AN ENQUIRY INTO SELECTED LOCAL RAW MATERIALS FOR THE FORMULATION OF ELECTRIC PORCELAIN BODY IN NIGERIA

BY

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DEPARTMENT OF INDUSTRIAL DESIGN
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DECLARATION

I solemnly declare that this thesis is an original work of mine and that it has not been submitted elsewhere for the award of any degree. All references are duly acknowledged in "References".



George Ẽsele Uduigwome.

CERTIFICATION

This thesis entitled AN ENQUIRY INTO SELECTED LOCAL RAW MATERIALS FOR THE FORMULATION OF ELECTRICAL PORCELAIN BODY IN NIGERIA, by Uduigwome George Esele, meets the regulations governing the award of the Degree of Master of Arts (Industrial Design) of Ahmadu Bello University, Zaria and is approved for its contribution to knowledge and literary presentation.

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DEDICATION

This thesis is dedicated to my parents: Chief Fred Ahmadu Uduigwome and Comfort E. Uduigwome, my brothers, sisters, nephews, and nieces - for wonders too numerous to mention.

ABSTRACT

The study was concerned with the Identification, beneficiation, characterization and utilization of locally available raw materials for the production of slip-cast electrical porcelain insulator. In the absence of the required characterization tools an approach which transferred, to some extent, the problem of characterization to that of process control was adopted. There are 25 tables, 12 figures, and 7 plates.

The raw materials procured for the study were "Fetramin" processed talc, raw talc from Tsanlu in Kwara State, primary clays from Kankara and Pakwamni in Katsina and Sokoto States respectively, secondary clay from Bomo in Kaduna State, Quartz from Okene in Kwara State and Baure in Katsina" State while the feldspars were got from Zaria in Kaduna State and Okene in Kwara State, thus placing restrictions on the generalizations of the findings of this study as, amongst other things, final properties of wares are affected by the sources of materials used.

The following basic assumptions were made:

1. That the procured materials would be suitable for the formulation of a porcelain body suitable for electrical insulation.
2. That, in the absence of facilities required for material characterization, preparation and property evaluation, simulated tests would suffice.

Based on the analyses of data collected, the following major findings were pertinent:

1. Addition of Bomo plastic clay, within defined limits, enhances castability, dry strength and densification.
2. Where water constituted about half the weight of solid ingredients, a lower limit of 3gm and an upper limit of 4gm deflocculants (combination of 75°TW sodium silicate and sodium carbonate to every 500gm solid ingredients, were found to be adequate for optimum dispersion.
3. Translucency is almost always acoompanied by a fall in bulk density and increase in apparent porosity and fired shrinkage.
4. There is a minimum attainable apparent porosity, maximum bulk density and, by necessary implication, a maximum attainable mechanical strength for each body.
5. The bodies that were finally selected have apparent porosity, bulk density and total shrinkage ranges of 0.05 - 0.16% 2.36 - 2.4gm per cc, and 14.1 - 17.8% respectively when fired to about 1300°C with a soak period of 30 minutes.
6. Translucency was found to be more pronounced in the relatively high-feldspar compositions.

The general conclusion of this study is that the raw materials required for production of electrical porcelains are locally available.

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DEFINITION OF TERMS

AGGLOMERATES:

A general term describing a small mass having a network of interconnective pores. It is comprised of primary particles bonded together by surface forces and/or solid bridges. (Onoda and Hench 1978).

AIR-FLOATING:

Process of sorting particles of similar size by air seperation.

BODY:

A clay for a special purpose created by blending different clays or by adding to clays other minerals such as feldspar and flint in order to produce a desired workability or finished result. (Hamer, 1975).

CALCINATION

A purification process which involves heating a material during which it is dehydrated and volatile matter is expelled.

CATALYST:

An agent which promotes chemical change. (Rhodes, 1973).

COMPRESSIVE STRENGTH:

Ability to withstand pressure when a component is subjected to forces acting towards each other with a squeezing or crushing action. (Shirley, 1973).

CRAZING:

A glaze defect indicated by the presence of fine cracks on the galze. It is caused by lack of fit between a glaze and the body.

DEFLOCCULATION:

The action of dispersing the fine clay particles in a slip so that the slip becomes more fluid. Deflocculation is achieved by adding, to the suspension water, suitable soluble alkalis called deflocculants. (Hamer, 1975).

EUTECTIC:

The lowest melting mixture of two or more substances 2. (adj.) Describing the temperature of= lowest melting. (Rhodes, 1973).

FILTER PRESS:

A device used in removing excess water from clay slips.

MOHS SCALE:

Mohs scale of mineral hardness is used to provide information about the hardness of substances. On this scale minerals are ranked according to their hardness.

MULLITE:

An important alumino-silicate which is created within pottery bodies during firing. Mullite crystals are long needles which interlace to produce a strong body. (Hamer, 1975).

PHASE:

Any portion of a system which is physically homogeneous within itself, is bounded by a surface, and is spatially separated from other portions according to the interfacial energy relationships existing in the system. Most ceramic materials are "polyphase" as they consist of several phases which are commonly both crystalline and glassy. (Ford, 1967).

RESISTIVITY:

The material property that provides a relative measurement of a materials opposition to current flow. It depends only on the material and not its dimensions. An insulator or dielectric has a low resistivity. (Blotter, 1981).

RHEOLOGY:

The science of flow and deformation usually applied to flow characteristics of plastic clay and casting slips. (Fournier, 1973)

SINTER:

To fasten together by mutual adhesion without apparent fusion. All ceramic materials, simple oxides or complex minerals, pass through a stage of sintering before melting. There is no liquid (molten) phase involved and yet the particles stick to each other. (Hamer, 1975).

SLIP:

A clay-water mixture

SOAK

To hold the kiln at one temperature for a period of time. (Rhodes 1973).

SPECIFIC GRAVITY:

Ratio of the density of a material to that of water at $4^{\circ}C$. It is a pure number and requires no conversion to the various units. It is numerically equal to the density when expressed in grammes per cubic centimeter (gm/cm^3). (Singer, and Singer, 1963).

THIXOTROPY:

The ability of certain clay suspensions to "thicken up" on standing, and is particularly marked with slips that have been only partially deflocculated or that have been overdeflocculated. (Worrall, 1982).

TENSILE STRENGTH:

The power of a material to withstand fracture when being stretched. (Shirley, 1973).

CHEMICAL SYMBOLS	NAME
Fe	Iron
Fe_2O_3	Ferric Oxide
Al_2O_3	Aluminium Oxide
CaO	Calcium Oxide
SiO_2	Silicon Dioxide
MgO	Magnesium Oxide
MnO	Manganese Oxide
TiO_2	Titanium Oxide
K_2O	Potassium Oxide
Na_2O	Sodium Oxide
ZnO	Zinc Oxide
BaO	Barium Oxide

CHAPTER ONE

INTRODUCTION

1.1. Introduction

The discovery by Oersted in 1819 that magnetism could be produced by an electric current presented scientists with the problem of how to bring about the reverse effect. This problem was solved by Faraday in 1831, with his discovery of the "electromagnetic induction phenomenon" whereby electricity could be produced without any batteries. By implication, larger electric currents could now be produced continuously and at low cost. Concomitantly, there was an increase in demand for electricity and this called for electric power transmission over great distances.

Increased use of electric telegraph for signals in Europe by 1840, coupled with the inventions of the telephone and filament lamp, necessitated the provision of insulation components and conductors. According to Irabor et al (1987), "At the early stages (about the mid-nineteenth century), most of the applications of electric power required fairly low voltages which made no great demand on the electrical and mechanical properties of insulators. "The earliest type of support for conductor wires included a porcelain insulator mounted on a spindle.

Waye (1967), observed that during the period of the First World War (1914-1918), electrical porcelain was used mainly for low-tension, direct current, or for low

frequency alternating current in telegraphy, electric lighting and a few power applications. For such purposes the ordinary pot insulators were adequate as there were no stringent specifications.

By 1930, high-tension electric transmission was well established and this called for porcelain of much higher quality while new compositions were required for radio communication using very much higher frequencies that was just getting into its stride.

Porcelain, apart from its low cost of production, is demanded in the electrical industry because of its high insulation resistance, high dielectric and puncture strength, mechanical strength, low porosity , weather resistance, and the ease of cleaning its surface.

While demand for electrical porcelain insulators was met by the already existing industry involved in porcelain production, special factories had to be set up in England and U.S.A. for the purpose. Norton (1978), reported that "during the Second World War (1939-45), work was active on electrical ceramic bodies, especially in Germany. For high frequency use steatite porcelains were made in large quantities. At the same time, research was going on in the field of titanates. In this country, development also occured rapidly with ferroelectrics and ferromagnetics."

Porcelain insulators of early manufacture largely lacked standardized factory-control and testing methods and this resulted in the absence of a standard

uniformity. Since 1930, however, the product has been brought to a standard of uniformity with the development of instruments, coupled with the development of some earlier discoveries into routine laboratory tools such as energy spectroscopy for chemical analysis, laser applications, high pressure capabilities, and most especially, the development of computers capable of treating data emitted by the new instruments and able to handle calculations for complex systems.

In sophisticated ceramic cultures, a relatively new concept in electrical insulation has emerged. As indicated by Fink and Carroll (1968), this concept has resulted in complete systems for insulating electrical devices and is composed only of inorganic materials such as mica, glass, asbestos, cements and ceramics which have been used for years but mostly in conjunction with organic resins or insulation.

Some of the materials used in inorganic systems include: enamel and refractory glass, glass coatings, and chemical coatings for conductor insulation; mica and synthetic mica for flexible sheet insulation; asbestos sheet, glass-mica sheet for rigid sheet insulation; cements made from various inorganics such as alkaline silicate cement for inorganic encapsulants.

McLaren and Ott (1978) opine that the three main classes of materials; metals, polymers and ceramics "hold the key to unlocking the requirements for our future systems in all areas of development including our most essential current problem of energy related systems."

1.5. Delimitations:

The study was delimited to the following:-

(1) "Petramin" processed talc (from Gbagba Adigun in Oyo State), and raw talc from Isanlu in Kwara State, primary clay from Kankara and Rakwamni in Katsina and Sokoto States respectively, secondary clay from Bomo in Kaduna State, quartz from Okene in Kwara State and Baure in Katsina State, The feldspar was got from Zaria in Kaduna State, and Okene Kwara State. Therefore, restrictions are placed on generalisation of the findings of this study because, amongst other things, final properties of ceramic wares are affected by the source of the materials used.

(2) Formulation of a porcelain body suitable for electrical insulation.

(3) Shaping by the slip-casting method.

(4) Technological aspect of production without preoccupying itself with geological derivations of raw materials nor superfluous description of equipment.

1.6. Basic Assumptions:

The following basic assumptions were made:-

(1) That the procured materials would be suitable for the formulation of a porcelain body suitable for electrical insulation.

(2) That, in the absence of required facilities for material characterization, preparation, and

property evaluation, simulated tests would suffice.

1.7. Significance of the Study:

Today electric power is necessary for the processing of raw materials as well as for more complex manufacturing processes. Bailey (1976), records that between 1951 and 1960, output of electricity in Africa doubled and in the years to 1970, it doubled again. Though this rate is well above the world average, the actual level of production of electricity has remained low. This fact, underscored by the very uneven distribution of electricity, great backlog demand as well as the future demand caused by the continuously increasing living standards in Africa and other parts of the world shows that there exists a market for electrical components and insulators such as porcelain electrical insulators.

Following the Federal Government report on imported raw materials for the 1975-1980 development plan, pottery raw materials are among those imported at well over 90% of the total required raw materials despite the fact that Nigeria has almost unlimited reserves of all the major raw materials necessary for ceramic production. Predicated on this premise, the study again, coincides with one of the primary objectives of the Raw Materials Research and Development Council of Nigeria, which is the mobilization of the countrys latent resources for developmental purpose.

Commenting on the Nigerian Trade Index for 1985,

which indicated that Nigeria spent well over twenty million Naira on importation of electrical insulator equipment and components, Irabor et al (1987) opined that "whereas no distinction was made between the various types of insulators, it is estimated that, at least, a quarter of the expenditure (i.e. about five million Naira) went towards the importation of electrical porcelain insulators used for our transmission and distribution network." The colossal sum spent on the impartation of these insulators would be saved if production is done through local sourcing. Apart from this individual economic prospect, local production would mean the introduction of a relatively new industrial activity in Nigeria and, by implication, creation of jobs.

To adopt the argument of Bailey (1976), the Industrial Revolution, in Britain for example, was not singularly due to the invention of the steam engine by James Watt, nor to the application of steam power to textile machinery by Hargreaves and others. Rather what happened was the coincidence of a number of developments all taking place at roughly the same time. Ashton (1948) believes that, amongst other things, it was due to the application of science to industry and with a more intensive and extensive use of capital. Appreciating these facts would further concretize the industrial revolution toward which the country is working.

As a professional contribution to knowledge, the study seeks to provide a guide which would assist in the collection and interpretation of data necessary for a

better understanding of ceramic systems in general and those directly related to electrical porcelains in particular.

In the light of the above, this study should be seen as an industrial and academic resource aimed at, as contained in the travelogue of the National Accounts of Nigeria, 1973-1975, "a large-scale modification of the existing economic framework for maximum utilization of resources" with a view to making governments efforts in creating a vibrant economy a reality and not a pious hope.

CHAPTER TWO

REVIEW OF LITERATURE

2.1. Introduction

Having appreciated the background to electric power distribution and how porcelain came to be used as an insulator due to its favourable electrical insulating properties (chapter one), this chapter starting with the role of research in the rediscovery of porcelain by Western ceramic cultures, discusses the materials and production method of slip-cast porcelain electrical insulator. The discourse is with a view to identifying the variables to hold constant and those that should be varied where, with due regard to economic considerations, the objectives are to develop consistent predictable results.

2.2. Origin of Porcelain

The word "Porcellana", according to Savage (1954), was the term which the Venetian traveller Marco Polo used, in the recording of his visit to the court of Kublai Khan, to describe certain wares which he saw in the process of production. The analogy made was to the similarity between the glaze of the wares and the highly polished and variegated surface of the shells of the genus porcellana.

Following Cooper (1972), "Porcelain was the name given by the Chinese (sic) to any ware emitting a clear, ringing note when struck, it did not have to be either white or translucent." A more practical definition was

given by Rhodes (1960) who defined porcelain as a stoneware body that cannot be made from any single natural material but from a proportional mixture of such several ingredients as Kaolin, Feldspar and Flint, the particles of which fired over and above 1300°C, fuse to yield a strong impervious body. The T'ang potters (T'ang Dynasty A.D. 618-906), are believed to have mastered this technique which saw its full exploitation by the Ming Potters (Ming Dynasty A.D 1368-1644).

Through trade intercourse, oriental porcelain found its way into foreign markets where it was well received. Hughes (1955) observed that "The growing popularity of Chinese porcelain coincided with increased economic activities and so successful reproduction of Chinese porcelain became a matter of importance. The country first to acquire the secret of making porcelain was certain of an enormous profit making monopoly."

Due to economic reasons the importation of porcelain was banned by the English Court in 1694, and the need to acquire the secret of porcelain production was further increased. The potters were, therefore, faced with the onerous task of discovering the nature of a material which was white, hard, resonant when struck, and translucent. Lack of exact methods of chemical analysis meant they had to proceed from surface appearances and sound analogy.

All attempts by the European potters were futile

because they held the erroneous belief that porcelain clay could be obtained naturally. They were therefore unable to appreciate the role of the feldspathic rock "Petunse" which, chemically, is a silicate of potassium and aluminium. Savage (1954), has interpolated that it was also unlikely that their kilns could attain the exceedingly high temperature required to fuse the rock. This aspect of the problem caused considerable trouble at Meissen where the role of petunse was first appreciated by two Germans: Ehrenfried Walther von Tschirnhausen, who had had some previous success in melting a variety of refractory substances which required higher temperatures than could be attained at the time, and his assistant - Friedrich Bottger, an alchemist.

Savage (1954) indicated that the first specimens of an unglazed porcelain were made in 1708 - the year Tschirnhausen died - but it was not possible to glaze it until the following year. The discovery led to the establishment of a factory for the manufacture of porcelain in 1710, under the title of the Royal Saxon Porcelain Manufacture, at Meissen very close to the city of Dresden.

The first major variations on the discovered porcelain formula was from a factory at Bow, near London. (Umar, 1987). This saw the addition of calcined bones to the main ingredients of kaolin, quartz and feldspar. Other modifications were: varying the proportions of

the three main constituents; and minor additions of new materials.

