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CHARACTERIZATION OF AJEBO CLAY IN SOUTH WEST NIGERIA

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ABSTRACT

Ajebo clay deposit in Abeokuta, South West, Nigeria was characterized to establish its use industrially. The major properties investigated were drying and firing behaviour, apparent porosity, bulk density, water absorption capacity,, chemical compositions and X-ray diffraction. The qualitative analysis by inductively coupled plasma showed the mineral to be composed mainly of Aluminium, Oxygen and Silica, with low content of iron. The clay has modulus of rupture (strength) ranging from 15.04-35.62kgF/cm^2 and the colour ranged from white brown to light red on firing. The properties signify that Ajebo clay is stoneware clay. It can be used for the production of stoneware, flowerpot, tiles and brick making.

KEYWORDS: Ajebo clay, characterization, chemical composition, X-ray diffractometre, kaolin.

INTRODUCTION

Sourcing of appropriate raw materials from the abundant natural endowments in Nigeria for industrial use has not generated significant success because technical information on the integrity of these natural endowments is seldom available. Extensive investigation has been carried out on the liquid mineral endowment of the country, however, not much has been done on the solid mineral endowment of which clay is prominent and consequent upon this, adoption of solid mineral on industrial scale is scanty. The main policy thrust of the economic reform programme of the Nigerian government is mobilising national capability in converting the country's endowments into utility products and services for the common man Needs Document, (2004) .

A great emphasis is placed on exploiting the abundant solid minerals endowments in the country with a view to diversifying the economic base of the country, improving Gross Domestic Product (GDP) and industrial activity. One of these endowments with tremendous potential for economic utilisation is clay. Clay deposit is spread across the six geo- political zones of the country. (Adegoke, 1980). Clay is a fine textured earth that is plastic when wet but hard and compact when dry or a term used to refer to the finest grain particles in the sedimentary rocks and hydrothermal deposits. (Mc. Graw-Hill,

1992). There are two general types of clay: expandable and non-expandable. (Ahmed *et al.,* 1986). Expandable clay swells up when or if enough water is added to it. Non-expandable clay called bentonite is used to make drilling mud in the petroleum industry, also in the ceramics industry to make bricks, tiles pottery and porcelains. The important properties of clay are plasticity, colour, clay strength, drying and firing shrinkages. The percentage of the mineral oxides (Fe_2O_3, MgO, CaO, Na_2O) in the clay ultimately determine the areas of application of the clay such as in application of the clay such as in bricks, refractory, floor, tiles, paper, etcetera while the quantity of the alkali metal oxides (Na_2O, K_2O, CaO) indicate their suitability for making ceramic product. (Nnuka *et al.*, 2004).

Nigeria has appreciable distribution of industries engaged in metal and process industries hence the need for adequate and appropriate raw materials to support their growth. Clay products such as ceramics wares, burnt bricks, roofing and floor tiles are cheaper and durable building materials than cement especially under tropical conditions. (Nnuka *et al.,* 2004). This work therefore investigates the composition and physical properties of Ajebo clay deposit in Abeokuta, Nigeria for ceramic raw material, earthenware, tiles and brick making.

MATERIALS AND METHODS

Raw Materials

Representative samples of Ajebo clay deposit from Abeokuta in Ogun-State were taken by hand (as received) from two (2) different geological locations at their respective depths.

Determination of the different properties of Ajebo sampled clays

The physical, chemical, mineralogical and thermal properties of Ajebo clay was examined in the Materials Laboratory of Imperial College, London.

The results were compared with results published in literature, to ascertain if these clays are suitable for the manufacturing of floor tiles, ceramics, stoneware and firebricks.

Physical Properties

Sample Preparation

Ajebo sample was dried in a drying oven at 110°C for 24 hours, then crushed with a jaw crusher and milled in a ball mill for 8 hours. The raw materials slips were removed from the ball mill and again dried at 110°C, ground in a mill to a particular size of ~1mm. Moisture was added by means of a spray gun to achieve a moisture content of ~5%. The sample was then screened through a 1mm sieve in order to break up the lumps and to obtain even distribution of the added moisture.

Pressing

Dry pressing is commonly used to produce ceramic particles thicker than 0.5mm. Stages of dry pressing include: filling of the die, compaction, shaping and ejection.

