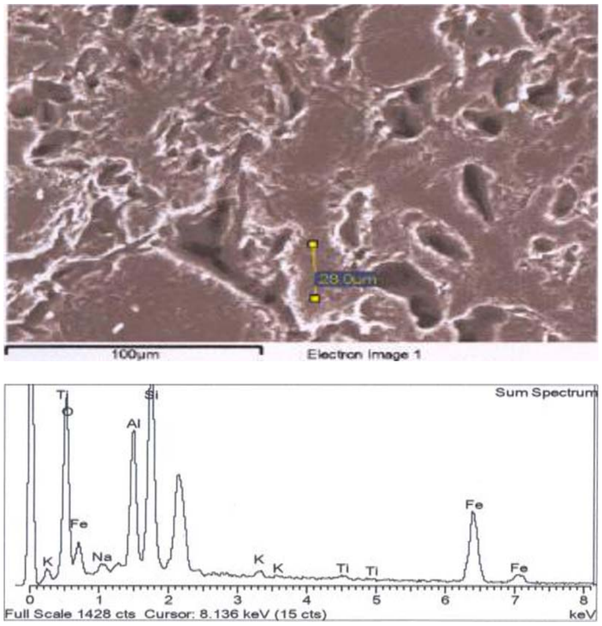


FIG. 5. SHOWS PRIMARY MULLITE OF SCALY APPEARANCE WITH REASONABLE AMOUNT OF IRON

4.5 Sample Heated At 1200°C

SEM-EDS results of the same samples reheated at 1200°C (Fig. 8 with marked EDS result) reveals the presence of silica rich matrix in the vicinity of α-quartz grain "Q" along with some impurity elements as iron, titanium and potassium. Very little amount of sodium was detected probably due to small amount of feldspar used in the initial ingredients of the body. Traces of Fe, Ti, Ca and K were always present in the vicinity of α-quartz grains. Primary mullite was observed as shown by "A, B, C" in Fig. 8(a). High clay relicts, lake of feldspar and improper mixing may be the possible reason for not observing secondary mullites in these samples, because secondary mullite forms in the regions in which flux particles were mixed well with clay or where flux has penetrated clay agglomerates ([Lee and Iqbal [6]). Secondary Mullite crystals originate from the clay relicts and grow into the feldspar relicts. Therefore less amount of feldspar used as body ingredient affect secondary mullite formation ([Iqbal and Lee [10]).

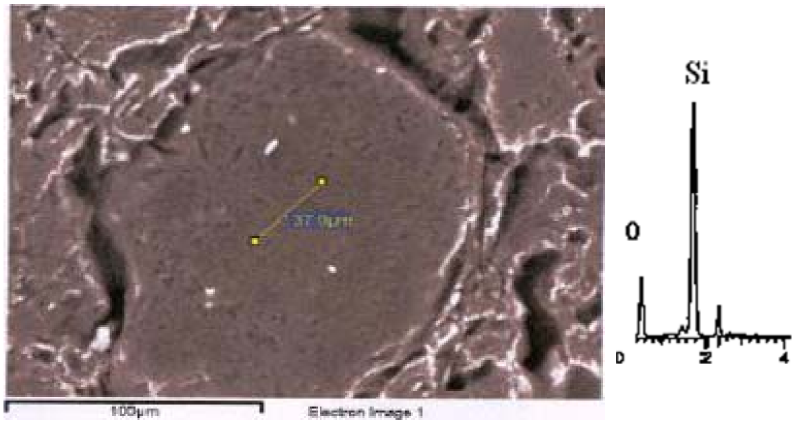


FIG. 6. AREA SCANNED OVER 37.0µm COMPRISES 100% SILICA, CONFIRMED BY INSET EDS

Glaze composition and morphology is shown in Fig. 9. EDS revealed the presence of Barium, Zinc and Zirconium as glaze pigments added to clay for coating the surface of the sanitary ware. A trace amount of Titanium was also detected in the glaze. Note the absence of Fe in the glaze composition that may have been carefully removed as it may affect the white colour of the sanitary-ware product.