Today, a "soft" and "hard" dichotomy exists for porcelain bodies. The terms, whilst referring to the firing treatment-soft porcelain fired below 1350°C, hard porcelain fired between 1280 and 1460°C - also denote the degree of hardness exhibited by the ware. Waye (1967), further identified two principal types of hard porcelain as Oriental porcelain and porcelain made in England and U.S.A. The difference in the two, he noted, stems from the different historical development of porcelain and the different raw materials, particularly clay, available. According to him, porcelain made in Dresden contained less fluxes than the original Chinese ware, hence the former required a higher temperature.

Soft porcelains would include frit and dental porcelain while chemical, mullite and electrical porcelains are examples of hard porcelains.

2.2.1. Electrical Porcelain

The term "Electrical Porcelain" is used to refer to a great variety of highly specialised components for the electrical and electronics industry. Such wares are usually made to closer dimensional tolerances than other wares and they range from high tension bushings over 10Ft in height to miniature parts of overall dimensions of a

few thousandth of an inch (Waye, 1967).

Electrical porcelains usually have water absorption of 0.5% approaching zero percent. This low porosity is not due to the application of glaze hence, they are produced either glazed or unglazed. The unglazed ware is suitable for use in dry conditions and can be used as fittings for open wiring, plugs, outlet boxes, fuse holders, sockets, lamp holders, appliance parts in equipments that use electric resistances to generate heat, such as electric irons and toasters.

The glazed ware is used for weather exposed pieces such as parts for neon signs, telephone and power supplies to houses. According to Waye (1967), electrical porcelain produced in England, as at 1967, had an annual value of about £11 million (i.e. about 7% of the total of all ceramic products.)

2.2.2. Porcelain Electric Line Insulators

As discussed by Irabor *et al* (1987), electrical devices consist of four seperate components: conductors, magnetic circuit, mechanical supports and electrical insulation. Insulation confines, restrains and directs the flow of electric currents. While restraining current flow from a conductor to ground or to a lower potential, insulations, often, must provide mechanical support, protect the conductor from environmental degradation and transfer heat to the surrounding environment. They further contended that since all materials conduct electricity to some extent, no materials are absolute

non-conductors so that the best insulators are those rating lowest on the scale of conductivity.

In spite of the developments in the area of materials for electrical insulation, porcelain and toughened glass remain highly valued for common line insulation due to their favourable thermal, mechanical and electrical properties (Table 2.1) and their low cost of production. Some advantages which toughened glass insulators have over porcelain are given by Waye (1967) as: much higher puncture strength, better ability to withstand primary arcs, greater transparency to solar radiation and therefore, less strains due to differential temperatures. These claims would appear to be gratuitous as the design and particular use of the insulators are generally more important.

Say (1973) said that "the design of an insulator is a compromise between arranging the surfaces along the equipotential lines of the electric field providing sufficient thickness in the body to avoid electrical puncture, ensuring sufficient mechanical strength, avoiding recesses that might accommodate dirt and providing suitable rain-washed surfaces"

Table 2.1:

Comparison of Some Physical and Electrical Properties of Standard Insuleting Materials

Insulator Type	Mechanical Strength kg/cm²	Relative Permittivity (Er)	Dissipation Factor (Tan.δ)	Volume Resistivity ohm/cm	Electric Strength KV/M	Thermal Endurance °C
Ceramic Porcelain	600–1000	6.0	0.017	10^{14-5}	35	200
Insulating Paper	–	2.4	0.002	10^{12}	8	105
Melamine Resin Moulded	–	6.0	0.500	10^{14}	8	105
Phenolic Moulded	–	4.0	0.300	10^{11}	8	120
Polyester Resin Moulded	–	3.6	0.030	10^{17}	10	120
Polyamide	–	3.7	0.025	10^{14}	40	100
Epoxy Resin Cast	–	3.3	0.006	10^{14}	20	130

Source: Irabor et al (1987).

Porcelain electrical line insulator is a ceramic product obtained by the high temperature vitrification of the major constituents of clay, feldspar, and silica. The dense ware, free from laminations, holes and cooling stresses, is coated with a "fitting" glaze which prevents the occurence of cracks as such cracks might lead to the breakdown of the insulator. These qualities make the ware ideal for insulating high voltage lines. Units are colour coded to indicate their strength ratings and they conform to USAS C29.2.

Irabor et al (1987) identified two main types of electrical insulators as:

(a) Low tensions electric insulators:- used for applications requiring under 500 volts. They are used for telegraphs, telephone lines, internal and external low voltage network. Low tension insulators are divided into line and insulation types such as pin and bushing types.

(b) High tension electric insulators: for voltages above 500 volts. They are usually divided into line, machine and power plant types for transmission line when pin type, suspension, bushing and large insulators are used.

2.3. Raw Materials

The raw materials required for production of porcelain electrical insulators are: naturally occuring ones: substances extracted from minerals and chemically purified; or purely synthetic materials. These include kaolin, silica, and feldspar. Minor amounts of such additives as MgO (as magnesite, talc or dolomite), CaO,

ZnO, BaO, borum phosphate or mono calcium phosphate in apatite form, have been observed (Alvarez-Estrada, 1979) to contribute to mechanical strength, and lowering of firing temperature.

It is imperative that the selected materials be iron and titanium free because the presence of a high percentage of these would leave a marked effect on microstructure and final properties.

Waye (1967) observed that close control of grain size and, in some materials, shape, are as important as chemical purity because they affect shaping, vitrification and the degree of reaction between solids during the calcination process used to synthesize certain compounds. In a related view, McLaren and Ott (1978) emphasized the need for characterization throughout the forming process commencing with the raw materials, the microstructure of the "green" compact, the microstructure of the sintered ceramic and, finally, how these structures relate to the engineering properties of the ware. The absence of these, they continued, would give rise to frequent problems arising from uncontrolled and unidentified process variables which may be as a result of raw material variations or their interactions and these have a marked effect on any of the final physical properties. The implication of this view is that the ability to control ceramic processes, frequently, depends not only on skill but on the availability of adequate characterization tools to determine the nature of process controlling variables.

Heystek (1978) said that "the most valuable instrumental characterization techniques are X-ray diffraction, thermal methods, electron optical methods, and infrared spectroscopy. No one technique, however, is adequate on its own but should be used in combination with another and with total chemical analysis, cation-exchange capacity determinations, and surface area measurements"

2.3.1. Clay

Clay is a hydrated silicate of alumina. An ideal clay substance, according to Singer and Singer (1963), would contain 46.5% silica, 39.53 alumina and 13.95% water. They opined that: Silica content above 46.5% may indicate the presence of free silica or quartz which may /cause residual strain during cooling; A high alkali content generally indicates a vitreous fired product; while a loss on ignition greater than 14% indicates carbonaceous matter. Such a clay may be plastic and, unless soluble salts interfere, it is likely to deflocculate easily.

The percentage proportions given by Singer and Singer (1963), can be relegated to pedagogical purposes because (Heystek, 1978) pure systems do not exist for layer silicates even with beneficiated materials such as kaolins, ball clays, and talcs used in the whiteware industry.

The particle size is a very important variable considered in the selection of clays. Closely related to particle size is the specific surface area, that is,

the total area of all the particles of that clay (usually expressed in square meters per gram.). It is generally agreed that the particle size is in inverse proportion to the specific surface area: the finer fractions contribute more (unit per weight) to the surface area than the relatively coarser grains.

The effective surface area of the ultimate particles of clay is related to plasticity which Hamer (1975) defines as "the property that allows the solid to be reformed without rupturing and allows the new form to remain without any attempt to return to the original form." Other factors that influence plasticity are true clay content, moisture content, particle size distribution, plasticizers, strength of particle bond (Cardew, 1969) and type of exchangeable ions (Worrall, 1982).

Hamilton (1974) points out that as a general rule, those clays which need deflocculants in excess of half of one per cent of their dry weight will be unsuitable for slip casting as they will probably warp during drying and corrode the plaster mould too quickly.

Shrinkage always accompanies the drying of clay. As the film of water between the particles of clay evaporates, (Rhodes 1973), the particles draw closer together, thus, taking up the space previously occupied by the water. The cumulative effect of each particle drawing closer to its neighbouring particle is the shrinkage of the entire mass. Particle size, again, will be seen to have a decisive effect on the shrinkage of clay

while clays with larger particles sizes will shrink less/ clays having a very fine particle size will shrink more because of the presence of more water-filled interstices which close up during drying. Rhodes (1969) explanation is that the large percentage of moisture which, for example, plastic ball clays contain, is necessary to surround all the particles, with water accounting for 25 to 35% of the weight of the plastic ball clay, "it is not surprising that ball clays contract about 10% (linear) in drying from plastic to dry and further shrink from 10 - 15% (linear) in firing to vitrification".

Worrall (1982) interpolated that the leather hard moisture contents (i.e the moisture content at which shrinkage ceases in a drying plastic clay) ranges between 10 to 32%. In practice however, this effective shrinkage will be retarded by the presence of other materials.

The fired colour of a clay (Ford, 1967, Worrall, 1982) depends primarily on its percentage of iron oxide, state of oxidation of the iron; the degree of subdivision, size of the ferric oxide crystals, and the extent of vitrification. Ford (Ibid) further noted that the presence of carbon and sulphur coupled with the oxygen pressure of the atmosphere will affect the state of oxidation.

Following Waye (1967), in a porcelain body for electrical insulation, clay is chiefly introduced as kaolin with minor additions of ball clays to enhance shapeability. Rhodes (1960) view on this is that "ball clay not only has the effect of adding to the plasticity and the workability of the porcelain mixture but also promotes an earlier

densification and fusion in the body." Addition of 1-2% of bentonite (a very plastic clay of volcanic origin) is sometimes made in place of ball clay, according to Waye (1967), the fineness of bentonite slows down filter pressing and gives problems during drying.

At this point, a brief discussion on the individual clays, that is, Kaolin and ball clays will make for a better understanding.

"China clay", "Cornish clay", and "Kaolin" are terms used to qualify a light burning, fine-textured clay usually found around its original place of formation.

The view has been expressed (Cardew, 1969), that kaolin, which in nature and commerce is never so pure as the ideal analysis suggests, does not vitrify at ~1300°C though it is strongly sintered at this temperature. He attributes this refractoriness to its chemical purity and its comparatively large ultimate particle size which averages about 0.6 to 1.0μ. (making it, at least, ten times larger than that of a typical ball clay.) This average particle size, observed Cardew (Ibid), provides, in some cases, a useful bridge between the ultrafine material which makes up a large part of most plastic clays and the coarser fractions (whether natural silt or milled material) which are generally about 10μ and upwards. The following data given by Worrall (1982), show the size distribution of standard grades of China clay (Table 2.2)

Table 2.2.

Particle Size Distribution of Kaolin

Type of Clay	Particle Size(%)		
	Below 1μ	Below 2μ	Above 10μ
Porcelain clay	61	73	5
Bone china clay	36	47	13
Sanitary clay	20	31	23
Earthenware clay	30	43	24

Harman and Fraulini (1940) investigated the properties of size fractions of a "pure" kaolin and found that base exchange capacity, heat of wetting and drying shrinkage, although vary with particle size, are not simple functions of it but they are linear functions of each other. The deduction was then made that the larger particles are aggregates and therefore, have a corrugated surface of larger area than a sphere of the same radius. This non-linearity of clay, noted Singer and Singer (1963), makes it imperative to control the sizes and distribution in an industrial product as variations will give unpredictable results.

In the interest of uniformity in case slight changes occur in the composition of the clays ("In all layer-silicate deposits, horizontal and vertical variability occurs to a greater or lesser degree, creating a problem for the ceramic manufacturer's intent on producing products of uniform quality", Heystek, 1978), Rhodes (1973) recommends the use of several kaolins in a particular clay /body/. Today the trend is to use materials

guaranteed by suppliers to conform to specified compositions and particular ranges of physical properties. (Heystek, 1978).

Kaolin for electrical porcelain should have a low-mica content - about 12% - analysis calculated on the Mica convention. (Waye, 1967). Mica has been observed (Singer and Singer, 1963) to have a marked effect on the firing and physical properties of porcelain. During firing, the mica expands when the mica-free body would contract thus retarding the vitrification, attainment of the final specific gravity and some other desired properties. This leaves the friable biscuitware more porous and weaker. In a related view, Worrall (1982) postulated that "high contents of mica tend to give a high contraction, probably because the associated alkalis produce a high proportion of melt at the firing temperature." Waye (Ibid) put the fluxing power of mica at about 0.4 times that of feldspar.

Owing to their low cation exchange capacity (Worrall, 1982), kaolins in general require less deflocculants than ball clays and are more sensitive to over-deflocculation.

The hypothesis by Waye et al (1963) that the large differential shrinkage between diameter and length (recorded for the extruded samples used in their investigation), was probably due to the alignment of kaolin particles during extrusion has support in Robinson (1978) who explains that "the process of extrusion causes particles of flaky habit to orient parallel to the faces of the die. This orientation causes a greater shrinkage

in the direction perpendicular to extrusion than in the lengthwise direction. "Robinson (Ibid), further surmises that the orientation may cause a differential expansion during the removal of the last 2% of water from an extruded material.

Kankara clay is a primary clay occuring in commercial quantity in Kankara, Katsina State. Hendsman (1974) analysis of Kanakara clay is:

Silica	SiO_2	44.55
Alumina	Al_2O_3	31.45
Iron Oxide	Fe_2O_3	1.07
Magnesia	MgO	0.87
Lime	CaO	0.28
Sodium	Na_2O	0.17
Potassium	K_2O	0.30
Excess H_2O	H_2O+	12.03
Chemical H_2O	H_2O	9.06
Titania Oxide	TiO_2	0.41
Manganese	MnO	0.01
Sulphur	SO_4	0.05

For the same Kankara kaolin, Nwankwo (1985) gave:

	SiO	48.52
	Al_2O_3	17.01
	Fe_2O_3	0.80
	Na_2O	0.30
	K_2O	0.19
Fe(total)		0.56
Ignition Loss		14.15

Ball clays on the other hand, are clays transported far from their original place of formation. During transportation, particles of such clays are reduced by mechanical abrasion. This movement also accounts for the relatively high impurity level and the chemical composition variations observed in such clays. (Table 2.3). As indicated by Rhodes (1960), the specific gravity of plastic clays varies from about 2.5 to 2.7, according to the impurity level. According to Rhodes (1960), ball clays are often described as aluminous (containing 30 to 40% alumina) or siliceous (containing 60 to 80% silica).

Table 2.3

The Variation of Chemical Composition of Ball Clays

Oxide	Range of Variation %
SiO_2	40–60
Al_2O_3	25–40
Fe_2O_3	0.25–4.0
Na_2O	0–0.75
K_2O	0.5–4.0

Source: Worrall (1982)

Worrall (1982) stressed that if certain deposits exist outside the limits given in table 2.3, then such deposits cease to be regarded as clays and fall into the category of sands or loams. He further opined that the wide variation that exists in the composition

of ball clays implies that categorization can only be by location and geological age. This view, however, appears to be at variance with Rhodes (1960) "aluminous" and "siliceous" categorization for ball clays. Since both views are tenable, a suitable categorization, will be a hybrid of the two approaches.

Bomo clay from Zaria city in Kaduna State is a dark-firing ball clay. It has been used as a plasticizer in bodies for porcelain slip casting (Okoruwa, 1986), and bone china (Umar, 1987).

As plasticizers, ball clays are added to otherwise comparatively non-plastic bodies. As noted by Waye (1967), discretion is needed in making such additions because, too low plasticity in a body may give cracks during severe working of the clays and low dry-strength resulting in high handling losses. Conversely, too much of the fine material in a body may cause undue slowing down of filter pressing and high drying shrinkage. Rhodes (1960) observed that the degree of translucency of porcelain decreases with increasing amounts of ball clay.

Worrall (1982) is of the view that as a result of their high cation exchange (and depending on the percentage of organic matter present), a considerable amount of deflocculant is needed to disperse the particles of ball clays. According to him, the presence of soluble salts in the clay, if in excess of about 0.5% would inhibit deflocculation.

For electrical porcelain, control tests conducted on clays include drying and firing shrinkage, dry and fired

strength (sometimes using a 50:50 clay:quartz mixture, Waye, 1967), vitrification, and less frequently chemical analysis and grain size determination. The base exchange values are determined on a routine basis when slip casting is to be used.

