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Effect of Sintering Condition and Temperature on the Characteristics of Porcelain Rich in Alumina

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Abstract

The elemental composition of clays, feldspar, and quartz and mechanical properties of washed clays were determined by Energy Dispersive Fluorescence (ED-XRF) method. Six different body mixtures with varying weights of added alumina (Al_2O_3) were prepared and sintered at 1230°C under reduction condition and also at 1300°C under oxidation condition. In this research work, an investigation was done on the difference between the effect of sintering under reduced atmosphere and the real atmosphere. The differentiation between strength of glazed and unglazed bodies were also studied. Their physico-chemical properties (color, shrinkage, bulk density, water absorption, and apparent porosity), mechanical properties (bending strength as modulus of rupture), thermal property (thermal conductivity), and electrical properties (electrical resistivity, dielectric constant, and dielectric loss) were determined. The processed electrical porcelains, G1, G2 and I2 have potential to use as insulators where high mechanical strength is a criterion.

Key words : porcelain, body mixtures, alumina, kiln, mechanical properties

Introduction

Porcelain is a ceramic material made by heating raw materials, in a kiln to temperatures between 1,200 °C (2,192 °F) and 1,400 °C (2,552 °F). Porcelain possesses all the major characteristics of technical ceramics. The more demands on ceramic components for electric power generation and distribution systems and mechanical engineering uses, the better material properties and the development of new manufacturing methods. The proportions of the raw materials vary over an extremely wide range depending upon the type of body and the product being made.

The chemistry of the atmosphere inside a kiln has a marked influence on the color and surface of the finished product and effects achieved by depriving the kiln atmosphere of oxygen – known generally as reduction firing – have been very popular with non-industrial for many years. Reducing atmospheres are easily achieved by increasing the supply of fuel and at the same time reducing the supply of air so that the hot

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vaporized carbon and hydrogen cannot obtain sufficient atmospheric oxygen for combination. In these circumstances, the oxygen-hungry gases attack any exposed particles of oxides in the clay or glaze, taking away some or all of the oxygen contained in their molecules and leaving them reduced. Controlled reduction is usually commenced at about 1050°C, well before the glazes have begun to fuse, so that the iron oxide throughout much of the thickness of the clay and the glaze powder can be changed by the gases. In the high-voltage system, the most used insulator material is porcelain and glass due to its dielectric characteristic. Insulators made from porcelain rich in alumina are used where high mechanical strength is a criterion. (Green, 1979)

Hence the objective of present study was to obtain high-tension electrical porcelain bodies having high mechanical strength with good electrical properties.

Materials and Methods

Materials

Locally available clay materials: ball clay (Taungnaut) from Kyaukpadaung Township, Mandalay Region, ball clay (Yankintaung) from Minhla Township, Bago Region, china clay (Kanpauk) from Kyaukpadaung Township, Mandalay Region, china clay (Shwetaung) from Shwetaung Township, Bago Region, and china clay (Yozayat) from Pakoku Township, Magwe Region were used for the preparation of porcelain. Feldspar from Tharzi Township, Mandalay Region was used as a flux material and quartz (Myeik) from Tanintharyi Region was used as filler. Germany made Al_2O_3 was used to get high alumina body mixtures. Besides these, some local raw materials such as dolomite, calcite, barium carbonate and China raw materials Al_2O_3, MnO_2, Cr_2O_3 and Fe_2O_3 were used for glaze slip.

Methods

Mineralogical Analysis of Raw Materials

The ED-XRF (Energy Dispersive X-ray Fluorescence Spectrometer, SFA-2220 A ID-1364) was used for determination of chemical composition of raw materials.

Mechanical Properties of Clays

Automatic Modulus of Rupture Machine was used to find out the breaking load of porcelain body as well as the calculated dry Modulus of Rupture (MOR) directly.

Preparation of Test Circular Bars

Slip for various bodies was prepared with composition as described in Table (1). 25% by weight of water was added to form the required consistency that was obtained after operating the pot mill for 8 hours. The slip obtained was poured into POP moulds to get round shaped test bars And dried at 110ºC for 12 hours.

Preparation of Glaze Slip

Glaze was prepared according to the composition as shown in Table (2). The glaze was obtained after operating the pot mill for 48 hours.

Glazing and Sintering the Circular Bars

Some circular bars obtained were glazed by dipping method. The glazed bars were sintered in the shuttle kiln.

