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Effects of the Sintering Process on Properties of Triaxial Electrical Porcelain from Ugandan Ceramic Minerals

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***Abstract*—**Porcelain specimens were fired at 6°C/min to 1250°C (dwell time 0.5-3h) and cooled at 6°C/min to room temperature. Additionally, three different slower firing/cooling cycles were tried. Sintering profile and effects on MOR, crystalline phase content and morphology were investigated using dilatometry, 4-point bending strength, XRD and FEG-SEM respectively. Industrial-sized specimens prepared using the promising cycle were tested basing on the ANSI standards. Increasing dwell time from 1h to 3h at peak temperature of 1250°C resulted in neither a significant effect on the quartz and mullite content nor MOR. Reducing the firing/cooling rate to below 6°C/min, for peak temperature of 1250°C (dwell time of 1h) does not result in improvement of strength of porcelain. The industrial sized specimen exhibited flashover voltages of 20.3kV (dry) and 9.3kV (wet) respectively, transverse strength of 12.5kN and bulk density of 2.27g/cm^3, which are satisfactory. There was however dye penetration during porosity test.

***Keywords*—**Dwell time, Microstructure, Porcelain, Strength.

I. Introduction

TRIAXIAL porcelains are primarily composed of clay, feldspar and a filler material, usually quartz or alumina. The clay gives plasticity to the ceramic mixture, quartz maintains the shape of the formed article during firing, and feldspar serves as flux. The three constituents place electrical porcelain in the phase system [$(K,Na)_2O$-Al_2O_3-SiO_2)] in terms of oxide constituents, hence the term triaxial porcelains [1]. The fired porcelain product contains mullite ($Al_6Si_2O_{13}$) and undissolved quartz (SiO_2) crystals embedded in a continuous glassy phase, originating from feldspar and other low melting impurities in the raw materials.

The effects of the individual phases on the mechanical properties have been elucidated under the mullite hypothesis, the matrix reinforcement and the dispersion strengthening hypotheses [2], [3]. Notably also, in most efforts to increase strength in porcelains, emphasis has been placed on minimization of quartz in the porcelain formula because of the β to α phase inversion of quartz which occurs at 573°C during cooling. The inversion results into decrease of quartz particle volume and may lead to cracks in the body. So far, there are successful studies on improvements in mechanical properties of porcelains by reducing/eliminating the use of quartz. These include replacements of quartz with kyanite [4], alumina [5], [6], rice husk ash [7], sillimanite sand [8], fly ash [9], silica fume [10], partial replacement of feldspar and quartz by fly ash and blast furnace slag [11], with a mixture of rice husk ash and silica fume [12].

On the other hand, there is evidence that under optimized conditions of firing and for a particle size of 10-30μm [13]-[15], quartz has a beneficial effect on the strength of porcelain, in conformity with the pre-stressing theory. For small particle sizes, the dissolution is more rapid leaving less quartz crystals in the glass and hence yielding a low pre-stress and low strength of the material. For large quartz particle sizes an interconnected matrix with favourable crack path is formed leading to low strength [3]. Hence, quartz grain size affects bending strength in two ways, that is, directly through the induction of compressive stresses to the vitreous phase and indirectly through the development of a microstructure with favorable strengthening effect for small enough grains [16]. Carty and Senapati [3] concluded that the typical strength controlling factors in multiphase polycrystalline ceramics are thermal expansion coefficients of the phases, elastic properties of the phases, volume fraction of different phases, particle size of the crystalline phases and phase transformations.

Recent studies on improvement of the mechanical and dielectric strengths have shown that each phase has its specific influence on the mechanical and dielectric properties of the body depending on its concentration and microstructural attributes [17]. Microstructural attributes and concentration of the phases are largely influenced by temperature and compositional differences [18]-[19]. Earlier investigations presented in Olupot *et al* [20] reveal that the proportions of these phases and the resultant properties are dependent on firing temperature for a given composition.