2.3.2. Silica

Following Cardew (1969), free silica, as opposed to the combined silica in rock-forming materials is found in nature in four forms: Microcystalline Silica: quartz, tridymite, and crystobalite; Microcrystalline Silica: flint, chalcedony, agates; Amorpheus Silica: diatomite; and Silica Glass caused by the fusion of sand when struck by lightening.

According to Waye (1967), silica could be introduced into an electrical porcelain body as ground quartz such as white sand or quartzite or, less frequently, as flint. It is also introduced to a varying degree by other raw materials such as clays, pegmatites, and feldspar sand.

In an earlier study, Hirsch (1926) investigated the effect of using different materials for introducing the quartz into the body, the body composition being Clay 50; Quartz 30; Feldspar 20. In place of quartz, the researcher used different pure or mixed clays together with 4 types of rock quartz; 3 sands; flint; raw and calcined; sand by - product from washing kaolin. While firing temperature, shrinkage, fired colour and water absorption were little affected throughout, expansion

coefficient, being partly affected by particle size, was irregular. It was discovered that the true quartz undoubtedly gives the best result for transparency. Calcined flint and one sand give best values for ball compressive strength. Impact bending strength improves with the kaolin washing by-product, tensile strength is enhanced by the washings sand followed by calcined flint while, puncture strength is best with calcined flint and sand and least with raw flint.

Selection of flint and feldspar for porcelain body (Rhodes, 1960) must be considered not only from the standpoint of chemical purity and freedom from iron, but also for their exact firing temperature. It should however be noted here that commercial flint is prepared from various types of quartz and not necessarily from the mineral flint.

Okene, rock quartz is found in Kwara State, while Baure quartz sand exists in large deposits in Baure, Katsina State. Two analyses are presented for Baure quartz sand. The analysis given by Ajayi (1985) is:

SiO_2	99.4%
Al_2O_3	0.6%
Fe_2O_3	0.01%

A slightly different but more detailed analysis by Nwankwo (1985) is:

SiO_2	96.91
Al_2O_3	0.63
Fe_2O_3	0.80
Fe	0.56
K_2O	0.19
Na_2O	0.19
L.O.I	0.42

Pure quartz, with a specific gravity of 2.65 at 20°C (Worrall, 1982), occurs as transparent crystals and has a slightly cramped framework. Cardew (1969) has observed that "heat increases the activity of molecules and (in crystals) loosens the rigidity of the inter-atomic bonds" with the increased mobility resulting in thermal expansions. In the ceramic industry, these linear expansions are measured as percentages over a given range or as the coefficient of linear expansion (i.e. expansion in unit length per degree Celsius).

The three silica polymorphs; quartz, tridymite and critobalite, each has a low (alpha) and a high (beta) modification. According to Singer and Singer (1963), the change from one to the other, which is quite sharp, is reversible because it is not a radical change involving bond breaking and rearrangement of the atoms but merely a change of bond angles and slight adjustment of inter-atomic distances.

Alpha quartz heated to 573°C. or over, will see the Si-O-Si bonds straighten somewhat resulting in the atoms becoming less closely packed with a marked expansion

(quoted as I.03 mean linear per cent by Cardew, 1969). The practical implication of this expansion are: the failure of a kiln at around this point can lead to severe shattering of wares (Fournier, 1973); In a body containing a high proportion of free silica of fine particle size, this is the time when cracking will take place if the temperature is increased too quickly (Cardew, 1969), the finer the particles, the greater the danger of cracking will be. Rhodes (1973) stressed that this change of volume in the quartz portion of a clay body, although rather slight, must be accomplished slowly. He points out that if one part of a ware in the kiln is heated up faster than another, the unequal expansion in the piece may also cause it to crack. Likewise, care is needed in cooling so that the contraction at this critical point is passed safely.

Similarly, cristobalite heated over temperature range of 220 - 280°C (this variation is probably caused by the differences in the degree of crystal structure, Ford, 1967, Worrall, 1982), has an inversion from alpha to beta which differs from that of quartz. Green (1963), reports that at 220°C., the beta cristobalite in the clay suddenly reverts to alpha cristobalite with a decrease of volume of 3%. When this contraction takes place, the glaze is already set hence, it is put into compression at a point referred to as the "cristobalite squeeze". However, if the strain set up between the clay and glaze is too much, the glaze is put in tension and must crack or "craze". It then follows that for maximum

durability and fit, a glaze should be in slight compression over the body.

In cases where firing is carried to a point where the free silica in the body goes into glassy melts with the other materials, there is no crystalline change upon cooling and as such, there is no loss in volume to put the glaze into compression. It was observed (Fournier, 1973), that "most of the silica in a glaze and, to a lesser extent, in the body will unite with the bases to form silicates in which case the reverse beta to alpha inversion does not occur." Stoneware and porcelain glazes at 573°C. and all glazes at 270°C., have become rigid; they have passed below their annealing temperature range (between 800°C and 650°C). Containing no free silica in crystalline form, the glaze itself is unaffected by the changes. But the body to which it is welded if it contains any quartz or cristobalite, will suddenly shrink and put the glaze into a state of compression. (Cardew, 1969).

To control crazing which is caused by a high expansion and contraction in the glaze relative to the expansion of the body, the inversion of quartz at 573°C is used to, as it was, narrow the contraction differential.

Green (1963) suggests reducing the contraction of the glaze itself by changing its composition by the addition of silica. He explained that "in a glaze, all the silica is dissolved by the fluxes and there is no question of cristobalite being formed at all. Some fluxes such as

potassium oxide and sodium oxide do cause considerable shrinkage in a glaze", so that when silica content is increased it reduces the effect of the offending oxides by reducing their proportion. Green (Ibid) advised that if an additional 6% does not stop the crazing some of the fluxes must be changed for boric oxide or zinc oxide.

Cardew (1969) talks of other rearrangement processes which occur between the inversion point of quartz (573°C) and the melting temperature of quartz (usually quoted as 1713°C). He calls these reconstructive changes because, here, bonds are broken resulting in a more open structure as reflected in the volume increase and the lower specific gravity of the new forms: quartz 2.65; cristobalite 2.32; tridymite 2.26. According to him, the non-quilibrum change is/of more practical interest in pottery firing because it involves a direct change from quartz to cristobalite which begins at as low a temperature as 1025°C. These new forms of silica induced by heat will keep their new shape on cooling. Ford (1967) listed some variables which determine the rate of conversion of quartz as: the temperature; the grain size; the nature of the quartz; and the fluxes present.

Fine quartz will not necessarily convert more rapidly than coarse quartz. Ford (1967) showed that in many cases if the degree of strain in the quartz crystals is high, the conversion rate of very coarse

quartz will be so great as to cause shattering in firing.

Apart from opening the body and decreasing both plasticity and shrinkage, improving resistance to warping and improving the receptivity of the body to glaze, the presence of quartz gives hardness and stability to the final product. In its powdered form, inhaled quartz lodges in the lungs and causes silicosis. Measuring the level of dust in the air is a recommended practice to compare with the Threshold Limit Value (TLV) for Crystalline silica. TLV is Described as that concentration of dust to which it is believed that all workers may be repeatedly exposed without adverse effect.

If quartz sand is to be used, a specified finess of about 50-55% less than 0.01mm is recommended by Waye (1967) who further suggests determination of the finess by the Sedimentation (Hydrometer) Method for routine control.

2.3.3. Feldspar

Optical examination of a fired porcelain insulator and, as a broad generalisation, fired heterogeneous clay bodies, reveals the most obvious reactions to be that part of the body softens and surrounds the more refractory grains and, by capillary and surface tension forces, partly dissolves them together (Singer and Singer 1963). Cutler (1978) contends that additives that form a second phase without forming a liquid

inhibit the densification process while the liquids appear to increase densification. A familiar example of the latter is the case of porcelain where the feldspar contributes a liquid phase that promotes densification.

Feldspar in a body lowers the temperature of liquid formation during firing. On cooling this liquid forms a glassy matrix which brings the grains of the body together. Since the aim is not to produce more glass than is necessary, care is taken in making such additions. Worrall (1982) argued that though feldspars, within limits lower considerably the softening point of any alumino silicate (e.g. clay) to which they are added, the amount will depend to a first approximation on the number of equivalents of Na_2O,K_2O, or CaO that have been added. The fluxing power of alkali-bearing minerals therefore, depends on the molecular proportion Na_2O. CaO or K_2O contained therein.

There are twelve types of true feldspar and many other feldspathic materials called feldspathoids. The twelve true feldspars can be separately identified by chemical composition and will, in carefully selected specimens correspond to idealized formulae. (Hamer, 1975).

The general properties of feldspars are their cleavage into angular pieces with hexagonal tendencies, polarized reflections at the fractured edges, hardness (6 on Mohs Scale), and sometimes, colour. Some feldspars, noted Hamer (1975) show signs of plasticity like clay and similarly react to flocculation.

According to Rhodes (1973), commercial feldspars usually have higher amounts of silica and alumina than the theoretical composition, and are often a blend from different parts of the same quarry or from different quarries, to keep the material uniform in composition.

The most common feldspars are orthoclase, anorthite, and albite. As indicated by Fournier (1973), feldspars consist of the alumino silicates of potassium, sodium, calcium and, more rarely, barium. The theoretical formulae are: Potash feldspar (orthoclase) $K_2O.Al_2O_3.6SiO_2$; soda feldspar (albite) $Na_2O.Al_2O_3.SiO_3$; lime feldspar (anorthite) $CaO.Al_2O_3.2SiO_3$. Feldspars are named according to the predominant flux.

The observed physical properties of both Okene and Zaria feldspar seem to justify their categorization as potash feldspar. Nwankwo (1985) analysis of Zaria feldspar would seem to negate this classification. The analysis is:

SiO	64.71
Al_2O_3	14.49
Fe_2O_3	0.80
Fe	0.56
K_2O	0.67
Na_2O	0.76
LOI	0.39

Singer and Singer (1963) contend that feldspar is the most convenient form for introducing nearly insoluble

alkalies because the alkalies in massive feldspar are not water soluble though fine grinding, nevertheless, releases some of the alkali. The alkali content of the feldspar will be some-what lowered by wet grinding followed by filter pressing and drying. A dry ground feldspar will, on addition to a wet batch, release free alkali and alter the pH (i.e the relative alkalinity).

Williamson (1978) propounded that dry strength of extruded pieces may have higher values still if the interstitial liquid in the plastic mixture contain dispersants - these promote more intense particle parallelism and closer particle packing - deliberately added or which may have resulted from reactions between the interstitial liquid and solid particles of feldspar, nepheline syenite, glass and so forth, which release alkali metal ions.

Following Singer and Singer (1963), electrical conductivity is lowered by complete exclusion of sodium replacing it with potassium. Power factor (i.e. a measure of the energy lost when an insulator transforms part of the electrical energy into heat) rises with the alkali content and with any porosity that could absorb humidity. Sodium feldspars, due to their premature decomposition with vaporisation of the soda, can give rise to porosity. Carbonaceous matter can also lead to pore formation. High-Soda feldspars have been observed (Singer and Singer, Ibid) to reduce vitrification in a body leaving such bodies susceptible to warpage during firing.

Irabor et al (1987) concluded that certain feldspars of

the soda variety and those with an appreciable mica content are unsuitable for production of porcelain electrical insulators. The presence of mica in the body was found to create micro-pores in the insulator thereby reducing its physical and electrical insulation properties.

While commercial high potash feldspars melt in the 1100 to 1200°C. range, pure potash melts incongruently at about 1150°C to yield crystals of leucite and a silica-rich liquid of high viscosity which makes the ware translucent without collapsing. The high viscosity of potash feldspar is illustrated in the work of Morey (1934) who heated a mixture of the composition $K_2O.Al_2O_3.6SiO_2$, with intermediate cooling and grinding when a spongy sintered mass resulted. Heating was continued until the platinum crucible containing it melted (melting point of platinum is 1773°C). No molten homogeneous glass could be obtained, thus 600°C above the temperature at which melting began and 200°C above the temperature of complete fusion, the viscosity was still so high that the mass could not flow together.

In selecting a feldspar, Rhodes (1960) and Waye (1967) recommend the use of a fusion button fired to glaze temperature during which the feldspar should fuse to a smooth, white, glassy mass. If reducing fire is to be used for making the ware, the feldspar should not show any sign of gray discoloration from carbon as a result of the fire.

Waye (Ibid) proposed that, although grain size is not quite so important as in the case of quartz, it should be controlled within the usual range of fineness of quartz. Control of its chemical composition is important because, variations in alkali content and also in the potash/soda ratio may have important effects on firing behaviour and fired properties. Apart from melting at different temperatures, potash and soda feldspars form eutectics of different melting points with other body constituents such as silica and metakaolin.

Feldspar is sometimes introduced in form of feldspar sand and, occasionally, pegmatities and nepheline syenite are used. For product standardization as far as the fusibility and colour are concered, a fusion button test involving a sample of each batch fired together with a standard sample is a suitable method of a routine control (Waye, 1967)

2.3.4. Talc

Raw talc, variously referred to as "soapstone", "French chalk", - the term "Steatite" is reserved for the massive form - is a magnessium silicate with the formula $3MgO.4SiO_2.H_2O$. It has a greasy feel and is not easily wetted. Its specific gravity and hardness on Mohs Scale, according to Singer and Singer (1963) are 2.6-2.8; and 1-2 respectively.

The colour of talc varies from white to light green. Woakes (1988) opinion on the variation in colour of talc

outcrops is that it is indicative of compositional disparities: those bearing tremolite and/or chlorine tend to be greenish; outcrops that are largely talc are whitish and may be massive. He further opined that "those that are rich in diopsidic pyroxane or tremolite would be less desirable for ceramics since enhanced lime content reduces firing capabilities." In their own argument, Hauser and Gunzenhauser (1948) held that the important requirements for talc is that it should be "soft, substantially free from foliated, flaky or fibrous material and from gangue which produces darkspots when fired." The colour of the raw material, they continued, is of no importance as long as it fires white after heating to 1350°C in an oxidising or neutral atmosphere.

Talc is an idealized mineral which in nature has according to Hamer (1975), a variable formula of proportions and nearly always contains some calcium, iron, and alumina. Petramin (1988) contention is that of the world reserves, only a small percentage could be regarded as "pure" and that talc is very difficult to purify by gravity seperation, but, as noted by Singer and Singer (1963), it lends itself favourable to froth flotation methods: it floats readily with pine oil and also with creysylic acid or alcohol. Petramin (1988) interpolated that when pure talc products are reprocessed by froth flotation or similar selective separatory methods, very high purity talc mineral can be produced.

Waye (1967) estimates the percentages of MgO and SiO_2

in a good quality talc to be about 51-53 and about 57-62 respectively with about 5% ignition loss. Typical chemical analyses of some talc ores and products are given in Table 2.4:

Table 2.4:

Typical Chemical Analyses of Talc Ores and Products

	Pure Talc (Theoretical)	1	2	3
SiO_2	63.36	59.16	59.80	61.89
MgO	31.89	31.34	27.45	28.02
Fe_2O_3	-	3.36	0.05	5.88
TiO_2		-	-	Trace
Al_2O_3	-	0.26	0.57	0.98
C_3O	-	0.15	6.80	0.50
K_2O	-	-	-	Trace
Na_2O	-	-	-	Trace
CO_2	-	1.76	1.18	-
H_2O	4.75	4.30	-	2.17
MnO	-	-	0.39	Trace
S	-	-	0.07	-
NiO	-	-	-	0.22
Cr_2O_3	-	-	-	0.34
CaO	-	-	-	Trace
FeO	-	-	0.15	-
LOI	-	-	4.75	-
	100.00	100.32	101.21	100.00

Source: Petramin (1988)

1. Flotation talc, Johnson Mine Vermont.
2. Average talc Ore, Talcville, Gouverneur District, New York.
3. Petramin Talc, Nigeria. (Brink Jones)

Commercial "talcs" may or may not be predominantly talc, as associated minerals such as calcite, dolomite, quartz, magnesites and chlorites are common (Heystek, 1978).

Singer and Singer (1963) indicated that vitrification range of talc is around 1270° to 1350°C., with a fairly sharp melting point at about 1500°C. When talc is heated, it undergoes decomposition and several transitions. The temperatures at which such changes occur will however depend on such variables as the impurity levels and the fineness of grinding. Ford (1967) showed that dehydration of talc yields the equilibrum products, enstatite and silica with the silica content of the dehydrated mixture very close at 66.7% to that of the eutectic between enstatite and silica at 65%, so that a large amount of melt is produced at the eutectic temperature of 1543°C.