Approximately 8mm diameter die were pressed using a laboratory hydraulic press Model 15010. The desired pressure applied, 40 MPa was maintained for about 60 seconds to allow for particle rearrangement. Freshly compacted test specimen were measured, weighed, marked for further identification and dried in an oven type MOV 112 at 110°C overnight prior to firing. Dry shrinkage, fired shrinkage and total shrinkage of these samples was determined at various

temperatures of 600, 700, 800, 900, 1000, 1100, 1200°C. A micrometer with accuracy of 0.01mm was used to measure the length of the samples directly after pressing, and the length of the dried samples. After firing at specific temperatures, the fired length for each temperature was measured. The formula for the calculation of dry shrinkage is:

$$\% \text{ DS} = \frac{L_2 - L_3}{L_2} \times 100 \quad (1)$$

Where, DS = Dry Shrinkage
L_2 = length of sample directly after pressing (mm)
L_3 = length of dry pressed sample (mm)

The formula used for the calculation of the fired shrinkage at each temperature is:

$$\%TS = \frac{L_2 - L_4}{L_2} \times 100 \quad (2)$$

where, TS = Total Shrinkage
L_2 = length of sample directly after pressing (mm)
L_4 = length of fired sample at a specific individual temperature (mm)

Water Absorption

Water absorption measurements were carried out according to British Standard EN 99 (1991). Fired pellets were soaked in boiling water in a beaker, and left cooling down to room temperature still soaked in water. Excess water was removed from the pellets surface using a moistened cloth prior to weighing. The wet weight (W_w) was then measured. Pellets were left drying in an oven at 110°C overnight, and the dry weight was measured (W_d). Water absorption (WA) was computed as:

$$WA = \frac{W_w - W_d}{W_d} \times 100 \quad (3)$$

$$BD = \frac{S_m}{S_v} \quad (4)$$

Assuming that the composition of the glassy phase and that of the crystalline materials present in the samples fired at neighbouring firing temperatures, and thus their theoretical density, are very similar, it is then possible to use bulk density measurements to give an idea of the total porosity of the ware. The higher the bulk density, the lower the total porosity.

Linear Shrinkage

Dry-to-fired contraction or linear shrinkage (LS) was determined by measuring the diameter of the dried pellet (D_d) and that of the fired pellet (D_f), according to the following equation:

$$LS = \frac{D_d - D_f}{D_d} \times 100 \quad (5)$$

The measurements were made using digital calliper. As the ceramic body densifies, it shrinks in all direction. The shrinkage along the disk diameter gave more results than that of the height.

Pressing Moisture

A representative batch of pressed test pieces was weighed (M2), dried at 110°C for 24 hours and then weighted (M1) again. The difference in mass was divided by the initial mass, multiplied by the initial mass, multiplied by the average pressing moisture of the sample is:

$$\%MC = \frac{M_1 - M_2}{M_2} \times 100 \quad (6)$$

where, MC = moisture content
M_1 = weight of the sample after drying
M_2 = weight of the sample after pressing.

Moisture Expansion

Most fired clay materials exhibit irreversible expansion over a period of time as a consequence of moisture absorption from the atmosphere. This expansion is usually rapid during the early life of the material but slows down to a negligible value over a period of 5years. The expansion is irreversible over the temperature range experienced in building according to the specified standard BSB 1993-2005.

RESULTS AND DISCUSSIONS

The loss on ignition corresponds to water vapour from dehydroxylation reactions in the clay minerals, carbonate decomposition into CO_2 and oxides as well as burning out of organic matter. From the chemical formula, a pure kaolinite clay sample could lose 13.9wt% of its mass on ignition due to dehydroxylation with water release. In characterising Ajebo sample clay it has 17.7wt% loss on ignition of which is 3.8wt% higher than 13.9wt%. This is due to the fact that the Ajebo clay was not treated (either by sedimentation or deflocculation). Ajebo clay is rich in Alumina and Silica as presented in Table 1 and can be compared to Malaysian clay in Yung-Feng Chen *et al.* (2004a) whereas manganese oxide is absent. This kaolin mineral has an appearance that is whitish/greyish in colour.