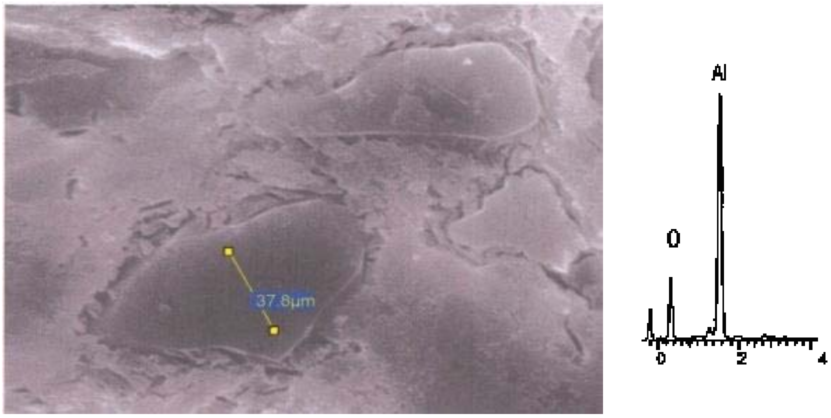
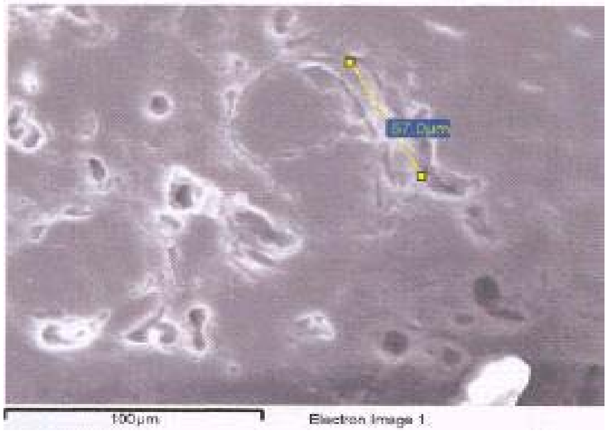
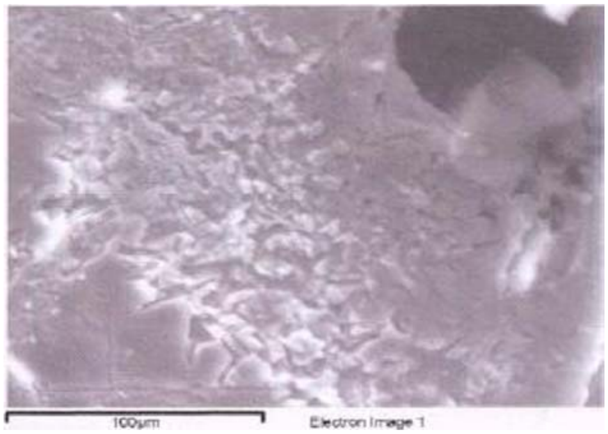


FIG. 7 .REGION SCANNED OVER 37.8µm IS PURELY ALUMINA, INSET EDS CONFIRMS THE CORUNDUM GRAIN



Compounds	Weight%
Na_2O	2.82
Al_2O_3	21.27
SiO_2	71.88
K_2O	1.08
TiO_2	1.32
Fe_2O_3	1.62
Total	100.00

FIG. 8. SEI OF SAMPLE REHEATED AT 1200^{O}C . THE EDS OF THE MARKED REGION REVEALS THE PRESENCE OF SILICA RICH MATRIX IN THE VICINITY OF α-QUARTZ GRAIN "Q" ALONG WITH OTHER IMPURITIES SUCH AS IRON, TITANIUM POTASSIUM. LABELS A, B AND C SHOW PRIMARY MULLITES



Compounds	Weight%
Na_2O	2.24
Al_2O_3	13.36
SiO_2	57.72
CaO	8.23
TiO_2	1.977
ZnO	3.89
ZrO_2	9.49
BaO	3.08
Total	100.00

FIG. 9. GLAZE COMPOSITION AND MORPHOLOGY OF THE SAMPLE FIRED AT 1200^{O}C

4.6 Sample Fired at 1300°C

The microstructure of sample reheated at 1300°C contains elongated needles of secondary mullite (Fig. 10). The high quantity of iron oxide detected (Fig. 10) in these needles makes the precise analysis ambiguous. Crystals of similar morphology observed at temperatures above 1200°C in others studies of vitreous ceramics using pure raw materials are identified as secondary mullite ([Lee and Iqbal [6,13]). The presence of iron oxide may be due to the iron-rich glass covering the crystals. The presence of other elements tabulated in (Fig. 10) also indicates that the SEM probe used for chemical is not precise enough to isolate the crystal from the glassy matrix and peaks due to other elements in the vicinity overlap the peaks from the marked region (Fig. 10).