Suitable quantities of talc or steatite introduced into certain ceramic bodies have been observed (Wetzel, 1925, Singer and Singer, 1963,) to impart a number of advantageous properties: where talc is the major constituent - 70 to 90% - a highly suitable electrical insulating body type known as 'steatite' is produced. Similarly, where the body composition approximates to that of Cordierite ($2MgO.2Al_2O_3.5SiO_2$), bodies of low thermal expansion and, therefore, high thermal shock resistance together with good electrical properties are obtained. In smaller quantities, the fluxing action of

the magnesia gives the highest value for the impact bending strength, modulus of elasticity, low coefficient of expansion, and a lowering of maturing temperature.

Singer and Singer (1963) suggested a 3 - 4.5% addition of talc previously fritted with the feldspar This view is shared by Alvarez-Estrada (1979) who talked of porcelain pastes that have been put into manufacture with additions of 3% talc and 2% zinc oxide. Such bodies, he observed, mature at 1290°C with little deformation, good transparency degree, good whiteness and strength to bending and shock. A lowering of maturing temperature by 100°C was also recorded.

The fluxing action of talc was explained by Hamer (1975). According to him addition of talc increases resistance to thermal shock because it produces a flux low in expansion rate. This flux involves the free silica left from mullite formation and the silica introduced in the talc. In this process, magnesia is used as a flux and not as a catalyst. The free silica is thus involved in a melt which, due to its magnesia content, is a viscous melt and not one so destructive to the ware as fluid/silica melts. By becoming molten the silica, whether quartz or cristobalite, loses its crystalline nature and becomes of low thermal expansion.

The alumina, iron and titania contents have been observed to affect the vitrification, final microstructure and electrical properties. An ignition test was suggested by Waye (1967), in detecting any specking

(due to large particles) and colour changes (which may indicate changes in iron content.) Very large platelets cause lamination during extrusion. Waye (Ibid) reported that some manufacturers take recourse to calcination of the more platey talc before use.

2.4. Body Formulation

Three canons emphasized in preparing a body for electrical insulation are: freedom from contamination: accuracy in batching; and homogeneity.

The compositions given in table 2.5 below will not enjoy universal applications because, raw materials vary and with them the properties of the ware manufactured. Rational Analysis, introduced as a basis of comparison for materials, does not always give a true picture of the constitution of a clay. Apart from the fact that it operates from an arbitrary point, the assumption of a definite chemical formula for a given mineral is misleading. Other variations from the compositions given (table 2.5) will be determined by firing temperature and facilities available.

Table 2.5

Typical Recipes for Electrical Porcelains

	ENGLISH		GERMAN		U.S.A	
	21c	20c				
Clays	25b	35b	40	61	37	53
Feldspar	34	25	11	10	23	24
Pegmatite	–	–	44	15	–	–
Felspar Sand	–	[illegible]	5	15	–	–
Quartz	20	20	–	–	33	21
Whiting	–	–	–	–	2	2

Source: Waye (1967)

Key: c – Kaolin
b – Ball

According to Waye (1967), the quantity of clay to use in an electrical porcelain requires a compromise between having insufficient to give desired plasticity and having so much that the resulting excessive drying shrinkage could cause cracking during drying and distortion in firing. More than 40% of a plastic clay is dangerous in this respect and the total clays are kept under 55%: a preponderance of kaolin, apart from giving more rigidity during firing, also increases the firing range (around 50°C for porcelain) over which vitrification occures. A high percentage of the ball clays at the expense of the kaolins, on the other hand, would lower the firing range.

Feldspar content kept below about 25% would require

rather high firing temperature while feldspar content in excess of about 35% may give difficulty during firing and an excessive amount of glass which would be inimical to the thermal shock resistance of the ware.

As a filler, the silica reduces the total shrinkage and imparts rigidity to the body during firing, so that the original geometrical form is preserved; although on a small scale (Davidge, 1979). Though the effect of quartz on firing temperature and range will depend on the type and quality of other components present (Waye, 1967), it should be noted in passing that if in excess, say 40%, the ware becomes more subject to dunting or cracking on cooling.

The discourse so far would seem to justify Waye (1967) approximate limits of 40-60; 20-35; and 20-40, for clay substance; feldspar; and quartz respectively.

2.5. Body Preparation

Air-floated ball clays do not require any preparation and may be added to the body mix as they come. (Rhodes, 1960). Rhodes (Ibid) recommended that lump ball clays should be first blunged with plenty of water to a thin fluid slip, then 150 - mesh screened to remove all particles of grit, lignite or other foreign matter. The slip is then dried and the clay is ready for use.

The industrial solution to the problem of iron impurities which usually get into the clay during mining or transportation, is running the slip over a powerful magnet (magnetic filters) which attracts and holds the

metallic impurities.

A calcination temperature of 900°C was used (Okoruwa, 1986) for both quartz and feldspar followed by jaw crushing and ball milling for 24 and 18 hours respectively. Grinding times for materials have been found by various investigators to depend on the following variables; operating speed of the mill; size of the mill; hardness of the material.

Correct granulation has been identified (Waye, 1967) as being vital to the whole process and must be strictly controlled in order that the fired product, apart from conforming to close dimensional tolerances, be of uniform density both from piece to piece and within each piece, have zero open porosity and also sealed porosity (therefore high bulk density) give high mechanical strength and acceptable electrical or magnetic properties.

Cutler (1978) said:

> The theory makes it clear that particle size is a very important variable in the densification process. The rate of densification, regardless of which theory or equations and mechanisms are employed, is inversely proportional to particle size for a simple viscous system at any temperature and inversely proportional to particle size to the 1.5 volume for volume diffusion. This means that the smaller the particle size of the material, the more active the powder will be toward sintering.

On the other hand, (Cutler, 1978), using fine particle sizes (less than 0.1 microns) decreases the green density of particle compacts of very fine powders and increases

sintering shrinkage.

The particle size of feldspar, explained Singer and Singer (1963), has considerable influence on the reaction occuring during firing. If too coarse, insufficient homogeneity leads to centres of pure feldspar glass, while overground feldspar reacts too fast with the kaolinite before the resultant liquid attacks the quartz and leads eventually to clusters of mullite crystals unevenly dispersed in the body hence, medium grind gives an even network (see table 2.6).

Table 2.6

Proportion (volume %) and Particle Size of Materials in an Electrical Porcelain Body (After Dinsdale and Wilkinson, 1966)

Material		Approx. Median Size (μm)	Electrical Porcelain
Clay	Ball Clay	0.5	25
	China Clay	1.5	25
Flux	Feldspar	10	25
Filler	Flint	10	25

SOURCE: Davidge (1979)

The quartz particle size is very important as will be seen shortly. According to Singer and Singer (1963), if too coarse it does not dissolve in the fluxes. The transparency of the ware is reduced and finishing temperature is also generally raised. Kingery (1978) observed that "the rate of dissolution of the quartz grains is the slowest process taking place during chemical

equilibration". He was further of the view that as a result, for usual particle sizes, the quartz will completely dissolve to form a glass only after long high-temperature firing, but that "at lower temperatures, a solution rim that has very different thermal expansion characteristics forms around quartz grains and gives rise to stresses on cooling, often cracks, "Fine-ground quartz, on the other hand, (Singer and Singer, 1963) assists in the fluxing action of the feldspar, while overfine quartz, among other things, leads to crazing.

At this point it becomes necessary to examine the two theories - Mullite Hypothesis; Strain Theory - usually advanced to explain the observed fired properties of electrical porcelain, particularly, the mechanical strength.

Under the Mullite Hypothesis, the strength of porcelain is largely dependent on the interlocking network of Mullite crystals formed during firing. Protagonists of this view point (e.g. Rieke, 1925) present evidence to show that "high-clay bodies give higher strength than high-quartz bodies due to a decrease in internal strain as a result of the smaller difference in thermal expansion between the crystalline phase and the glassy phase as the formation of Mullite increases while the quartz content decreases."

Similarly, Mason (1957), Palatzky and Tummler (1958) indicated that progressive replacement of the quartz by porcelain grog ground to the same size results in a corresponding increase in strength. This again amounts

to a negation of the strain theory suffice it to say that under the strain theory, a reduction in internal stresses would be expected to result in a reduction in strength, even though a member of the opposing school (Winterling 1961) obtained even higher strength through substitution of the composition used by Mason (1957) and Palatzky and Tummler (1958) with size classified quartz for grog.

In a seeming subscription to the mullite hypothesis, Singer and Singer (1963) contend that since undissolved quartz is detrimental to strength, particularly tensile strength improvements can be obtained by replacing it with glost pitchers, calcined ball clay, alumina or zirconia.

Under the strain theory, the increases recorded in strength is ascribed to compressive strains imposed on the glassy phase by the greater contraction of the free quartz during cooling.

An exponent of this school, Mattyasovsky - Zsolnay (1957) failed to see any effect of Mullite on strength. He therefore, suggested the use of a low-kaolinite high-quartz body. He surmised that grains larger than about 30 microns were the more likely to crack resulting in lower strength, while grains smaller than 10 microns would be dissolved in the glassy phase resulting in a decrease in the pre-stressing and therefore, the strength. This then implies that maximum strength is achieved when particle grains are kept within the 10 - 30 micron limit.

Though they do not give a size limit, Kingery (1978)

and Davidge (1979) agree that the solution to making stronger electrical porcelain lies in using quartz particles of a finer size or, alternatively, in using a different filler such as alumina which "is more compatible with the matrix from an expansion point of view."

Waye et al (1963) investigated the properties of a conventional English type of electrical porcelain, in particular as a function of its composition and grain size of the quartz over a range of firing temperatures. Using the composition: blue ball clay 12.5% black ball clay 12.5% China clay 25.0%, feldspar 25.0%, quartz 25%. The approximate rational composition, using the Feldspar Convention for the clays was: 44%; 28%; 25%, for clay substance: feldspar; and quartz respectively. Nominally, the quartz was 50% below 10 microns and 55% below 10 microns for the feldspar. Determination by the Hydrometer method gave 52.3% less than 10 microns for the quartz and 47.3% for the feldspar, percentage of the quartz between 10 and 30 microns being 34%.

From the study by Waye et al (1963), it was revealed that grinding quartz for the excentionally long period of 36 hours results in 15% reduction in strength. It was assumed that this decrease was as a result of lower-maturing temperature of the finely ground material resulting in either overfiring or excessive solution of the quartz grains or both. It was also discovered that strength increased with increasing quartz content even

though the body with a high clay content had the highest strength of all.

Waye et al (1963) then proposed that a satisfactory alternative to the strain theory would presumably have to ascribe the observed increase in strength with diminishing grain size of the quartz (down to a certain limit) "to a decrease in the localisation of stresses." They were further of the view that it would appear plausible to expect that small grains would be less likely to be associated with cracks or that such cracks would be less damaging than those associated with large grains. The decrease in strength recorded when the fineness of the quartz exceeds the optimum, they concluded, could be due to the inhibition, by dissolved quartz, of the development of mullite in the glassy phase.

It is sometimes claimed that grinding the quartz and feldspar together gives stronger products with lower thermal expansion coefficients. (Waye, 1967).

Wet milling is generally preferred over dry mixing which is usually restricted to bodies to be shaped by pressing. Somansundaran (1978) adopted the views of Lin and Mitzmager (1968) in ascribing the efficiency of wet grinding to a "reversible reaction between unsatisfied surface bonds and water molecules." Water, in the form of vapour is also expected to produce such "hydrolytic corrosive effects". Somansundaran (Ibid) listed some physical reasons responsible for the increased efficiency of wet grinding. According to him; "Cushioning effects

due to the presence of a bed of fines" will be less in wet grinding ("where the particles tend to remain suspended in the water") than during dry grinding; Effects of viscosity and specific gravity of the medium.

In considering the amount of water to use in milling the 11 to 18 percent of the jars cubic capacity given by Cardew (1969) will serve as a reference point. During the milling of some materials (e.g. feldspar), some alkali is dissolved. The dissolved alkali deflocculates the suspension and increases the fluidity of the mixture. Such materials therefore, require less water than that required for materials that do not release deflocculating substances.

2.6. Forming

The methods of manufacture for electrical porcelain insulator, according to Irabor et al (1987), are: lathe turning, dust pressing, plastic/hydrostatic pressing and slip casting. For lathe turning and plastic pressing, the body is passed through a de-airing pugmill or allowed to age for several days or, sometimes, weeks, before use. In dust pressing, the material is pressed at between 10% - 15% moisture content. For plastic pressing in dies or rubber moulds, the body should have good plasticity and about 25 - 30% moisture content.

2.6.1. Slip Casting

Casting is perhaps the best method of forming porcelain because it does not depend on extremes of plasticity in

the clay (Rhodes, 1960).

Phelps and McLaren (1973) said:

> Casting slips are fluid suspensions of one or more particulate ceramic materials dispersed in a liquid, usually water, at high solids volume by deflocculating agents. A casting operation involves consolidation of suspended solids into a semirigid mass through removal of a portion of the liquid by an absorbent mould. Casting slip suspension particles are normally predominantly non colloidal (less than 0.5μm), although some colloidal material must be present to ensure slip stability and good rheological qualities.

Deflocculation is achieved by adding to the suspension water, suitable soluble alkalis which exchange their ions (charged atoms) with those of the clay particles so that the clay particles are all of similar "electrostatic charge." (Hamer, 1975).

Worrall (1982) contends that as long as the zeta potential (i.e measure of the energy of repulsion of the particles) is high; of the order of 50 mv or more, the particles repel one another so much so that they do not come together no matter the proximity of the particles. They slide past one another without making contact to form "flocs." The zeta potential, continued Worrall (1982), is determined to a great extent by "the type of exchangeable ions present on the clay".

Hamer (1975) classified deflocculants into;

(i) Alkali cation type which "promotes cation exchange between itself and the clay particle with the new cation increasing the zeta potential

of the particles." Alkali cation type deflocculants include sodium carbonate, potassium carbonate, sodium hydroxide, and sodium oxalate. It should however be noted in passing that there is a limit to the deflocculatory effect which can be achieved by cation exchange;

(ii) Polyanion type which, while "providing cations for the outer layer" as does the alkali cation type, enters the inner layer where "an anion exchange is effected by the polyanion half of the deflocculant replacing the anions on the clay particles. Polyanion deflocculants include sodium silicate, sodium poly-phosphate, sodium-acrylate and sodium tannate.

Wunnik (1962) is of the view that soda ash should be used only as an initial addition to activate the organic colloids, while adjustments should only be made with sodium silicate.

Singer and Singer (1963); Hamer (1975); and Worrall (1982) recommended the use of a combination of sodium carbonate (cation exchange) and sodium silicate (polyanion) because, apart from giving full control, precipitates from this combination are not harmful both in the suspension and the fired ware. Norton (1956) suggested 1gm of deflocculants to 125gm of solid ingredients and that variation from this should not exceed 1.7gm to 125gm of solids.

Effective deflocculation is usually affected by the

following; soluble salts such as calcium sulphate, $CaSO_4$ (in the form of gypsum) in which case Ryan (1978) and Worrall (1982) suggest treating the clay with the requisite amount of barium carbonate, $BaCO_3$ (about.02 to .01 per cent by weight of the solids) to precipitate barium sulphate and calcium carbonate which are less soluble and do not interfere with deflocculation; Contaminated water which would have to be specially demineralised. (Singer and Singer, 1963); Slight fluctuation in moisture content (Rhodes 1973, Okoruwa, 1986); Some deflocculants (e.g. soda ash), when exposed combine with airbone water to produce sodium hydroxide - a flocculant - which will cause the clay to form lumps and sediment; livering. (Hamilton, 1974).

Barta (1960) according to Okoruwa (1986), gave a castable range of densities of industrial porcelain slips of 1.52, 1.67, and 1.77g/cm^3. Okoruwa (Ibid) also indicated that Norton (1956) had earlier given specific range of about 1.70 to 1.80 for porcelains. While recommending a specific gravity of 1.70 for porcelain bodies, and 1.40 for glaze slips, Okoruwa (1986) opined that "the specific gravity determination process will always produce slip of right amount of water which controls the level of fluidity, viscosity, thixotropy and rheology since all have profound effect on casting rate".