Sintering the Test Circular Bars at 1230ºC and 1300°C

Circular bars were sintered in the shuttle kiln. 6hr-sintering was required to reach 300°C and 12 hours for 600°C. Those were smoking period. 2 hours was needed for 650°C and 6 hr for 950°C. 2 hours was required for 950°C - 1000°C and 6 hours for 1000°C - 1230°C (under reduction condition).The annealing time was 2 hours and cooling time was 29 hours. Circular bars were also sintered as described above but final temperature was 1330°C (under oxidation condition).

Physico-chemical Properties of Porcelain

Physico-chemical properties of unglazed test circular bars (color, drying shrinkage, firing shrinkage, water absorption, bulk density and apparent porosity) were determined.

Mechanical Properties of Porcelain

Mechanical properties of porcelain bodies (glazed and unglazed) such as dry Modulus of Rupture (dry MOR) and fired Modulus of Rupture (fired MOR) were determined.

Determination of Thermal and Electrical Properties of Electrical Porcelain

Thermal diffusivities of porcelain samples were determined by Flash method. Electrical resistivities of samples were measured by using an electrometer (1V-200V). The capacitances of the samples at various frequencies were measured by using an LCR meter.

Table (1) Compositions of Porcelain Bodies with Added Alumina

Sr. No	Body	Ball clay (TN) (%w/w)	Ball clay (YKT) (%w/w)	China clay (KP) (%w/w)	China clay (ST) (%w/w)	China clay (YZY) (%w/w)	Feldspar (TZ) (%w/w)	Quartz (Myeik) (%w/w)	Alumina (%w/w)
1	G	5	20	6	7	4	38	10	10
2	H	10	30	-	-	-	45	-	15
3	I	30	-	3	-	8	33	22	4
4	J	3	20	4	-	12	36	22	3
5	K	-	9	-	15	18	38	-	20
6	L	3	20	4	-	15	36	20	2

G, H, I, J, K, and L = Prepared body mixtures
TN = Taungnaut, YKT = Yankintaung, KP = Kanpauk, ST = Shwetaung, YZY = Yozayat, TZ =Tharzi

Table (2) Composition of Glaze for Electrical Porcelain

Sr.No	Materials	Composition (%w/w)	Sr.No	Materials	Composition (%w/w)
1	Dolomite	8	6	$BaCO_3$	3
2	Calcite	5	7	Al_2O_3	2
3	Feldspar	43	8	MnO_2	6
4	Quartz	22	9	Cr_2O_3	1
5	Ball clay (YKT)	8	10	Fe_2O_3	2

Results and Discussion

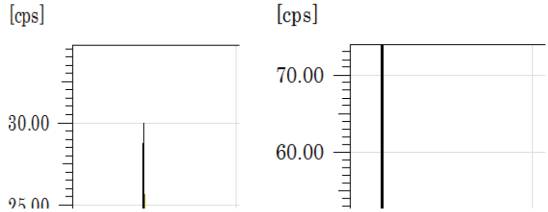
The local raw materials such as ball clay, china clay, feldspar, and quartz were used in production of electrical porcelain. Clays are chosen for the particular properties desired and are frequently blended to give the most favorable result. It was observed from ED-XRF spectra that ball clay (YKT) has higher Al_2O_3 than ball clay (TN). But ball clay (TN) has higher SiO_2, FeO and K_2O than ball clay (YKT). More traces compounds and elements were occurred with ball clay (TN) than ball clay (YKT). The highest Al_2O_3 content was given by china clay (YZY). The highest SiO_2 content was occurred in china clay (ST). But K_2O content was the same in both china clays (KP) and (ST). TiO_2 content was the same for all three china clays. But the highest FeO content was observed in china clay (KP). The amount of K_2O in the feldspar (TZ) is also important for glaze. Local feldspar containing 0.53% iron III oxide is suitable to use as raw material for making glaze. The content of SiO_2 in quartz (Myeik) was 97.25% with only 2.35% Al_2O_3.

Mechanical properties as Modulus of Rupture of clay minerals were measured and shown in Table (3). It was observed that fired MOR was not depending on dry MOR. The highest fired MOR was obtained from ball clay (YKT). Whereas the highest dry MOR was found in china clay (KP). Naturally, ball clay is finer than china clay. It was observed that fired MOR of ball clays was higher than that of china clays. Thus, highly dense clays would have high mechanical strength.