In this work, the firing process of a porcelain composition was investigated by continuously following the shrinkage with time and temperature in a dilatometer. Sintering was done at different holding times at a peak temperature of

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1250°C followed by investigations into the resulting modulus of rupture (MOR) and microstructure. The progress of phase changes under different soaking intervals was investigated in relation to the resultant properties in the formed porcelain products. Additional efforts were made to alter the sintering curves and the microstructures and strength of the resulting porcelains were investigated. It is clear from the results of this work that variation of soaking time after 1h holding does not influence the shape and content of the crystalline phases in the porcelain, neither does it have significant influence on the modulus of rupture of porcelains.

II. Raw Materials, Sample Preparation and Experimental Techniques

A. Raw Materials

The raw materials used were ball clay from Mukono, kaolin and feldspar from Mutaka and sand from Lido beach on the shores of Lake Victoria in Entebbe, Uganda. These deposits and their locations are described elsewhere [21], [22]. Representative samples were collected from the deposits and processed for use in the study. The materials were each separately wet-milled, run over a magnetic iron separating tray to remove iron contamination and sieved through aperture sizes of 45μm for kaolin and ball clay, 53μm for feldspar and 25μm for sand. They were subsequently dried with the exception of ball clay which was mixed as slip in the composition. The dry content of the ball clay in the slurry was calculated using Brogniart´s formula [23].

B. Sample Preparation

1. Formulation of Porcelain Specimens

A porcelain body with a composition of 27% kaolin, 13% ball clay, 40% feldspar and 20% sand was used, basing on results from previous studies [24]. Slurry comprising of the respective raw materials was prepared and milled for 3h in a ball mill using a quartz grinding medium to ensure homogeneous mixing. The slip was filter pressed and left to dry at room temperature to form a paste of sufficient plasticity for extrusion through a de-airing pug mill, producing cylindrical specimens of 100mm in diameter and cut to 100mm length. Some of the extruded cylinders were dried in air at room temperature, while others were used as starting blocks from which smaller cylindrical specimens of 10mm diameter by 80mm of length were extruded using cork borers. These smaller specimens were used for mechanical strength tests and other tests as will be explained below.

2. Specimens for Mechanical Testing and Microstructure Analyses

The extruded specimens of 10 mm diameter by 80 mm length were, first dried at room temperature, followed by drying at 110°C for 24h. The dried samples were split in five batches of 25 specimens each. The different batches were each separately fired to 1250°C at a heating rate of 6°C/min, soaked at the peak temperature for different intervals of time in the range of 1h to 3h, and finally cooled at a rate of 6°C/min to room temperature. The specimens prepared in this way were used for testing the bending strength and for characterizing the microstructure of the resultant porcelains. They are referred to as P1 to P5 respectively in the following sections. Additionally, four more sintering curves were adopted viz:

1. Heating at 6°C/min for 25-1250°C, 30min dwell at 1250°C and cooling at 6°C/min for 1250-25°C.
2. Heating at 6°C/min for 25-400°C, 3°C/min for 400-1250°C, 1h dwell at 1250°C and cooling at 3°C/min for 1250-400°C followed by 6°C/min for of 400-25°C.
3. Heating at 3°C/min for 25-1250°C, 1h dwell at 1250°C and cooling at 3°C/min for 1250-25°C.
4. Heating at 6°C/min for 25-500°C, 1°C/min for 500-700°C, 6°C/min for 700-1250°C, 1h dwell at 1250°C and cooling at 6°C/min for 1250-700°C, 1°C/min for 700-500°C followed by 6°C/min for 500-25°C.

The samples made by the latter sintering curves are designated S1 to S4.

3. Specimens for Dilatometry Tests

Some of the dried porcelain specimens described in paragraph 2.2.1 were cut into smaller pieces of approximately 15-25mm length and sintered in a dilatometer. In the dilatometer, the specimens were held on a refractory support and fired at 6°C/min up to 1250°C, soaked for different periods in the range of 1-3h and allowed to cool in the dilatometer at 6°C/min down to room temperature.