For a body of composition:

China clay	20lbs
Ball clay	40lbs
Cornish stone	25lbs
Flint	10lbs
Whiting	5½lbs
Grog 60s/dust	40lbs
Distinguishing stain	2oz
Sodium silicate 140° Tw	2 fluid oz
Sodium carbonate	2 oz
Water	40lbs plus

Hamer (1975) suggested that approximately half the water be put into a blunger and approximately a quarter of the clay added. This is blunged for a few minutes while the sodium silicate and sodium carbonate are dissolved in very hot water. About a quarter of this solution is added to the mixture and blunged for about five minutes. The remaining water is now added together with the distinguishing stain which has been mixed with hot water and passed through a fine sieve (120s to 200s). The remainder of the clay and the deflocculant solution are added and blunging continued for about half-an-hour. The slip should now be extremely fluid. The cornish stone flint, and whiting are now added and the whole slip blunged for another half-an-hour. It is now sieved through a 120s mesh. The grog is screened separately and added to the slip and mixed in thoroughly. The fluidity of the slip can be adjusted at this point by adding still more water.

Slip cannot be stored indefinitely, but if the storage tank is non-corrosive and airtight, it can be

stored for about 20 days. A skin forms on the surface of the slip where it meets the air during storage. Hamer (Ibid) stressed that the skin must not be stirred into the slip because it contains carbon dioxide from the air which, due to its flocculatory effect, spoils the fluidity of the slip. He indicated that a layer of oil on top of the slip will prevent this neutralization.

Plaster of Paris also known as calcium sulphate hemihydrate, is prepared by heating gypsum between 120° and 160°C. It is used for the making of moulds for slip casting. (Worrall, 1982). The 130s mix (i.e 130 parts by weight of plaster to 100 parts by weight of water) is used for casting moulds.

To calculate amounts of water and plaster of Paris powder required, Hamer (1975) suggested estimating the volume of space to be filled by measurements. This volume, multiplied by 5 and divided by 7 will give the volume of water that will be required. The volume of water is taken as the unit from which the weight of plaster is calculated for 120s, 130s or 140s mix.

To mix the plaster, Hamer (Ibid) suggests sifting the measured amount of powdered plaster through a 20s mesh before sprinkling into the measured amount of water. The sprinkling is intended to give a thorough wetting to each particle. This is further ensured by allowing the mix to remain unstirred for one minute. A thorough wetting prevents formation of lumps. When stirring begins, it must be thorough and continuous, avoiding

frothing or entrapping of bubbles. Pouring time commences from the stirring to the time setting begins, and this varies with the mixture ratio. While a fresh powder in good condition will give 3 to 6 minutes of pouring time, an old plaster which has absorbed moisture from the air may give only one minute.

When slip is poured into a plaster mould, water is removed locally from the casting-slip by the capillary action of the pores (Worrall, 1982) and flocculation by the calcium sulphate (Singer and Singer, 1963) makes the body set.

Porcelain slips especially if little or no ball clay is used, will cast with rapidity which makes it difficult to gauge the thickness of the piece or to handle more than one mould at a time. (Rhodes, 1960). This rapid setting rate is usually retarded by wetting the mould. Phelps and McLaren (1978) found that once the specific surface (i.e colloid fineness)is reduced below some critical point, rate of cast will rise sharply.

The observation was made (Rhodes, 1960), that porcelain slips, being quite non plastic does not shrink excessively in drying, but in casting it does shrink rapidly at first and this may cause the ware to crack in the moulds, before it can be removed.

Drying of the ware is usually done in humidity dryers, chamber, or tunnel.Waye (1967) cautions against too rapid local drying and too high moisture gradient in the ware particularly in the thicker sections and bodies having a

high wet to dry shrinkage. Small parts are sometimes air-dried on racks and further dried at about 120°C. The trouble associated with this approach, however, is that such pieces tend to warp and, sometimes, crack if the rate of evaporation from upper and lower surfaces is uneven.

2.7. Glaze

Glazes on porcelain insulators help maintain adequate electrical resistance over the surface in humid conditions. According to Waye (1967), suitable once-fired glazing can give quite a large increase in mechanical strength due to the compression developed in the body during cooling and also to the filling in of scratches, small surface pores and other surface defects by the glaze. Singer and Singer (1963) had earlier observed that unglazed porcelain always has tiny irregularities in its surface which can offer focal points for incipient cracks. "Vanishing" of the surface, they said, raises the modulus of rupture 10 to 15%.

Rhodes (1973) reported that if a clay body is even slightly porous and moisture can enter it through exposed, unglazed surfaces, it will gradually take on what humidity is available. Such moisture, over a long period of time, brings about a slight hydration of the fired piece, which is attended by a slight increase in volume. This increase may be sufficient to make it craze (moisture crazing).

To test for the likelihood of moisture crazing, Rhodes

(Ibid) suggested subjecting the ware to steam pressure in an autoclave, which is the equivalent of extended time under normal conditions of humidity. A sample that is able to withstand 100 pounds of steam pressure for a period of two hours without crazing, is unlikely to ever craze in normal usage.

The actual tensile strength of the glaze, noted Singer and Singer (1963), is a determining factor in the extent to which the glaze can improve a body and the tensile strength of the glazes, in turn, depends on the composition of the glaze. They gave the ranking of minor glass consitituents in order of their favourable influence on the tensile strength (after Gehloff and Thomas, 1926) as:

(1) Boric oxide

(2) Barium oxide

(3) Alumina

(4) Magnesia

(5) Zinc oxide

Singer and Singer (Ibid) also referred to Thiess (1936) who investigated the influence of glaze composition on the mechanical strength of electrical porcelain and concluded that high contents of alkali and lime in the chemical composition of the glaze are detrimental to mechanical strength. Singer and Singer (1963), interpolated that the firing treatment will also affect mechanical strength. In this application, a porcelain glaze fired at 1410^{o}C (cone 14) for example may improve the mechanical strength by about 100%

whereas another at 1230°C (cone 7), will only record a 50% improvement.

Usually, industrial earthenware and bone china vitrify at the bisc stage hence, their glazes are mixed to thick viscous state which adheres to the ware by surface tension and requires to be dried in a warm air current. Stoneware and porcelain, when bisc fired do not vitrify; therefore, they absorb glaze by capillary action so that a very much thinner mixture suffices. It dries almost instantly as the moisture is sucked into the porous clay. (Green, 1963)

For a glaze maturing between 1200 and 1250°C, Waye (1967) gave a typical formula of:

0.6 CaO
0.2 K_2O
0.1 BaO 0.5 Al_2O_3; 5.0 SiO_2
0.1 ZnC (or MgO)

For outdoor applications, glazes are frequently coloured brown by the addition of calcined mixtures of nickel, zinc oxide, iron, chromium, and manganese. (Waye, 1967). Singer and Singer (1963) are of the view that a suitable addition of these staining oxides, particularly that of manganese, can increase the mechanical strength of the glazed piece, with the colour also playing a role in the thermal properties of the ware, an example being the "rate at which it will warm up."

Singer and Singer (1963) and Waye (1967) recommend making the portion of the glaze adjacent to the metal

fitting, slightly conducting. The reasons given for this are:

(1) To enable the insulator "distribute the voltage evenly over a string of insulators", thereby preventing flashover (i.e. electric discharge over the surface which short circuits the insulator);

(2) To "minimize radio interference".

A semi-conducting glaze is composed of mixed, calcined, and ground semi-conducting oxides such as iron oxide with manganese oxide. After applying the glaze, the insulator is fired under controlled reducing atmosphere during which some of the iron is converted to the ferrous state, resulting in the formation of a semi-conducting crystalline network imbedded in a normal stained glaze.

As observed by Waye (1967), apart from developing the necessary conductivity by the formation, on cooling, this network of conducting crystals, joined together in long chains by virtue of their being ferromagnetic, have the advantage that less of the oxides is required to produce the necessary conductivity. Otherwise, the glaze would become mechanically weak and inferior in surface finish.

A problem associated with use of semi conducting glazes, however, is that the oxide networks, at points where they are not fully protected by the glaze, are susceptible to "electrolytic action." Waye (Ibid), attributed this problem to the presence of dissimilar materials at the glaze surface.

To overcome this problem, he suggests the use of an engobe - a layer of coloured slip - under the ordinary glaze containing titanium oxide, which is reduced to a controlled extent to give conduction.

It has been indicated (Rhodes, 1960), that in the case of porcelain, the best engobe is usually the clay body itself. Porcelain bodies are known to have a relatively low shrinkage, A layer of slip put on over the damp or dried clay will therefore adhere without cracking or loosening. According to Rhodes (Ibid), a satisfactory slip can be made by taking the body composition and adding colouring oxides to it for the desired colour. If engobe is to be used on a leather-hard piece, or on a dry piece, its composition must be such that it will have considerably less shrinkage than the clay body being used for the ware. Where the application is to be made on a dry ware rather than on damp or leather-hard clay, Rhodes (1960) contends that the amount of plastic clays in the engobe composition may have to be reduced by adding some calcined kaolin at the expense of the china clay and the ball clay. Arriving at the right degree of shrinkage in the engobe for the intended application process, he conceded, will require some experimentation.

A very thick application of engobe usually results in cracks unless, of course, the engobe is carefully adjusted. Screening is usually sufficient to mix the ingredients of the engobe together with the colourants.

2.8. Firing

For sintering of porcelain and similar materials, temperatures above 1200°C up to 1400°C are required. The reason for this high temperatures is that sintering the raw batches of porcelain and similar materials to dense (non-porous) products needs a certain amount of molten phase. The latter must be formed during the firing process by decomposition, melting and reaction between the various components of the batch. In the usual composition, these processes only take place at temperatures above 1050°C. (Wiegmann, 1980).

The bisc firing (900 - 1000°C), of porcelain is to enable glaze to adhere to the body. In the firing circle, an oxidising atmosphere before commencement of vitrification - 900 to 1000°C - burns off organic matter. During the early stages of vitrification, - 1000 to 1350°C - the atmosphere is changed to reducing during which ferric iron is reduced to the colourless ferrous form. The formation of ferrous iron, according to Williams et al (1963), contributes to the fluxing of the body making "sealing" possible at temperatures lower than would be possible with only ferric iron so that the body becomes impermeable at about 1200°C.

Furthermore, oxidation of carbon and carbides deposited from the fuels at medium temperatures is prevented by reduction. Rhodes (1960) stressed that it is an observed fact that in reduction firing porcelain bodies have a long firing range and do not tend to bloat at as early a stage as in the

case of an oxidising fire. He interpolated that the ware does not seem to slump as readily either, and this may be as a result of the lack of combustion within the body of the ware itself.

The view has however, been expressed (Williams et al 1963; Waye, 1967) that insufficient reduction makes the ware bloated and the glaze has a mottled appearance. Bloating, usually associated with overfiring, is an expansion of the body produced by pressure build up of gases trapped in a partially fused mass.

From about 1350°C to the end of the soak period (ie maintained temperature for a considerable time), and the beginning of cooling, the atmosphere is made neutral or slightly reducing (Waye, 1967). During cooling, continued Waye (Ibid), care is taken over the transformation range of the feldspathic glass (800 - 900°C), and also over the beta-alpha inversion at 573°C if much free quartz remains. Mortel (1978) found that "The firing schedule, has no influence on the properties of the densified body. Only the soak period is of influence, but this fact corresponds to the batch composition: time required to dissolve the large quartz grains from a given grade of quartz flour is longer."

In an earlier study, Williams et al (1963) discovered a correlation between the bulk density and its thermal expansion coefficient, and between the density and the mechanical strength of hard porcelain. By a limited factorial experiment, they found that variable firing conditions encountered in kiln are almost entirely responsible for

variations in these properties.

From the study it was first established that thermal expansion was related to bulk density (i.e the ratio of mass to bulk volume of a material being the sum of the solid, closed-pore, and open-pore volumes. Worrall, 1982), and it follows that the far simpler measurement of density would serve to control production firing of articles. Furthermore, mechanical strength of the porcelain was also correlated with bulk density, hence, measurements of density could be a convenient check on quality of wares being fired, particularly high tension insulator bodies.

The method of Williams et al (1963) can be used to adjust the placement of ware in kiln when close quality control is required. It is however, to be used as a supplement and not as a substitute for conventional routine testing methods.

A correctly fired hard porcelain, indicated Williams et al (1963) is non-porous in the sense that it "is vacuum-tight and does not de-gas." It is porous, however, in the sense that it has a number of "closed unconnected pores, indicated by the difference between true and apparent densities."

A well-fired porcelain ware consists of tiny mullite needless, glass, and undissolved quartz. Little, if any, cristobalite is present. The composition at temperature of maximum bulk density, according to Waye et al (1963) is approximately 48% crystalline phase (18% quartz, 30% mullite), and 52% amorphous and glassy phases.

Apart from visual checks for obvious defects, and close dimensional tolerance tests, Waye (1967) identified three main groups of test applied to high tension insulators as:

1. Design or type tests - conducted to assess the efficacy of a design for a particular purpose. Such tests include: an impulse or flashover test to check the ability of the insulator to withstand "extremely short over-voltage surges."; dry flashover and one-minute tests: The former involves determination of the actual voltage at which flash over (i.e. electric discharge over the surface) occurs, while the latter checks the ability to withstand a specified (lower) voltage; wet flashover and one minute rain tests, similar to dry flashover and one minute tests but, conducted under simulated rain.
2. Sample tests - samples are taken on a percentage basis, such as 0.5% from each batch to ensure that the characteristics of the bulk supply conform to specification. The test includes temperature-cycle test, an electro-magnetical, mechanical tests on both porcelain and the metal parts, puncture test, porosity test, and galvanizing test on metal fittings.
3. Routine tests - includes electrical flashover test and mechanical strength tests.

The metallic cap and pin are usually attached to the insulator by cementing with portland cement or high alumina cement such as "Cement Fondu" which has a higher strength and more rapid setting. As discussed by Popper (1980), the top surface to which the metal is attached is "gritted", that is coarse grit of a material with thermal expansion coefficient similar to the glaze is mixed with the glaze and applied to the insulator before firing. During the firing, the grit is incorporated into the glaze and thus provides a rough surface for keeping of the cement.

CHAPTER THREE

RESEARCH PROCEDURE

3.1. Introduction

This Chapter contains the documented step-wise progression of the study: from the location, procurement, beneficiation and characterization of the selected raw materials, to the formulation of different bodies through various tests.

Also explained in this chapter are the methods used for property evaluation, production of experimental samples, and the firing schedule adopted for the study.

3.2. Raw Material Sources

The raw materials used for the study were Bomo clay and Zaria feldspar from Kaduna State, Kankara kaolin and Baure quartz sand from Katsina State, Rakwamni kaolin from Sokoto State, Okene quartz, feldspar, and Isanlu talc from Kwara State, "Petramin" talc from Petramin Nigeria Limited. (Table 3.1).

Selection, location and procurement of the samples were guided by information got from available literature, practising ceramists, professionals in related fields and the locals in the areas.

3.2.1. Sampling

To have samples which were fairly representative of the whole, the "Quartering" and "Shovel" methods (Cardew, 1969) sufficed in drawing samples from the clays while

TABLE 3.1.

Selected Raw Materials

MATERIAL	RAW STATE	TEXTURE	RAW COLOUR
Kankara Kaolin	Granular modules Fair Plasticity	Coarse	Cream White
Rakwamni Kaolin	Granular modules Fair Plasticity	Smooth Feel	Greyish White
Bomo Clay	Lumpy Good Plasticity	Smooth Feel	Grey
Okene Quartz	Large Rocks		White with very few specks of brown
Baure Sand	Granular Modules	Abrasive	Tan
Okene Feldspar	Large Rocks with Angular, Hexagonal tendencies		Pink
Zaria Feldspar	Same As Above		Dark Pink
Isanlu Talc	Lumpy	Greasy Feel	Greenish White
"Petramin" Talc	Powder	Slight Greasy Feel	Greyish White

the rocks were randomly picked from the deposits.

3.3. Beneficiation

While "Petramin" talc was used as supplied, the other materials were beneficiated according to the scheme presented below:

CLAYS	QUARTZ (SAND)	TALC
- Soaked: 24 hrs -	- Washed -	- Calcined: 900°C -
- Blunged: 3 hrs -	- Milled: 24 hrs -	- Jaw Crushed -
- 80 Mesh Sieved -	- 80 Mesh Sieved -	- Milled: 4 hrs -
- Levigated -	- Dried -	- 80 Mesh Sieved -
- Dried -	- Stored -	- Dried -
- Stored -		- Stored -

FELDSPAR	QUARTZ (ROCK)
- Rinsed -	- Rinsed -
- Calcined: 950°C -	- Calcined: 950°C -
- Jaw Crushed -	- Jaw Crushed -
- Milled: 18 hrs -	- Milled: 24 hrs -
- 80 Mesh Sieved -	- 80 Mesh Sieved -
- Dried -	- Dried -
- Stored -	- Stored -

The materials were ground by wet ball milling and stored in plastic receptacles so as to avoid contamination (due to corrosion) which is symptomatic of some metal receptacles.