To increase the strength and to upgrade the quality, electrical porcelains were fabricated with added alumina. The percentages of clays were varied with respect to percentages of feldspar and quartz, and again also percentages of added alumina were varied based on main raw materials composition. For high alumina porcelain bodies, increased temperature is required in order to get thoroughly vitrified electrical porcelain. Porcelains with added alumina were sintered at 1230°C and 1300°C in the shuttle kiln and an investigation was done on the difference between the effect of sintering under reduced atmosphere and the real atmosphere. The important thing, however, is that the original composition of the raw body in respect of quartz, feldspar and clay determines the distribution of the crystalline phases in the fired product.

Tables (4) and (5) show that, the highest bulk density of sample K1, 2.6 g/cm^3 was found among all samples. Thus, sintering under reduced atmosphere would give denser porcelains. The relative density of the samples was dependent on the composition of the bodies. An insulator must have 0% water absorption and 0% porosity. Drying shrinkage values were between 3% and 4%. Firing shrinkage was between 7% and 12%. Total shrinkage of all samples was 11% to 15%. Water absorption 0% and 0% porosity samples were G1, J1, L1 (sintered temperature=1230°C) and G2, I2, K2, L2 (sintered temperature=1300°C). Thus, high temperature was required for porcelains with added alumina. The atmosphere inside a kiln has a marked influence on the colour and surface of the finished product.

From Table (6), high strength was found in porcelains with added alumina sintered at higher temperature. Several important factors were found in gas/air kiln for ceramic production; size, refractoriness temperature, control, and operation manner of gas/air kiln would be included. Annealing is also an important stage to get high strength.



Al_2O_3	28.60(± 0.35)(wt%)
SiO_2	64.16(± 0.47)(wt%)
K_2O	1.61(± 0.05)(wt%)
TiO_2	1.42(± 0.08)(wt%)
FeO	3.19(± 0.05)(wt%)

Fig (1) X-ray Fluorescence Spectrum of Ball Clay (YKT)

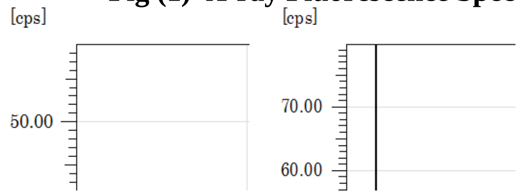


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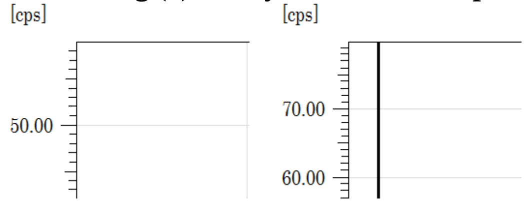
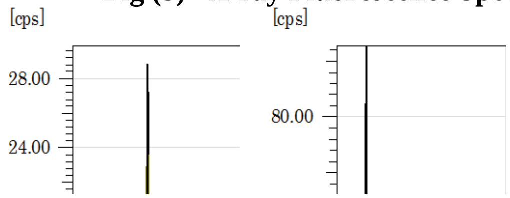
Fig (2) X-ray Fluorescence Spectrum of Ball Clay (YKT)



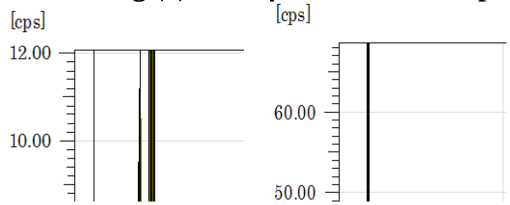
Al_2O_3	20.92(± 0.33)(wt%)
SiO_2	61.08(± 0.48)(wt%)
K_2O	2.06(± 0.06)(wt%)
$CaCO_3$	6.38(± 0.19)(wt%)
TiO_2	1.36(± 0.07)(wt%)
FeO	6.51(± 0.07)(wt%)

Fig (3) X-ray Fluorescence Spectrum of China Clay (KP)



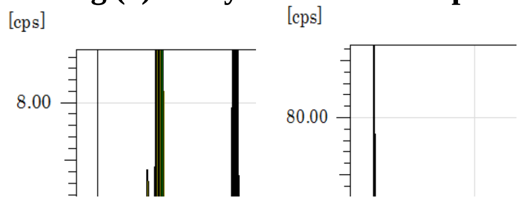
Al_2O_3	26.89(± 0.33)(wt%)
SiO_2	67.50(± 0.47)(wt%)
K_2O	2.05(± 0.05)(wt%)
TiO_2	1.37(± 0.08)(wt%)
FeO	1.21(± 0.03)(wt%)