The XRD analyses clearly showed kaolin as the main clay minerals present in Ajebo clay as shown in Figure 1. The thermal gravimetric analysis/Differential scanning calorimetric (TGA/DSC) trace of the untreated Ajebo clay is shown in Figure 2. The endothermic event at ~530°C is due to the organic burn off, and probably consists of hygroscopic water release. This event was followed by a

Table 1: Chemical Composition of Ajebo clay deposit from Abeokuta, South-West, Nigeria

Raw Material	SiO2	TiO2	Al2O3	Fe2O3	MnO	MgO	CaO	Na2O	K2O	P2O5	LOI
Ajebo1	46.4	1.69	34.0	2.49	0.00	0.04	0.02	0.03	0.08	0.04	17.7

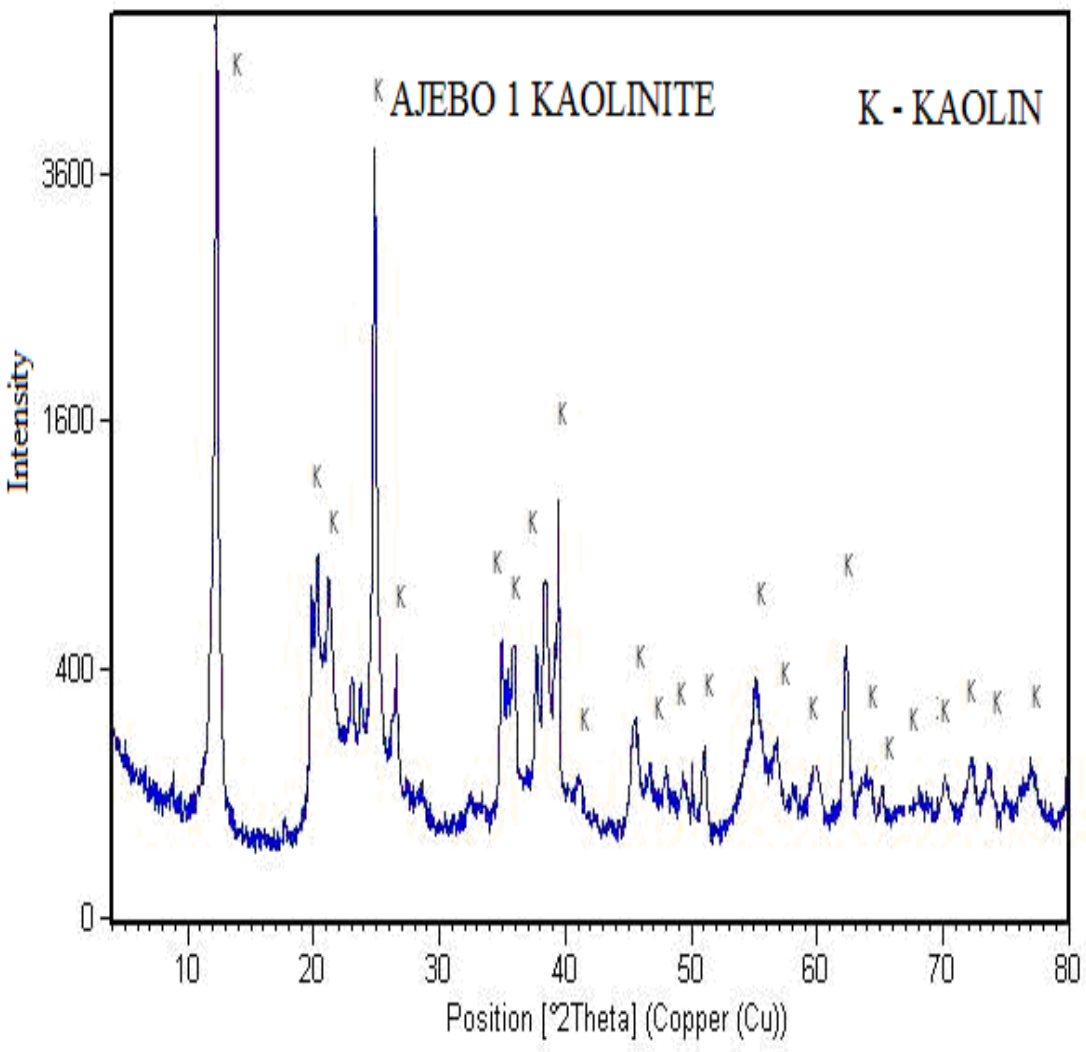

Figure 1: X-ray diffractometer traces of the untreated Ajebo raw clay using Phillips PW1700