3.4. Laboratory Tests on The Materials

The following laboratory tests were carried out on the beneficiated materials:

Particle Size Analysis:

Irrespective of the material, the microstructures of sintered products depend strongly on the particle size distribution, shape of the grains and surface area of the initial powder. (Waye, 1967, Ford, 1967, Paulus, 1978, Cutler, 1978). A combination of sieve - and hydrometer analyses was used to determine the grain size distribution of the beneficiated materials.

Sieve analysis: 100gm of specimen was washed through a 63μm lawn. Residue retained on the lawn and that passing through were separately dried at 110°C for 24 hours. After drying to 0% moisture content, the sample that passed through was weighed while the residue retained on the lawn was collected and dry-sieved through a set of sieves arranged in the order: 600μm, 425μm, 300μm, 212μm, 150μm, 63μm, and the bottom pan. After thoroughly shaking, residue on each sieve was weighed and calculated as a percentage of the whole:

$$\frac{\text{Mass Retained on Each Sieve}}{\text{Total Mass of the Soil}} \times 100$$

Hydrometer analysis: A known quantity (less than 50gm) of each sample passing 63μm lawn from the sieve analysis was weighed out .20cc of dispersing agent - prepared by mixing 33gm of sodium hetametaphosphate and 7gm of sodium carbonate in 1000ml distilled water - was added to the weighed sample. Prior to pouring into a 1000ml measuring cylinder placed on a flat and solid base, the mixture was mixed in an electric stirrer for about 3 minutes. After pouring into the measuring cylinder, the hydrometer bulb was dropped into it. Readings was then taken for specified time intervals - 1min, 2min, 4min, 8min, 15min., 30min., 1hr., 2hrs., 4hrs., 6 hrs., 24hrs., - taking care to ensure that the eye was correctly placed so as to avoid parallax errors.

In making calculations for the cumulative distribution curve, the following were taken into consideration:

R'H	Hydrometer reading: top of meniscus
R_H	Hydrometer reading corrected for meniscus ($R'H+C_M$)
H_R	Effective depth of hydrometer corresponding to R_H.
D	Equivalent Particle diameter in mm (from nomograph)
$W_S\%$	Percent by weight of particles smaller than D in whole sample.
C_M	Meniscus correction: 0.4 (constant for hydrometer)

C_D Density correction for dispersing agent: 1.0 (constant)

M_T Temperature correction for hydrometer 1.5 (from chart)

G Specific gravity of soil particles

W_T Weight of total specimen (100gm).

W_S Weight of dry soil in suspension (passing 63um sieve).

Water of Plasticity:

This test was conducted to determine the amount of water required by each clay to develop its optimum plastic qualities.

Procedure:- Sample was pulverized to pass through a 30 mesh sieve. 100gm of the pulverized sample was weighed onto a glass slab. Water was added to the clay drop by drop from a 100ml graduate filled with water while mixing was simultaneously carried on with a non absorbent spatula until the clay was about the right consistency for modelling. Amount of water required to get it into this consistency was noted and the percentage water of plasticity calculated:

$$\% \text{ Water of Plasticity: } \frac{\text{Wt of Water Used}}{\text{Wt of Dry Clay}} \times 100$$

Dimensional Change (Linear) Determination:

Amount of shrinkage of a clay is a very important characteristic moreso in technical ceramics where a good dimensional tolerance is emphasized. The dimensional change test was conducted to assess the plastic and firing shrinkages of the selected clays.

Procedure: Slabs of about 2.5cm thickness were made out of thoroughly wedged and kneaded samples. Whilst in the plastic state a 6cm line was made on each slab. After careful drying at 110°C for 24 hours, the original 6cm line was measured and percentage linear drying shrinkage calculated:

$$\% \text{ Drying Shrinkage: } \frac{\text{Wet Length} - \text{Dry Length}}{\text{Wet Length}} \times 100$$

The same slabs were then fired to 1000°C and 1300°C. The line was measured after each firing and percentage fired shrinkage calculated for each temperature using the formula:

$$\% \text{ Firing Shrinkage: } \frac{\text{Change in Length}}{\text{Original Length}} \times 100$$

Percentage total linear shrinkage was calculated thus:

$$\% \text{ Total Shrinkage: } \frac{\text{Total Change in Length}}{\text{Wet Length}} \times 100$$

Water Absorption Determination

The water absorption test which determines the ability of the clay specimens to absorb water by capillarity, indicates the proximity of a specimen to its vitrification point as a result of heat treatment.

Procedure: After firing to 1000°C and 1300°C, the slabs used in the dimensional change tests above were weighed whilst warm from each firing then boiled in water for 2 hours and left to cool with the water. The water on the surface of the slabs was wiped off with a damp piece of cloth (which will not "attract" water from the specimen)

before taking the soaked weight of the specimens. Increase in weight (if any) was then calculated as a percentage of the original dry weight:

$$\% \text{ Water Absorption: } \frac{\text{Soaked Wt} - \text{Dry Wt}}{\text{Dry Wt}} \times 100$$

Temperature Tolerance (fusion) Test:

This test was conducted primarily to determine the effect of heat treatment on the different materials selected for the study with further anticipation that the presence of certain impurities would be highlighted after heat application.

Procedure: Specimens were put in refractory plates and fired to 1300°C after which texture and colour of the specimens were observed.

3.5. Body Formulation

From the understanding of available literature, mechanical strength is improved in compositions nearer the high-clay corner of the clay-feldspar-quartz triaxial blend (Singer and Singer, 1963, Waye et al 1963, Waye, 1967, Davidge, 1979); The expansion coefficient rises with increasing feldspar and high free quartz content while it (coefficient of expansion) is in inverse proportion to the clay content (Singer and Singer, Ibid, Waye, Ibid, Ford, 1967, Mortel, 1978). The implication of this viewpoint is that thermal shock resistance increases with increasing clay content; Abrasion resistance increases with increasing quartz content and declines in high feldspar low-quartz

bodies as indicated by Singer and Singer (Ibid) who further observed that power factor (previously defined as the measure of energy lost when an insulator transforms part of the electrical energy into heat) rises with the alkali content.

Discussing the Variation in electrical resistivity with temperature, Waye (1967) noted that in porcelain, as in most other ceramic insulators, conduction is predominantly caused by the movement of ions (particularly of K and Na in porcelain) while thermal conductivity is mainly by lattice vibration since porcelain insulators contain few free electrons. Again this depends on the intrinsic properties of the material. To be considered are the atomic and microstructure, impurity level, residual porosity and the finishing temperature.

Based primarily on the above considerations, a finishing temperature of about 1300°C, and factors related to fabrication, eighteen likely compositions were selected from the high-clay corner of the clay-feldspar-quartz triaxial blend (Fig. 3.1.). The arrows in Figure 3.1 indicate the direction of progression of each material.

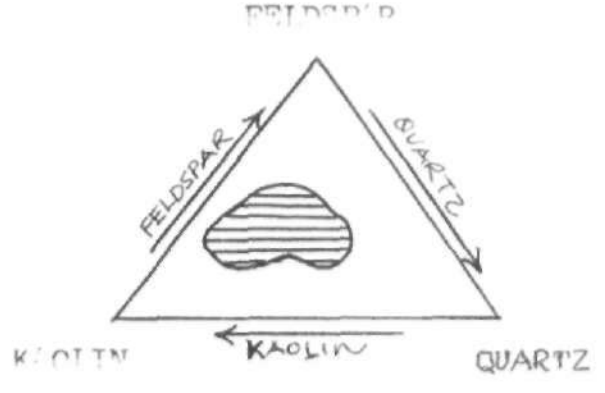


Fig. 3.1: Triaxial Blend Indicating Selected Area

Primary Test:

The percentage ranges of the compositions selected from Fig. 3.1 were 30 - 55; 30 - 50; 15 - 35, for Kaolin; Feldspar; Quartz respectively. Using Kankara Kaolin, Okene Quartz, and Okene Feldspar as starting materials, the batch components of each body were weighed out according to the percentage proportions. The powders were thoroughly dry-mixed, moistened with water and pressed into compacts of approximately 3cm in diameter by 1.5cm thick, using a laboratory handpress.

The compacts were dried in an oven before firing, first to 1000°C then 1300°C. The water absorption method was used to determine the porosity of the fired compacts while colour (internal, and external) bloating tendencies (if any) and texture were noted. From the eighteen different compositions that constituted the primary tests, eleven were selected for further investigations.

3.5.1. Additional Tests

Additional tests conducted on the selected bodies were designed to improve their castability, dry strength, densification (Bomo Clay Addition) enhance mechanical strength (Talc Addition) and also to investigate the possibility of substituting materials of "similar" chemical, composition in the interest of uniform quality in case slight changes occur in the composition of the tested materials. (Material Substitution).

Bomo Clay Addition Test:

Bomo Clay was the only plastic clay procured for the study so it was used for this test.

Procedure:

Two categories were created with each of the selected bodies represented in both categories. The first category (B.T.A) had 12% less Kaolin and 12% addition of Bomo Clay; bodies in the second category (B.T.B) with 19% less kaolin and 19% addition of Bomo Clay. The solid ingredients, in each case, were weighed out and wet ball milled for 6 hours in a jar mill. In all cases ratio of grinding balls to batch to water was held constant at 1:1:1. After grinding the resultant slip was passed through a 100 mesh lawn before thoroughly drying at 110°C.

Preparation of the casting slip: After drying, each body was pulverized to pass through a 40 mesh sieve and weighed out in 50gm packets as suggested by Rhodes (1973). Water, approximately half the weight of each batch, was measured into a bowl and the powder added to the water 50gm at a time with stirring simultaneously carried on to avoid the formation of lumps. Whenever the mixture was getting too pasty, some deflocculant was added.

The deflocculant was a mixture of sodium carbonate and 75°Tw sodium silicate dissolved in very hot water. Sodium carbonate, an "alkali cation type", was used to increase thixotropy while sodium silicate, a "polyanion" deflocculant, was used to increase fluidity as advised by Hamer (1975) and Worrall (1982). A lower limit of 1.5gm

deflocculant was set for every 250gm solid ingredients. The deflocculated slips were covered and left undisturbed for 24 hours at room temperature. Rheology of the slips was observed after the 24 hour rest. Prior to casting experimental bars in plaster test moulds, each slip was stirred (with care taken not to stir in air), and passed through a 100 mesh lawn. A 10cm line was made on each bar whilst in the plastic state. After drying and firing, percentage linear drying and fired shrinkage, porosity (water absorption and apparent) and bulk density, values were calculated for the different bodies.

The method of preparation of casting slip described above was retained for subsequent tests.

Materials Substitution Test (M.S.T):

This test involved a body composition selected from the Bomo Clay Addition test. However, the test was restricted to the Kaolin and Quartz sources: Rakwamni kaolin and Baure quartz sand were partly substituted for Kankara kaolin and Okene quartz respectively.

Procedure:

Holding all other variables constant, the kaolin corner was sourced from an equal combination of Kankara and Rakwamni kaolins. Similarly, the quartz corner was sourced from an equal combination of Okene quartz and Baure quartz sand.

The solid ingredients were weighed out, wet ball milled,

dried, deflocculated and left to stand for 24 hours at room temperature. After this time lapse, the slip was stirred, 100 mesh sieved and test bars cast. These were carefully dried, then bisqued and glost fired.

In addition to calculating the drying, firing and total linear shrinkages, percentage water absorption, apparent porosity and bulk density values of the cast bars, colour (internal and external), and bloating tendencies (if any) were noted.

Talc Addition Test:

The Talc Addition Test (T.A.T) was aimed at exploiting the low thermal expansion flux provided by talc. It was further anticipated that its presence in a body would enhance mechanical strength, give good transparency and reduce deformation as discussed by Singer and Singer (1963), Hamer (1975) and Alvarez-Estrada (1979).

Procedure:

2% talc (calculated against the percentage of the body) was added to the M.S.T. composition. Due to the fact that talc does not wet easily, the solid ingredients were thoroughly dry-mixed before milling for 6 hours. As with the other tests, the resultant slip was passed through a 100 mesh lawn, dried, deflocculated and left to rest for 24 hours. Rheology of the slip was observed before sieving and casting test bars which were carefully dried and fired. The following were also assessed: drying, firing and total linear shrinkages, water absorption, apparent porosity,

bulk density, internal and external colour, bloating tendencies if any.

3.5.2. Production of Experimental Samples

Experimental samples ("D" line insulator) were cast in a four-piece plaster mould. The model used in making the plaster mould was plastic-turned out of an air-dried lump of throwing clay body that had been wedged and kneaded. The turned clay model was made slightly bigger so as to accomodate the shrinkages of the body selected for the production of the experimental samples.

The plaster of paris was prepared by sieving through a 30 mesh sieve before using. It was then sprinkled in water and optimal saturation, at each point, was indicated by the appearance of dry powder patches on the water surface. The mixture was vigorously stirred and ready for use.

Segmentation of the mould was determined by the nature of protrusions and recesses in the insulator model. Then, according to predetermined dimensions, the clay model was surrounded with a vinyl sheet and the powder-water mixture poured in to the desired level. The finished mould segments were thoroughly dried ready for use.

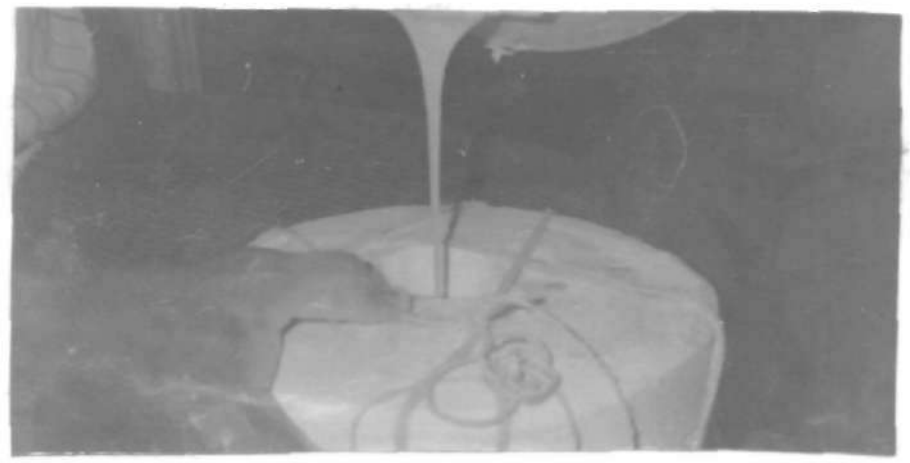


PLATE 1: SLIP POURING INTO MOULD

The dried segments were coupled and secured with a thick rubber band. Prepared casting slip was then poured steadily into the mould cavity through the orifice provided for the purpose. Each time the slip level dropped more slip was added until the desired thickness - thus firmness - was achieved. Excess slip was poured out and the cast removed from the mould, carefully dried fettled and fired.

3.6. FIRING

To achieve the desired heatwork (an imprecise term defined by Adegbite, 1975 as the combined effect of time and temperature on a ceramic process), specimens were bisque fired to 950°C in 8 hours in an electric kiln before glost firing to about 1290°C in 11 hours in a gas-fuelled kiln.

In the firing cycle, an oxidising atmosphere was used to burn off organic matter upto about 1000°C before commencement of vitrification (Singer and Singer, 1963, Williams et al, 1963, Waye, 1967). From about 1000°C,

the atmosphere was changed to reducing with a view to, as discussed by Singer and Singer (Ibid), Williams et al (Ibid), Waye (Ibid), reducing any iron present to the colourless ferrous form. When Orton cone 10 (large) collapsed, soaking was done for about 30 minutes (Mortel, 1978). After each firing, the kiln was gradually cooled before removing specimens.

3.7. Porosity And Bulk Density Determinations

Pores are usually discussed in terms of "open" and "sealed". According to Griffiths and Radford (1965) "Open pores are voids which are accessible (at the surface of the article) to penetration by fluid" caused by the imperfect packing of the individual particles of the material or "the escape of gases during drying and firing processes." Sealed pores, on the other hand, are "formed on firing when bubbles of gas are frozen into the glassy matrix, or when open pores are sealed by molten material."

To determine porosity specimen was weighed while still warm from firing, boiled in water for 2 hours and allowed to cool with the same water. The water on the specimen was then wiped off with a slightly damp piece of cloth before taking its soaked weight. The immersed weight of the specimen was also taken. The determination of the immersed (suspended) weight is based on Archimedes Principle which states that "When a body is immersed in a fluid, its apparent loss in weight

(upthrust) is equal to the weight of the fluid displaced."