4. Specimens for Industrial Type Tests

Upon evaluation of the properties of samples produced based on the firing cycle described in 2.2.2 above, specimens made from dried samples previously extruded to 100mm diameter by 100mm length were shaped by drilling and machining to industrial dimensions shown in Fig. 1. These samples were subsequently dried at 110°C for 24h, fired to 850°C at a rate of 6°C/min, soaked for 1h and cooled to room temperature at a rate of 6°C/min. These biscuit fired specimens were later glazed by first dipping them in water and then in glaze slip. After drying, the glazed specimens were fired at 6°C/min up to 1250°C and soaked for 2h before cooling to room temperature at 6°C/min.

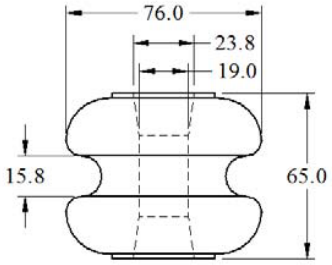

Fig. 1 Industrial size specimen of a low tension shackle insulator

5. Glaze Preparation

The glaze was prepared by mixing the dry processed sand, kaolin and feldspar described in paragraph 2.1 and adding a

low-melting oxide component, CaO, in the form of whiting ($CaCO_3$) supplied by Mineral Enterprises (Kenya). Approximately 2kg of glaze was prepared by crushing the mixture of powders using a small ball mill. The glaze slip was prepared by adding water and thoroughly stirring to ensure a homogeneous mixture. The Seger formula of the glaze used was $RO:0.57Al_2O_3:4.86SiO_2$, basing on the results of an earlier study [25].

C. Experimental Techniques

The chemical composition was determined by Analytica AB, Luleå, Sweden according to their standard analysis package G-2 [26]. The powders were first dried at 105°C according to Swedish standard SS 028113. 0.125g dried powder was then smelted with 0.375g $LiBO_2$ and dissolved in 5% nitric acid before the metal content was determined using inductively coupled plasma atomic emission spectrometry. The loss on ignition (LOI) was determined by heating to 1000°C for 2h.

Samples were examined with respect to length changes in a Netzsch Dil 402C dilatometer operated under Ar gas and a preload of 10cN to hold the samples. In all cases the heating and cooling rates were set to 6°C/min and the dwell time at the peak temperature were set to 1h, 1.5h, 2h, 2.5h and 3h respectively. Unglazed specimens fired at different dwell times were tested in 4-point bending fixture using a Testometric universal testing machine, model M500. The crosshead speed was 1mm/min and the spans between the upper and lower supports were 20 and 40mm, respectively. A minimum of 15 specimens from each sample batch were tested. Using Weibull statistics, described elsewhere [20], MOR, was evaluated for a fracture probability of 0.632.

The microstructures of the specimens were characterized using a FEG-SEM instrument, LEO 1530 with a GEMINI column. Sectioned and polished specimens of the extruded and fired cylinders were used for the investigation. The specimens were polished, cleaned and dried. Thereafter, they were dipped in 40% concentrated hydrofluoric acid for 25s, cleaned, dried and studied using FEG-SEM instrument. The cleaning procedures given to all specimens before examination included washing in water and alcohol before drying. The crystalline phases in the fired porcelain samples were determined by X-ray diffraction using an X'pertPRO PANalytical X-ray diffractometer, PW 3050/60, with Ni-filtered Kα Cu-radiation generated by a 40kV acceleration voltage and a 40mA anode current. Pulverized specimens were scanned from 10 to 75° 2θ operating the equipment at a 2θ scan-speed of 0.5 sec/step and a 2θ step size of 0.02°. The X-ray peaks of the different phases were identified with the software of the equipment.

Industrial type tests were performed under specified conditions as detailed in ANSI C29.5-1984 and ANSI C29.1-1988(R2002) standards for wet-processed porcelain insulators. The tests carried out include mechanical failing load, dry and wet power frequency voltage flashover, bulk density and porosity tests.

III. Results

A. Chemical Composition of the Porcelain Body and Raw Materials

The chemical composition in terms of the percentages of the major minerals in the formulated porcelain as well as the individual materials is as shown in Table I.