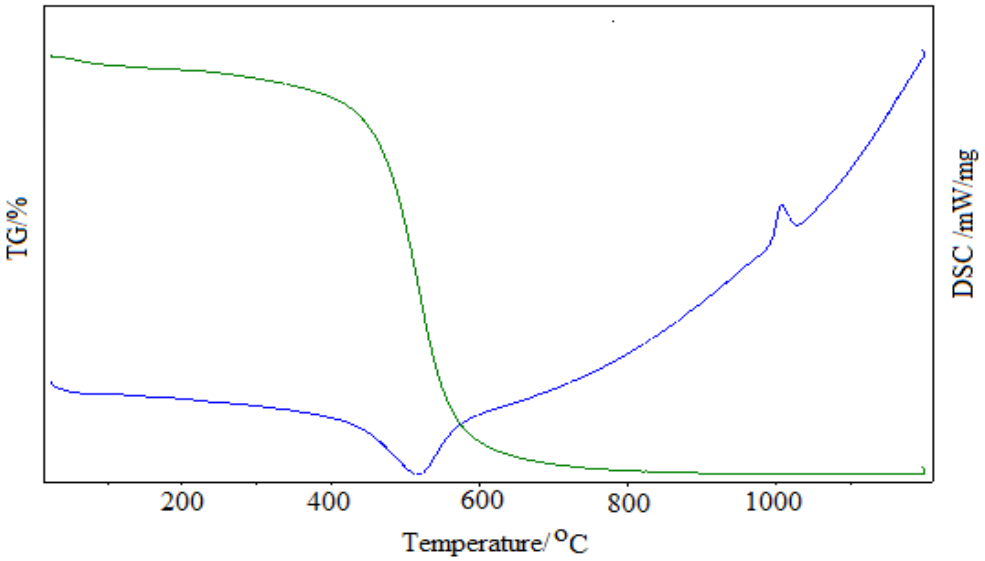

Fig 2: Thermogravimetric analysis/Differential scanning calorimetric (TG/ DSC) of Ajebo raw clay in Abeokuta, Ogun-State

wt % loss in the clay sample, as observed in the TG/DSC trace. The weight loss was due to removal of adsorbed water at ~100 ° C. Organic matter burn-off may be responsible for the upward slope on the thermogravemetric analysis (TGA) trace.

At 800-993°C, the crystallisation of a spinel structure Al_2O_3 was formed, followed by the release of free silica, which assist in the liquid formation leading to pronounced shrinkage ~2.5% of the clay body temperature.

Exothermic reaction was clearly observable from ~ 993-1044°C. This reaction may account for the beginning of the growth of crystallisation of mullite or Al_2O_3 spinel as reported in other studies by Liu *et al.* (1994), Schneider *et al.* (2004), Sainz *et al.* (1997), Chen *et al.* (2000), Castelen *et al.* (2001), Yung-Feng Chen *et al.* (2004b) and Viswabaskaran *et al.* (2004).

Bulk density, apparent density, percentage moisture content and apparent porosity were fired in oxidising atmosphere, shows the bulk density reached a maximum after firing at ~1100°C and its apparent density remained unchanged at ~900-1000°C.

Likewise, the percentage apparent porosity at ~9000. 1000°C followed a decrease at 1200°C as in the works of Souza, 2004).

It is evident from Table 2 that as the temperature increases dry shrinkage and fixed length reduces but percentage dry fired shrinkage and percentage wet dry shrinkage behaved in a way of Hook's law, that is they increases from temperature 600-800^{0}C when they start to reduce from 900^{0}C. Ajebo shrinkage test (average of12.4%) showed that the clay deposit lie within standard value for firebricks Singer *et al.* (2004). Shrinkage is the property of clay that is important for brick making hence the clay can be used for bricks production.