Fig (4) X-ray Fluorescence Spectrum of China Clay (ST)



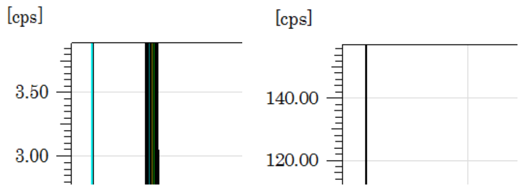
Al_2O_3	35.85(± 0.37)(wt%)
SiO_2	58.22(± 0.44)(wt%)
K_2O	0.93(± 0.04)(wt%)
$CaCO_3$	1.62(± 0.10)(wt%)
TiO_2	1.44(± 0.08)(wt%)
FeO	1.23(± 0.03)(wt%)

Fig (5) X-ray Fluorescence Spectrum of China Clay (YZY)



Al_2O_3	14.52(± 0.27)(wt%)
SiO_2	73.30(± 0.49)(wt%)
K_2O	11.46(± 0.13)(wt%)
TiO_2	0.07(± 0.03)(wt%)
FeO	0.53(± 0.02)(wt%)

Fig (6) X-ray Fluorescence Spectrum of Feldspar (TZ)



Al_2O_3	2.35(± 0.11)(wt%)
SiO_2	97.25(± 0.52)(wt%)
K_2O	0.21(± 0.02)(wt%)
TiO_2	0.02(± 0.03)(wt%)
FeO	0.08(± 0.01)(wt%)

Fig (7) X-ray Fluorescence Spectrum of Quartz (Myeik)

Table (3) Mechanical Properties of Clays

Sr. No	Property (kg/cm^2)	Ball clay (TN)	Ball clay (YKT)	China clay (KP)	China clay (ST)	China clay (YZY)
1	Dry MOR	16.6	15.4	32.5	7.4	8.7
2	Fired MOR	302.3	450.5	300.9	105.5	197.8

Table (4) Physico-chemical Properties of Electrical Porcelain Bodies with Added Alumina (Al_2O_3) Sintered under Reduced Condition

Sintering temperature = 1230°C Firing cycle = 65 hours

Sr. No	Property	Electrical Porcelain Body						Specification Accordance to Standard IEC-672
		G1	H1	I1	J1	K1	L1	
1	Bulk density(g/cm^3)	2.4	2.5	2.4	2.4	2.6	2.5	2.2 minimum
2	Water absorption (%w/w)	0	0.3	0	0.4	1.3	0	0
3	Apparent porosity (%)	0	0.8	0	0.8	3.15	0	0
4	Drying shrinkage (%)	4.0	3.5	4.0	3.0	2.5	3.5	-
5	Firing shrinkage (%)	9.0	9.0	9.5	9.5	10.5	9.5	-
6	Total shrinkage (%)	13.0	12.5	13.5	12.5	13.0	13.0	-

Table (5) Physico-chemical Properties of Electrical Porcelain Bodies with Added Alumina (Al_2O_3) Sintered under Oxidized Condition

Sintering temperature = 1300°C Firing cycle = 65 hours

Sr. No	Property	Electrical Porcelain Body						Specification Accordance to Standard IEC-672
		G2	H2	I2	J2	K2	L2	
1	Bulk density (g/cm^3)	2.4	2.5	2.4	2.3	2.5	2.4	2.2 minimum
2	Water absorption (%w/w)	0	0.4	0	0.3	0	0	0
3	Apparent porosity (%)	0	0.9	0	0.7	0	0	0
4	Drying shrinkage (%)	4.0	3.5	4.0	3.0	2.5	3.5	-
5	Firing shrinkage (%)	9.0	9.0	9.5	9.5	10.5	9.5	-
6	Total shrinkage (%)	13.0	12.5	13.5	12.5	13.0	13.0	-

Table (6) Mechanical Properties of Electrical Porcelains Sintered at Different Temperatures

Body	Sintering - temperature (°C)	Modulus of Rupture (kg/cm^2)		
		Before Sintering	Sintered	Sintered with Glaze
G1	1230	21.0	990.5	1005.9
H1	1230	25.0	905.5	1023.0
I1	1230	20.0	759.5	960.9
J1	1230	18.0	950.0	1008.8
K1	1230	15.0	991.0	1020.5
L1	1230	25.0	907.2	1095.5
G2	1300	21.0	936.3	1103.0
H2	1300	25.0	936.5	1038.0
I2	1300	20.0	790.0	989.0
J2	1300	18.0	998.0	1110.0
K2	1300	15.0	1000.0	1151.0
L2	1300	25.0	990.5	1109.5

From the Table (7), it can be seen that the highest thermal conductivity of electrical porcelain with added alumina was observed with body K1, and K2. Thermal conductivity values were within the acceptable range of IEC-672 Standard. Thus, alumina addition also affected thermal conductivity. It was estimated for all samples that the thermal conductivity was increased by increasing the relative density. Electrical resistivity and dielectric constant were determined for the porcelain samples and the resultant data are shown in Tables (7) and (8).