TABLE I
CHEMICAL COMPOSITION (WT %)

	Sand	Feldspar	Kaolin	Whiting	Porcelain body
SiO_2	97.00	68.10	57.10	4.74	70.90
Al_2O_3	0.22	18.70	30.20	0.18	18.00
CaO	<0.10	0.38	0.19	34.60	0.35
Fe_2O_3	0.09	0.08	0.32	0.10	0.78
K_2O	0.11	9.55	2.36	<0.06	4.28
MgO	<0.02	0.18	0.18	19.60	0.20
MnO	<0.01	<0.01	0.01	0.01	0.01
Na_2O	<0.05	0.36	0.12	<0.05	0.20
P_2O_5	0.01	0.09	0.04	0.08	0.06
TiO_2	0.08	0.01	0.01	0.01	0.37
LOI	0.50	3.50	10.30	42.30	6.10

B. Dilatometry and Differential Scanning Calorimetry Tests

The change in lengths of the specimens as firing and cooling progressed for the various soaking time intervals from 1-3h are as shown in

Fig. 2. The figures are identical, with the exception of additional shrinkage occasioned by holding for different time durations. This suggests that for the firing schedule used, all transformations that could take place, had already taken place in the first 1h of soaking. Any further soaking did not have any further effect on the sintering curve. A typical DSC curve for this porcelain soaked at 1250°C for 2h is shown in Fig. 3, clearly showing the α- to β- quartz transition at about 573°C and the corresponding transformation at about 980°C corresponding to the spinel phase formation preceding mullite formation.

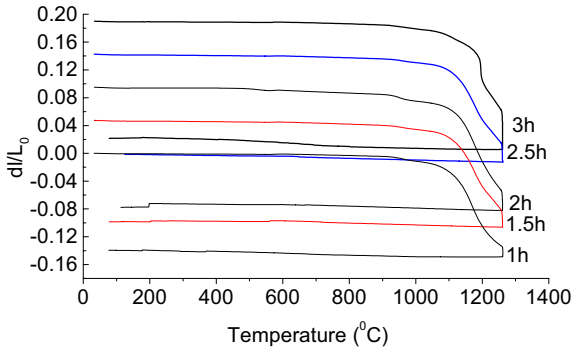

Fig. 2 Sintering profiles in the dilatometer, the graphs have been offset from each other for clarity

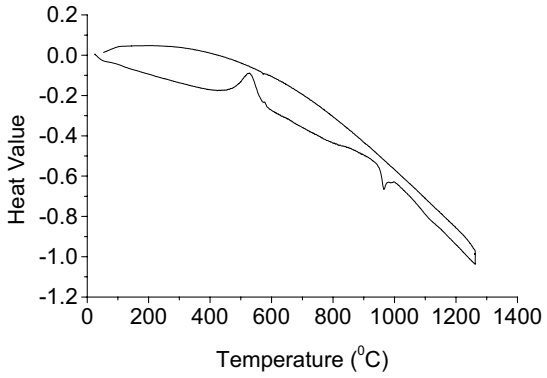

Fig. 3 Differential scanning calorimetric curve

C. Bending Strengths of Samples

A sample of the Weibull plots for the specimens is given in Fig. 4. As can be noted from the summary data given in Table II, changing dwell time at the peak temperature from 1h to 3h resulted in marginal variation in strength of the porcelain samples. Due to this effect, the subsequent procedure used to make final samples used a dwell time of 2h since this showed the lowest degree of variation in the results, as can be seen from the scatter of the data in Fig. 4 (for sample P3), represented by the gradient in Table II for other samples, but also from earlier works by Olupot et al [25], the chosen glaze matured well after 2h dwell using the same firing cycle. In addition, the supplementary firing schedules adopted for samples S1 to S4 did not result in any significant improvement of the bending strength of the formulated porcelain specimens, as can be noted from Table II.