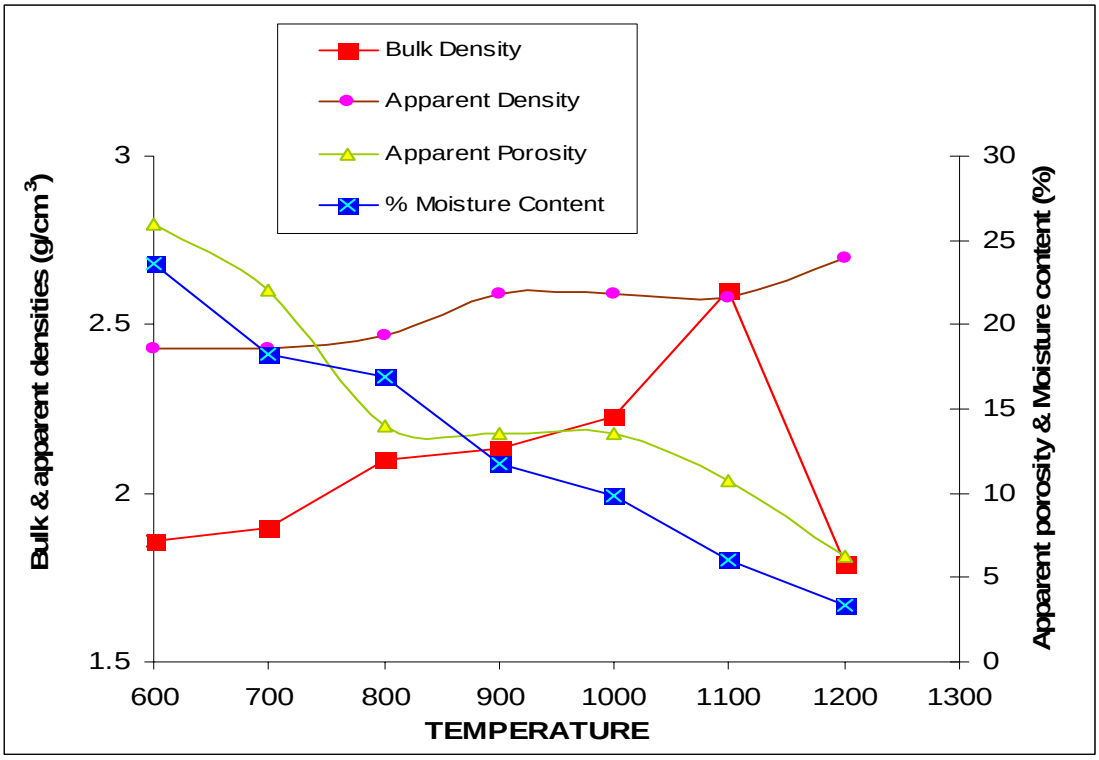

Figure 3: Bulk density, apparent density, moisture content, and apparent porosity as a function of firing temperature

Table 3 showed that the greater the apparent porosity, the greater the water absorption capacity of the clay which has very strong relationship on its firing behaviour, as during drying this absorption water must be driven out. This usually leads to a high drying shrinkage.

It was observed that at different temperatures some major properties studied on Ajebo clay deposit showed different behaviour. At temperatures 600-900^{0}C, there was no crack formation and the respective colour varied from whitish brown to light red. This could be attributed to the content of iron oxide of 2.49%. The ferrous iron impacted a red colour on the fired sample due to conversion from ferrous to ferritic compound. The colour variation is considered usable for the manufacture of earthenware Rhodes, (1973).

As presented in Table 4, the modulus of rupture is the load bearing capacity of Ajebo clay ranging from 15.04-35.62KgF/cm^2, as temperature increased from 600-1200^{0}C.

The strength behaviour was found to increase with temperature and this could be attributed to bond formation in the glassy phase.

Table 2: Shrinkage Test for Ajebo Clay in Abeokuta South-West, Nigeria

Temperature ^{0}C	Ajebo clay					
	Original diameter (mm)	Dry Shrinkage (mm)	Fixed length (mm)	% Dry fired Shrinkage	% Wet Dry Shrinkage	Total Shrinkage
600	8	7.94	7.30	7.54	10.29	8.8
700	8	7.92	7.23	7.62	10.40	9.6
800	8	7.83	7.09	7.76	10.60	11.4
900	8	7.81	7.06	7.67	10.48	11.8
1000	8	7.72	6.97	7.58	10.35	12.9
1100	8	7.66	6.78	7.49	10.22	15.3
1200	8	7.35	6.65	7.38	10.01	16.9

Table 3: Bulk density, apparent porosity, water absorption, apparent density, moisture content as a function of firing temperature up till 1200^{o}C, behaviour and colour variation