The highest electrical resistivity occurred in samples G1, G2 and sample I2. The low resistivity was found in sample H2. It was found to be semiconductors. Moreover, it can also be seen from Table (8) that the dielectric constant at different frequencies of the above high resistivity values were narrow, and so data was reliable. Low electrical resistivity samples were not efficient to get reliable data for the dielectric constant.

It was found from Table (8) that all porcelain samples were dependent on frequency. Their dielectric constant values were gradually decreased as the frequency was increased. But exceptional case occurred at sample G. Electrical properties did not depend on the sintering condition (reduction or oxidation) and the addition of Al_2O_3 did not show the clear effect on the electrical properties.

Table (7) Thermal Conductivity and Electrical Resistivity of Porcelains

Body	Sintering - temperature (°C)	Thermal Conductivity (W/m/K)	Electrical Resistivity (Ωcm)	Body	Sintering - temperature (°C)	Thermal Conductivity (W/m/K)	Electrical Resistivity (Ωcm)
G1	1230	1.8	2.99E+13*	G2	1300	1.9	3.89E+13*
H1	1230	1.8	3.00E+11	H2	1300	1.7	3.79E+10
I1	1230	1.6	3.00E+11	I2	1300	1.7	1.35E+13*
J1	1230	1.6	5.27E+12	J2	1300	1.7	1.26E+12
K1	1230	2.3	5.27E+12	K2	1300	2.4	3.09E+12
L1	1230	1.8	6.88E+11	L2	1300	1.6	6.45E+12

* = The highest electrical resistivity values

Thermal Conductivity Accordance to Standard IEC-672 = 1 - 2.5 (W/m/K)

Table (8) Dielectric Constant of Electrical Porcelains

Body	Sintering - Temperature(°C)	Bulk Density (g/cm^3)	Dielectric Constant			
			100Hz	1000Hz	10000Hz	100000Hz
*G1	1230	2.4	2.2	2.1	2.1	2.0
H1	1230	2.5	9.4	8.3	7.9	7.7
I1	1230	2.4	1.6	1.4	1.4	1.3
J1	1230	2.3	9.8	8.9	8.5	8.4
K1	1230	2.5	10.4	9.4	9.1	9.0
L1	1230	2.4	5.2	4.8	4.5	4.4
*G2	1300	2.4	1.9	1.9	1.9	1.9
H2	1300	2.5	10.1	9.3	8.9	8.8
I2	1300	2.4	8.7	8.1	8.0	7.9
J2	1300	2.4	9.6	8.9	8.6	8.4
K2	1300	2.6	9.7	9.0	8.8	8.6
L2	1300	2.5	8.7	8.1	7.9	7.9

* = The dielectric constant at different frequencies were narrow and reliable.

Fig (8) Unglazed and Glazed Electrical Porcelains

Conclusions

Aluminous body required higher sintering temperature than siliceous body. Properties of the finished bodies varied to a remarkable extent depending upon the sintering temperatures (1230°C and 1300°C) and condition (under reduction or oxidation). Increase in bulk density was observed under sintering at reduced condition and consequences were zero porosity, no water absorption and higher strength. Glazed porcelains were found to be higher in strength than unglazed porcelains. The disadvantage of sintering under reduced condition was that sooty colour was observed on the body due to excessive carbon deposits.

There was a tendency that the Al_2O_3 addition was effective to increase the thermal conductivity. The relative density of the samples depended on the composition of the samples, and it was estimated for prepared samples that the thermal conductivity and the resistivity increased by increasing the relative density of the samples. The addition of alumina distinctly increased the mechanical strength and required rather higher temperature. The chemical composition of the porcelain had also some advantages for higher conductivity. Samples: G1, G2, and I2 exhibited high resistivity. The scattering of the measured dielectric constant of these samples was narrow, and the data are reliable. These materials have potential to use as insulators. The high-tension insulator must have good electrical properties as well as high mechanical strength.

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