TABLE II
MOR OBTAINED FROM WEIBULL STATISTICS FOR A FRACTURE PROBABILITY OF 0.632

Sample	Dwell time (h)	MOR (MPa)	Curve gradient
S1	0.5	48.68	4.88
P1	1.0	60.23	4.38
P2	1.5	51.04	3.90
P3	2.0	58.93	4.81
P4	2.5	53.70	3.98
P5	3.0	58.46	3.05
S2	1.0	54.82	4.58
S3	1.0	57.39	4.31
S4	1.0	51.93	3.48

D. Microstructure Analyses

All specimens consist of quartz and mullite as the major crystalline phases as shown in Fig. 5 and Fig. 6, together with vitreous phase and pores (Fig. 7). The intensity of the quartz peaks does not have any marked relationship with the dwell time of the specimens at the peak temperature (Fig. 6).

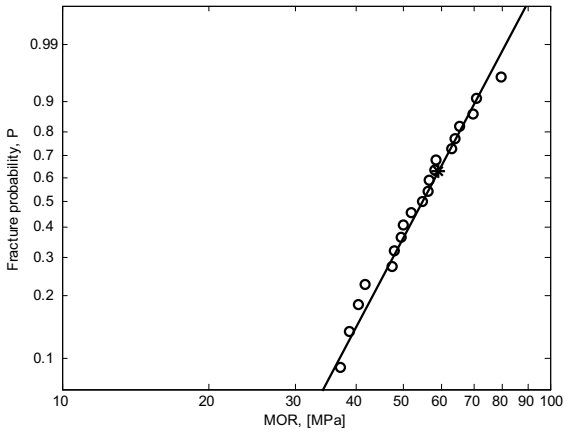

Fig. 4 Weibull plot for sample P3

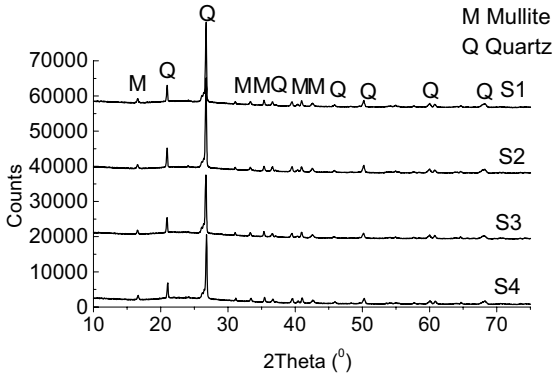

Fig. 5 Powder diffractograms of samples sintered using different sintering curves. For clarity, the curves are shifted vertically by equal proportions from each other

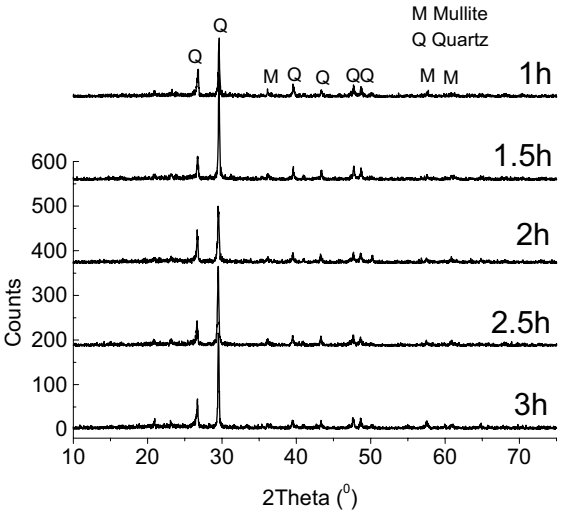

Fig. 6 Powder diffractograms of samples soaked for different times at the peak temperature of 1250°C, using the same heating/cooling rates. For clarity, the curves are shifted vertically by equal proportions from each other