Calculations were done using the following formulae:

$$\% \text{ Water absorption: } \frac{\text{Soaked Wt - Dry Wt}}{\text{Dry Wt}} \times 100$$

$$\% \text{ Apparent porosity: } \frac{\text{Soaked Wt - Dry Wt}}{\text{Soaked Wt - Imm.Wt}} \times 100$$

Bulk density, being a relationship between mass (weight) and volume was calculated thus:

$$\frac{\text{Mass}}{\text{Soaked Wt - Immersed Wt}} : \frac{\text{Mass}}{\text{Total Volume}}$$

CHAPTER FOUR

ANALYSIS OF RESULTS, DISCUSSION AND FINDINGS

4.1. Introduction:

Being "An enquiry into selected local raw materials for the formulation of electrical porcelain body in Nigeria", several tests were conducted on the procured materials and the formulated bodies. The results and analyses of the various tests are presented in this Chapter.

Several test pieces were used in the determination of any characteristic hence Mean Value (for single set disparities) and Dispersion/Scatter (for small groups) were used to align results.

4.2. Results of Laboratory Tests

Results of the property tests carried out on the clays are presented in Table 4.1:

TABLE 4.1.

Properties of Selected Clays.

Test Conducted	KANKARA	RAKWAMNI	BOMO
% Water of Plasticity	55	39	36
% Drying (Linear) Shrinkage	8.3	5.0	9.4
% Firing (Linear) Shrinkage: 1000°C	3.6	0.6	0.0
% Firing (Linear) Shrinkage: 1300°C	5.6	1.7	4.9
% Total (Linear) Shrinkage	16.6	6.6	13.3
% Water Absorption: 1000°C	25.0	18.4	14.0
% Water Absorption: 1300°C	14.4	17.3	0.5
Colour at 1300°C	White	Greyish White	Dark Brown

Water of Plasticity Result:

The water of plasticity (variously refered to as "Lubricant Water", "Seperate Water"), according to Hamer (1975)" provides the lubrication for the clay particles and enables them to slide past one another." This then presupposes that there is an optimum amount of water required by each clay. Thus, from Table 4.1, more water is required by Kankara kaolin (55%) than either Rakwamni kaolin (39%) or Bomo clay (36%) to render the same amount (100gm) of clay workable. What this means in essence is that the ratio of water to clay required to

develop optimum consistency in, for example, Kankara kaolin is 55 parts of water to 100gm of clay.

Dimensional Change Results:

Bomo clay has the highest wet to dry shrinkage of 9.4% (Table 4.1), but between 110°C - after drying at 110°C - and 1000°C, the highest linear shrinkage of 3.6% was recorded for Kankara clay. Fired to around 1300°C, Kankara clay sintered heavily to give a further shrinkage of 5.6%, and a total linear shrinkage of 16.6% which is clearly the highest.

It should be noted here that percentage firing shrinkage is based on the dry length while the wet to dry is based on the wet length so that for simple arithmetic reason, the sum of the percentage wet to dry shrinkage and percentage dry to fired shrinkage will not be equal to the percentage total shrinkage. This then calls into question the total shrinkage figures given by Gukas (1985).

Water Absorption Result:

The water absorption has been defined by Griffiths and Radford (1965) as "The ratio of open pore volume to the weight of the test piece. "The water absorption was adopted for the study to measure the maturity of the selected clay samples at different temperatures.

Looking at Table 4.1. the 0.5% calculated for Bomo clay indicates that it has almost reached its point of vitrification (glassification) at around 1300°C so that

with an absorption value of 17.3% at 1300°C, Rakwamni clay is the most refractory.

Temperature Tolerance Result:

The behaviour of the different clays has been discussed above and their fired colours presented in Table 4.1.

As was expected, no fusion was observed in the Okene quartz and Baure quartz sand which both fired white (though few brownish specks were discernible in the Baure quartz sand). Similarly, there was no fusion in both Isanlu talc and petramin talc. While the former fired tan, the latter simply burnt off leaving a small remnant; a trait which was considered unacceptable.

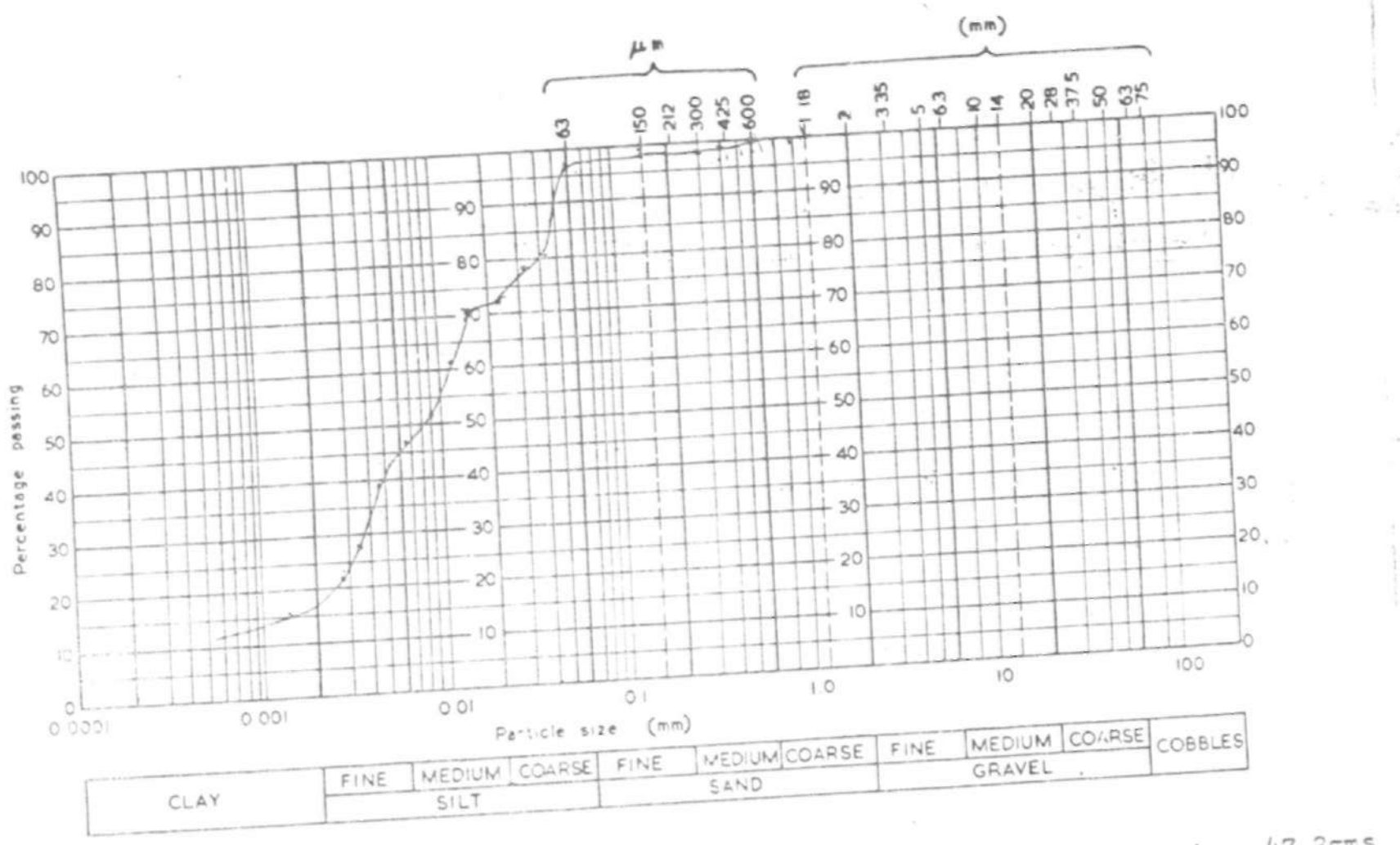
Okene feldspar, fired to the same 1300°C as the other materials, produced a thick white melt while the colour of the fused Zaria feldspar was in the Olive family: another unacceptable trait.

Particle Size Distribution:

The particle size distribution of the sampled materials (with the exception of Bomo and Rakwamni kaolin) as aforementioned, involved the sieve and hydrometer methods. The results are however presented separately. Basically, the hydrometer test is used to measure the sedimentation rate of particles in a quiescent liquid as a function of time, relating it to the sphere of the same density "that would exhibit the identical free fall velocity" of the particle. According to Orr (1978), this measure "readily

TABLE 4.2: SIEVE ANALYSES OF SELECTED MATERIALS

MATERIAL	MASS OF SAMPLE	MASS PASSING 63μM SIEVE	PERCENTAGE RETAINED (μm test sieve)					
			600	425	300	212	150	63
KANKARA KAOLIN	100	94.70	0.10	0.10	0.10	0.10	1.30	3.50
OKENE QUARTZ	100	99.80	-	-	-	-	0.10	0.10
BAURE QUARTZ	100	98.50	-	-	0.10	0.10	0.10	1.19
ISANLU TALC	100	99.80	-	-	-	-	0.10	0.10
PETRAMIN TALC	100	99.70	-	-	-	0.10	0.10	0.10
OKENE FELDSPAR	100	93.40	0.30	0.10	0.10	0.50	1.30	4.20
ZARIA FELDSPAR	100	99.70	-	-	-	0.10	0.10	0.10



Dry Soil in Suspension: 47.2gms

Fig. 2: Hydrometer Analysis of Kankara Kaolin.

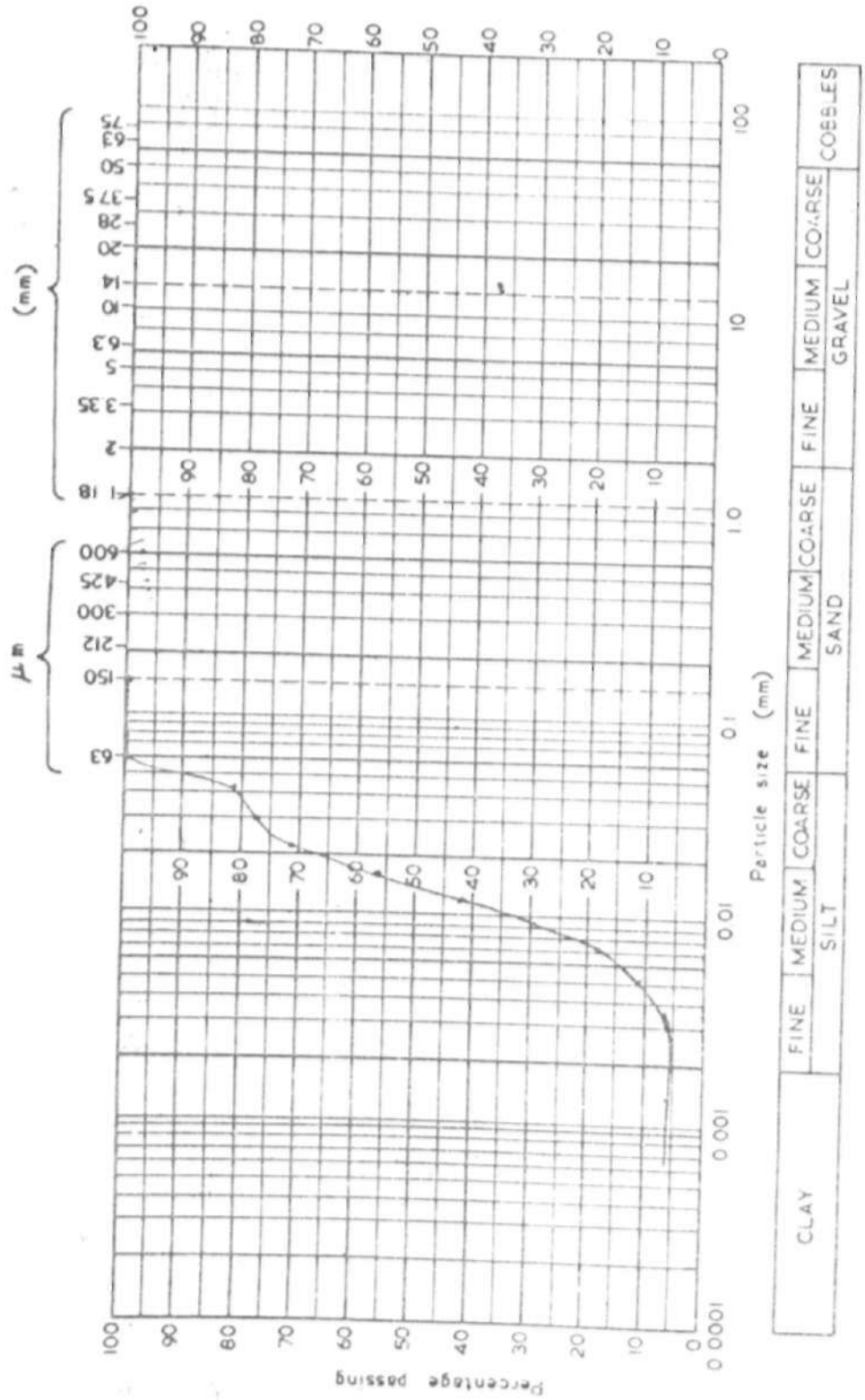


Fig. 3: HydrometerAnalysis of Okene Quartz. Dry Soil in Suspension: 47.2gms

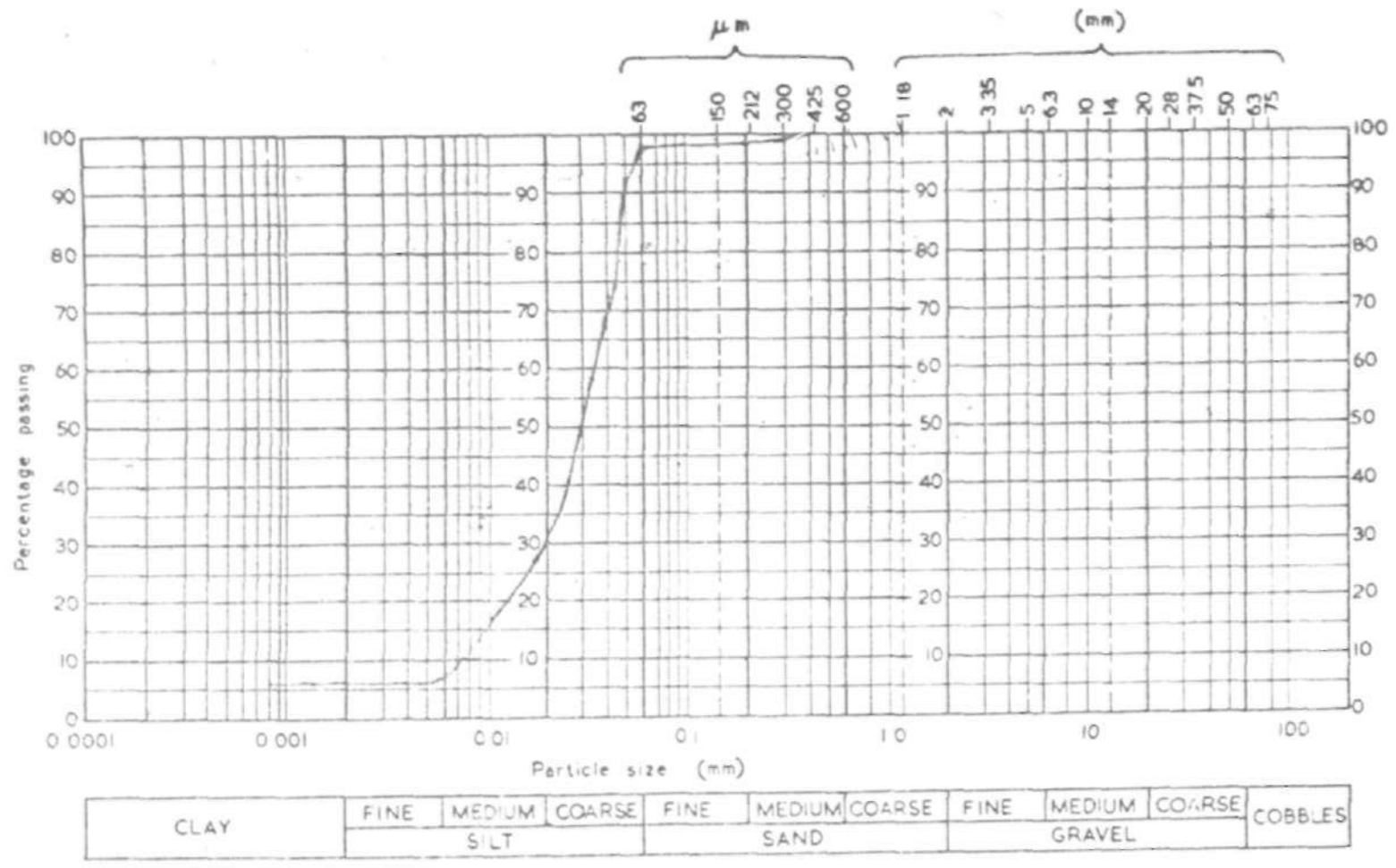


Fig. 4: Hydrometer Analysis of Baure Quartz Sand. Dry Soil in Suspension: 44.6gms