Fig. 11 shows the denteric growth of mullite in a region, where 50% SiO_2 exists (Table 3). Sodium feldspar conferring the fluidity upon the matrix is generally believed to allow faster mulllite growth than other alkali silicates. In our study, the amount of Na detected in the vicinity of crystal is low enough to enhance mullite growth to the observed size ([Schuller [12] and Kobayashi et. al. [28]). In vitreous ceramics, the mixing of raw materials is always incomplete and the composition changes from region to region which may be the possible reason for enhanced growth rate of mullite in one region and depressed growth rate in the other.

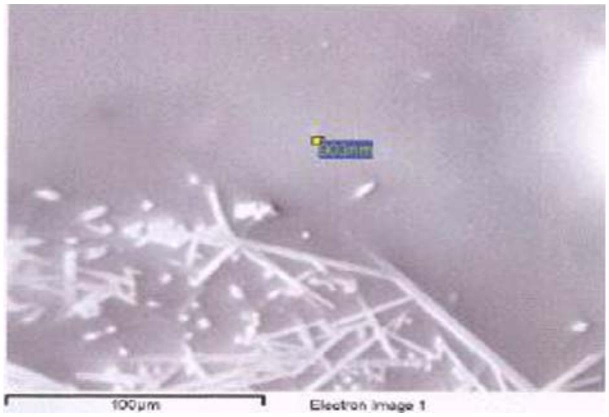
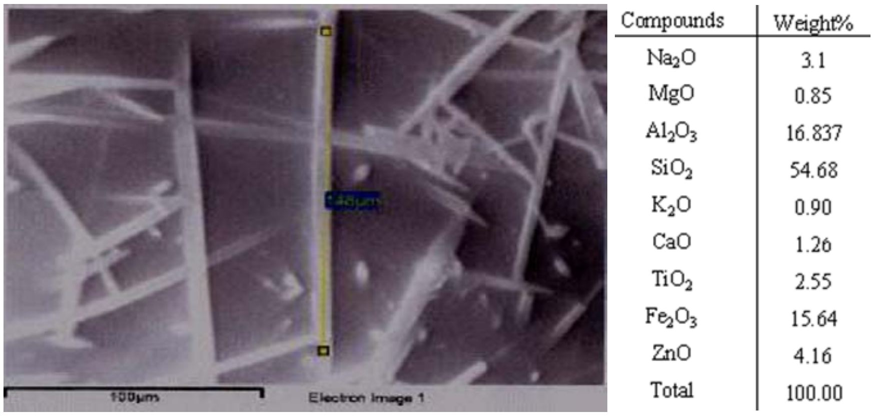


FIG. 11. DENTERIC GROWTH OF MULLITE IN SILICA REGION

TABLE 4. OXIDES OF SAMPLE HEATED AT 1300^{O}C

Compounds	Weight (%)
Na_2O	5.02
Al_2O_3	14.87
SiO_2	50.38
K_2O	0.55
CaO	1.20
TiO_2	2.26
Fe_2O_3	9.95



Compounds	Weight%
Na_2O	3.1
MgO	0.85
Al_2O_3	16.837
SiO_2	54.68
K_2O	0.90
CaO	1.26
TiO_2	2.55
Fe_2O_3	15.64
ZnO	4.16
Total	100.00

FIG. 10. 148µm SCANNED AREA OF THE MULLITE SHOWS SOME OTHER COMPOSITION SUCH AS IRON, Zn, Ca, Ti, Mg, K, WHICH CONTRADICTS THE COMPOSITION ($3Al_2$:$2SiO_2$) OF MULLITE

5. CONCLUSIONS

(i) XRD revealed the presence of α-quartz and mullite as major crystalline phases in all the sanitary ware samples fired at 1100 and 1300°C. A small number of low intensity XRD peaks were also present which could be due to albite and sanidine, however, their peak intensities decreased with increase in temperature.

(ii) Consistent with our XRD analysis, SEM also revealed the presence of Mullite and α-quartz in the investigated samples.

(iii) Consistent with previous studies, SEM revealed the presence of two types of mullite in the investigated samples. Only Primary mullite was present in samples fired at 1100°C, and both primary and secondary mullite were present in samples fired at 1200 and 1300°C.

(iv) One of the samples also contained corundum grains but the absence of XRD peaks due to corundum from the relevant spectra reveal that the percentage of corundum added was too low to be detected with XRD.

(v) The level of impurities such as titanium and iron in the raw materials used was much higher than those used in the previous studies.

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