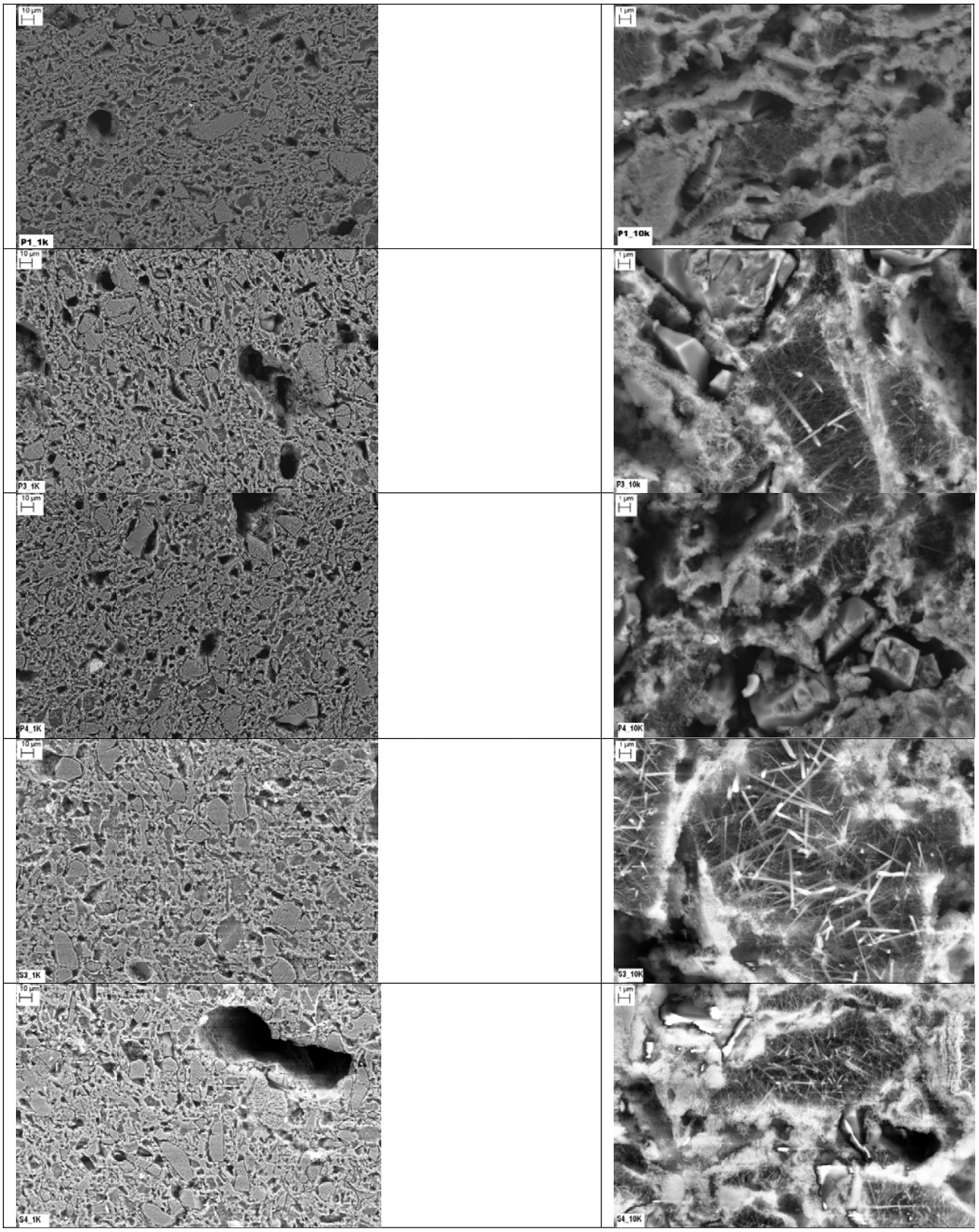

Fig. 7 FEG SEM for samples P1, P3 & P4 and S3-S4 at x1000 (Left) and x10000 (Right), respectively

E. Industrial Type Tests

These tests were conducted according to ANSI C29-1&3 for visual inspection, dry power frequency flash over test, wet power frequency flashover test, transverse strength test, porosity test and bulk density tests. All fragments from the transverse load test showed dye penetration upon subjection to porosity tests and were considered porous according to the specifications of the standard used. Despite the poor porosity, the properties listed in Table III are considered satisfactory for the specimens produced.