Temperature ^{0}C	Bulk density g/cm3	Apparent Porosity	apparent density g/cm3	Water Absorption. %	Colour formation	Crack formation
600	1.86	26	2.43	23.6	White Brown	No Crack
700	1.90	22	2.43	18.2	White Brown	No Crack
800	2.1	14	2.47	16.9	Brown	No Crack
900	2.13	13.6	2.59	11.7	Light Red	No Crack
1000	2.23	13.6	2.59	9.8	More Red	Slight Crack
1100	2.60	10.8	2.58	6.1	Very Red	Slight Crack
1200	1.79	6.25	2.70	3.34	Yellowish Brown	Crack at the bottom

Table 4: Modulus of Rupture for Ajebo clay

Temperature ^{o}C	Distance between Support (cm)	Modulus of Rupture kgF/cm^2
600	8cm	15.04
700	8cm	17.55
800	8cm	20.05
900	8cm	22.60
1000	8cm	32.95
1100	8cm	33.40
1200	8cm	35.62

CONCLUSION

This clay was characterised using standard techniques. The clay was found to have moderate shrinkage 8-16%, the colour characteristics ranged from whitish brown to yellowish brown on firing and a good strength of 15.04-35.62KgF/cm^2 at temperatures 600-1200^{0}C. The strength behaviour was found to increase with temperature and this could be attributed to bond formation in the glassy phase.

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REFERENCES

Adegoke, O.S. 1980. Guide to the Non-Metallic Mineral Industrial Potential of Nigeria. Proceedings of the Raw Materials Research and Development Council. Pp110-120.

Ahmed, K.S., Onaji, P.B. 1986. The Effects of Beneficiation on the Properties of Some Nigerian Clay. *Nigerian Society on Chemical Engineering,* 6(2): 119-129.

Castelein, O., Soulestin, B., Bonnet, J.P. Blanchart, P. 2001. The influence of heating rate on the thermal behaviour and mullite formation from kaolin raw material. *Ceram. Int.,* 27: 517-522.

Chen, C.J., Lan, G.S., Tuan, W.H. 2000. Microstructural Evolution of Mullite during sintering of kaolinite powder compacts. *Ceram. Int.,* 26: 715-720.

Liu, K.C. Thomas, G. 1994. Time-Temperature Transformation Curves for Kaolinite Alumina. *J. Am. Ceram. Soc.,* 77: 545-552.

Mc Graw-Hill Encyclopaedia of Science and Technology. 1992. Mc Graw Hill Book Company. 8th Edition, 3: 752-761.

NEEDS Document. 2004. Published by National Planning Commission, Abuja. ISBN 0-9741/04-81.

Nnuka E.E., Enejo, C. 2001. Characterization Nahuta Clay for Industrial and Commercial Applications. *Nigerian Journal of Engineering and Materials,* 2(3):,9-12.

Rhodes, D. 1973. Clay and Glazes for the porter, Revised Edition. Chilton Book Company, Rednor, Pennsylvania, A&C London. Pp.6-23.

Sainz, M.A., Serrano, F.J. Bastida, J., Caballero, **A.** 1997. Microstructural Evolution and Growth of Crystallite size of mullite during thermal transformation of kyanite. *J. Eur. Ceram. Soc.,* 17: 1277-1284.

Schneider, H., Okada, K., Pask, J. 1994. Mullite and Mullite Ceramics. John Wiley and Sons, New York.

Singer, F., Singer, S.S. 1971. Industrial Ceramics. Chapman and Hall Limited, London. Pp. 20-51.

Souza, G.P. 2004. Microstructural variations in Porcelain Stoneware as a function of flux system. *J. Am. Ceram. Soc.,* 87(10): 1959-1966.

Viswabaskaran, V., Guanam, F.D. Balasubramaniam, M. 2004. Mullitisation behaviour of calcined clay-Alumina mixtures. *Ceram. Int.,* 29: 561-571.

Yung-Feng, Chen., Wang, M.C., Hon. M.H. 2004a. Phase Transformation and Growth of Mullite in Kaolin. Ceram. *J. Eur. Soc.,* 24: 2389-2397.

Yung-Feng Chen., Chang, Y.H., Wang, M.C 2004b. Effects of Al_2O_3 addition on the phases, flow characteristics and morphology of the porous kaolin ceramics. *Mater. Sci. eng.,* A373: 221-228.

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