TABLE III
TYPE TEST RESULTS

Test Type	Test Value
Dry power frequency voltage flashover (kV)	20.3
Wet power frequency voltage flashover (kV)	9.3
Transverse strength (kN)	12.5
Bulk density (g/cm^3)	2.27

IV. DISCUSSIONS

The porcelain raw material used consists of kaolin, ball clay, feldspar, quartz, dispersed in water. The chemical composition was the same for all the porcelain specimens used in the study. Sintered samples consist of quartz, mullite and glassy phase. Dilatometer plots reveal identical sintering characteristics for all samples. The XRD diagrams (Fig. 5 & 6) show almost no difference in the crystalline phases generated in the samples at different dwell durations at the peak temperature. This illustrates that increasing the dwell time at the same sintering temperature has no effect on the amount of the crystalline phases that exist in porcelain bodies provided an appropriate heating and cooling rate is achieved. This is in agreement with the assertion that the amount of glass formed during firing is determined by the amount of flux and the soak temperature during heat treatment and becomes constant once steady conditions have been reached [27]. As can be seen from these results, altering the sintering curves did not result in any marked changes both in MOR, XRD profile and SEM micrographs. Earlier investigations [20] demonstrated to the contrary that varying the sintering temperature results into significant variation in the crystalline phase content, as well as mechanical properties for the same composition. Sintering temperature of 1250°C proved the best temperature for firing the porcelain from Ugandan minerals based on the sintering curve used for samples P1 to P5.

The highest MOR values were obtained after 1h soak at the sintering temperature as indicated in Table II. This demonstrates that holding for further time after 1h soaking and at 1250°C does not produce any changes in the crystalline features of the microstructure of the specimens as can be seen from FEG-SEM micrographs of the etched surfaces in Fig. 7. Holding for longer intervals does not influence the shape of the resultant microstructural features generated in the microstructure. This is clearly shown by the micrographs and XRD plots in Figs.5&6. The size and shape of mullite and quartz crystals are similar. The effect of the rate of sintering on the mullite morphology however, cannot be clearly discerned from this study and hence relation of these results with studies reported by Carty and Senapati [3] cannot be drawn.

According to the Weibull plots and the results in Table II, there is no significant variation in the strength of the products as a result of variation in the soaking duration at the sintering temperature. It is clear from Table II that the highest strength resulted from 1h dwell, however the fact that 2h-dwell samples exhibited similar strength values but with a low degree of variability in results as exhibited by the slope of the Weibull plot, and from previous studies where the best glaze matured after a 2h dwell, this dwell time was chosen for making industrial samples. This is in agreement with the fact that no further changes took place and thus it was unexpected for properties to change.

In regard to the industrial sized samples, the test values reported in Table II are considered satisfactory for industrial application for the sample shown in Fig. 1, only that the there was dye penetration in the fractured test fragments. This suggests that the structure is still porous. It is clear from this work that the technical characteristics of mechanical and dielectric strength can be satisfactory when an equally important application requirement of water absorption is below limits. For electrical application, water absorption in the unglazed body must be zero. Further investigations involving material preparation and firing shall be done to ensure that the required dielectric issue of water absorption is attained for successful marketing of this porcelain product and commercial adoption for small scale piloting in the country.

V. CONCLUSION

The present study reveals that increasing dwell time from 1h to 3h at the peak firing temperature of 1250°C has no effect on the crystalline phase content of the resultant porcelain body heated at a rate of 6°C/min and cooled at a rate of 6°C/min. This in effect does not cause any significant variation in the mechanical strength of the formed porcelain. A dwell time of 30min however results in low mechanical strength. Reducing the firing rate to less than 6°C/min, provided the peak temperature of 1250°C and a dwell time of 1h is used does not result in any significant improvement of strength of porcelain. In the firing regime adopted, the effect of firing rate, and dwell time at the peak temperature on the mullite morphology is not evident. Attempts to produce industrial sized specimen based on this sintering profile have demonstrated that good strength, both mechanical and dielectric can be attained, yet the porcelain body could still absorb moisture.

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