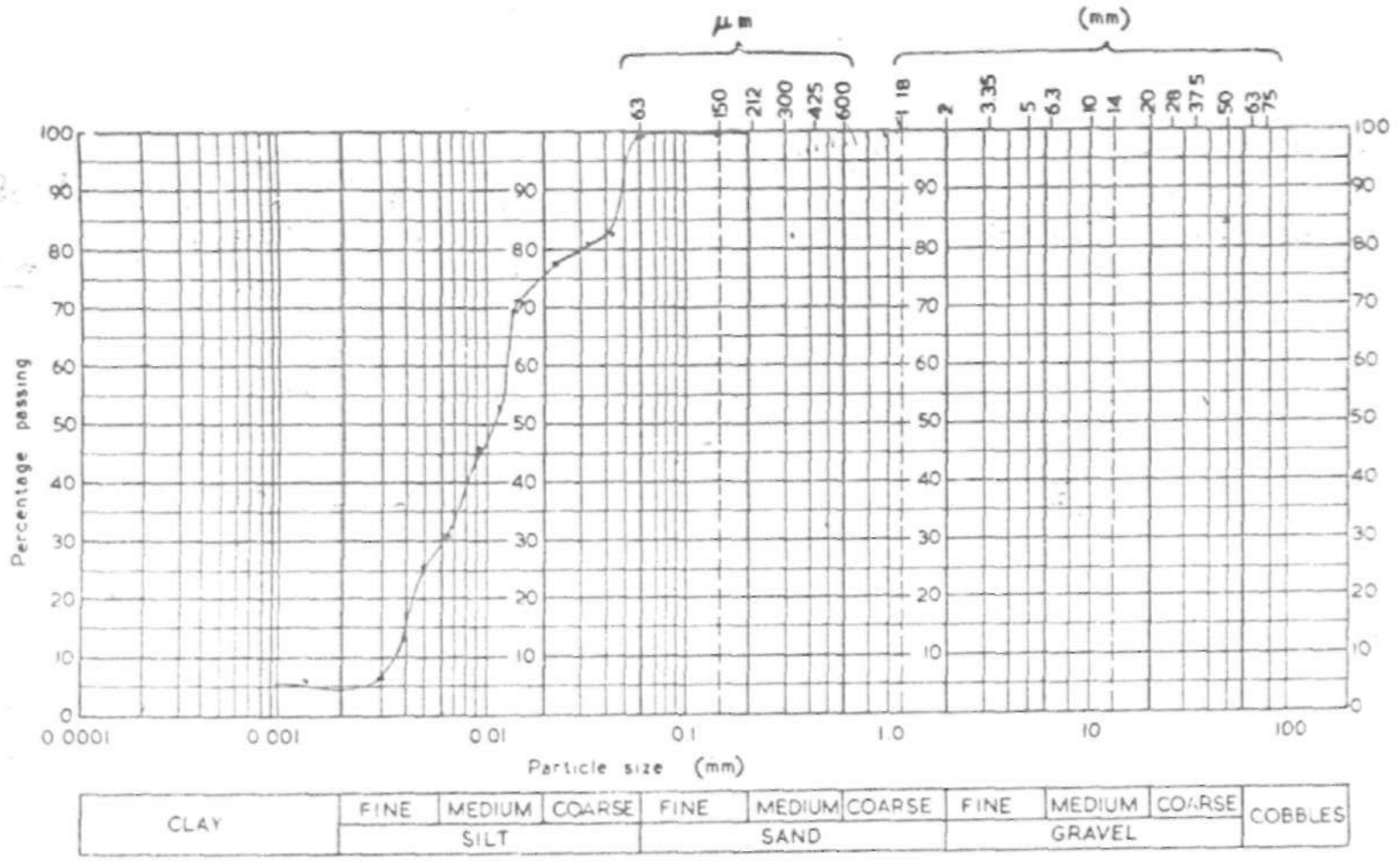


Fig. 5: Hydrometer Analysis of Isanlu Talc. Dry Soil in Suspension: 44.6gms

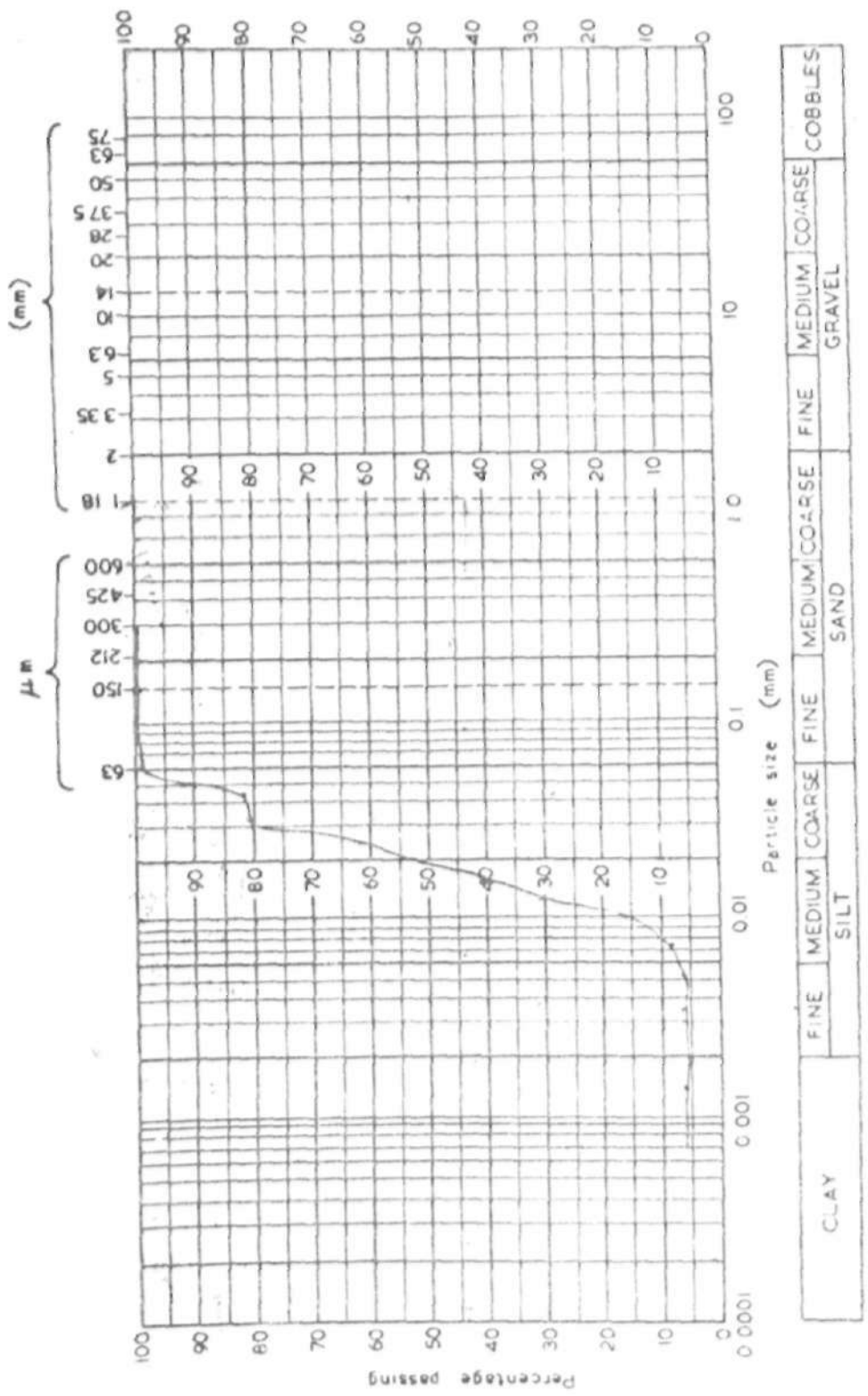


Fig. 6: Hydrometer Analysis of Petramin Talc. Dry Soil in Suspension: 43.3gms

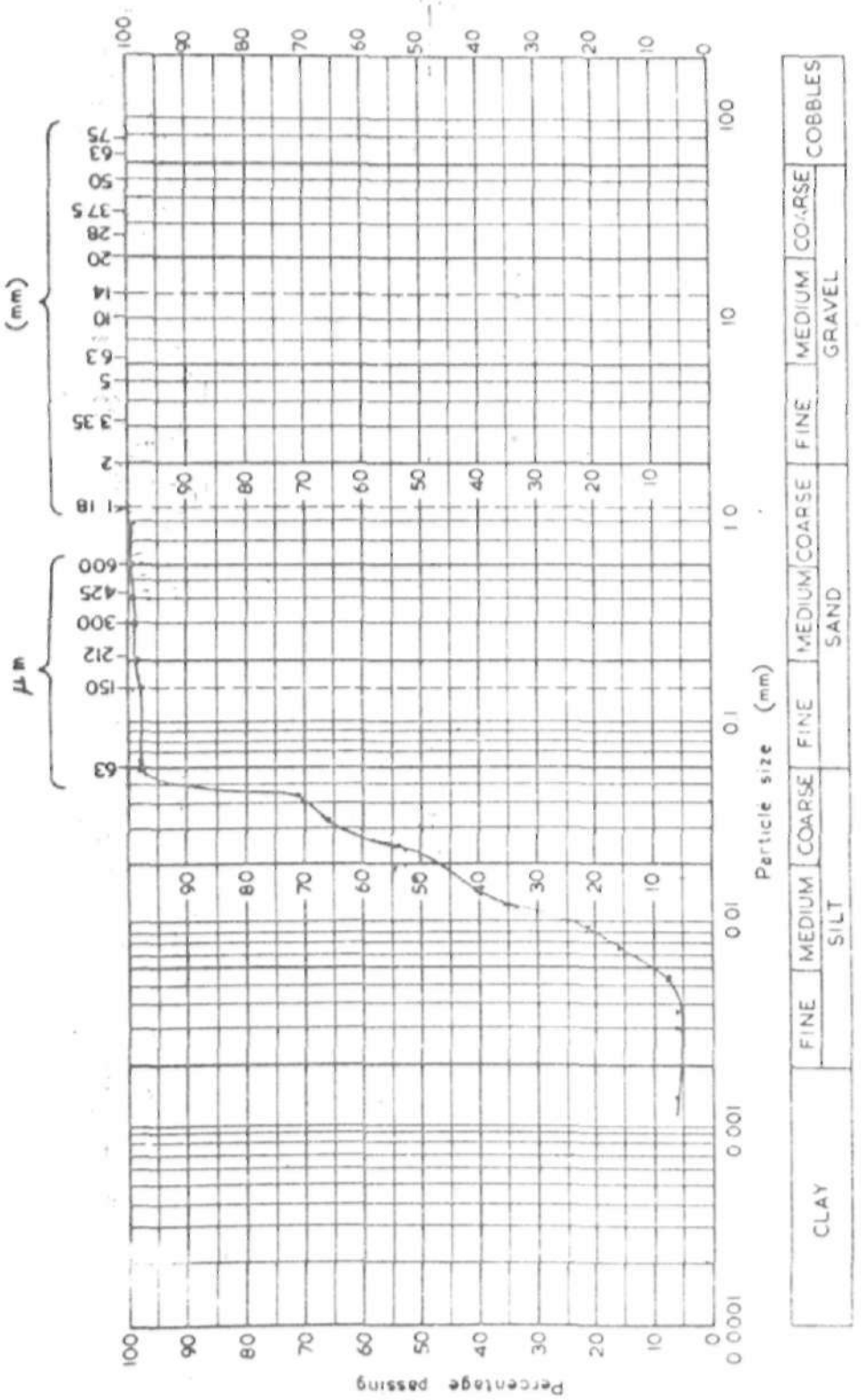


Fig. 7: Hydrometer Analysis of Okene Feldspar.

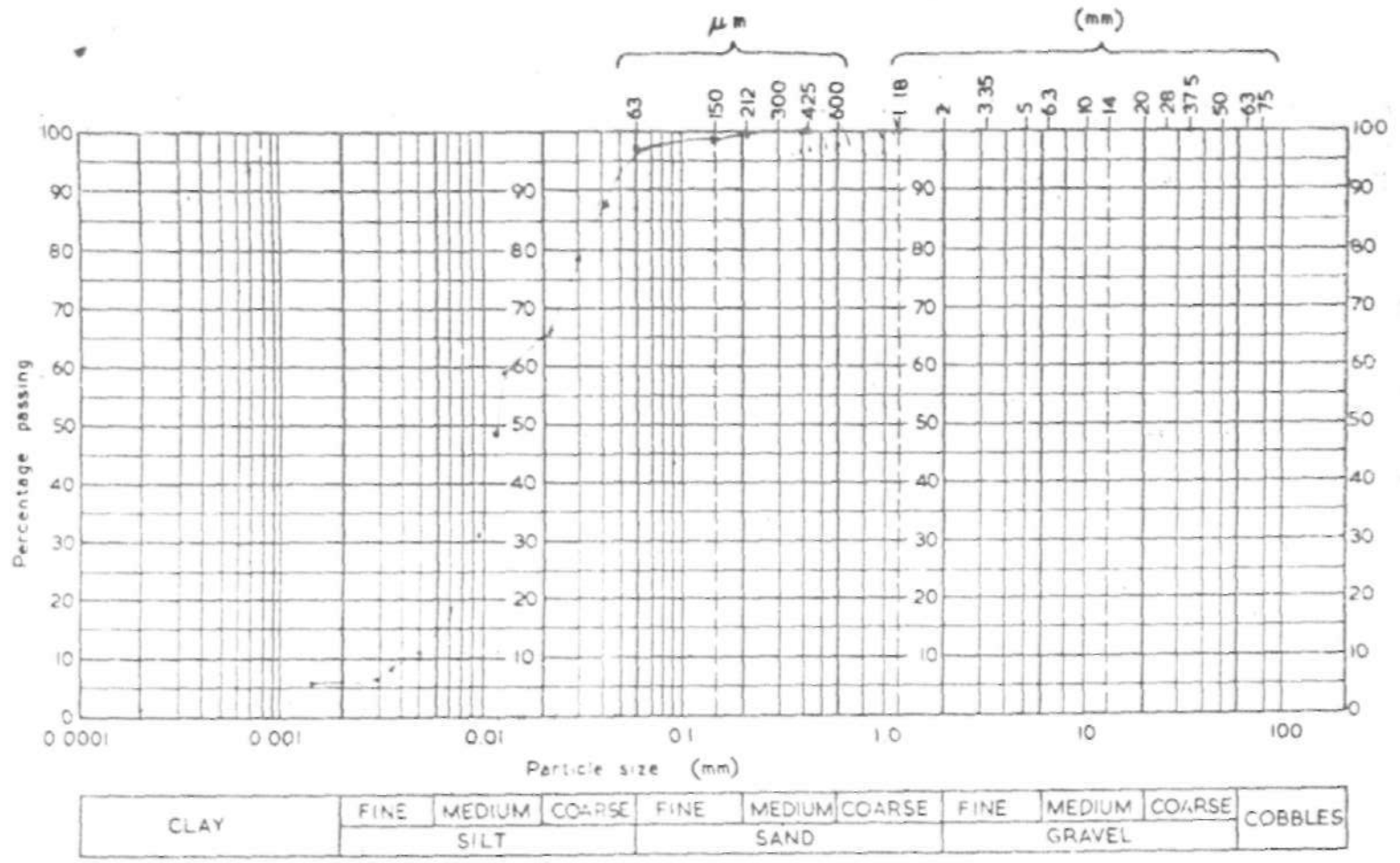


Fig. 8: Hydrometer Analysis of Zaria Feldspar. Dry Soil in Suspension: 45.3gms

correlates with many handling and processing parameters."

4.3. Body Formulation Results

Results of the property tests conducted on the fired pressed compacts can be seen in Table 4.3. Bodies selected from this table were subjected to additional tests namely Bomo clay addition (B.T.A and B.T.B), Talc addition (T.A.T.), and materials substitution (M.S.T.). The outcome of these are treated individually.

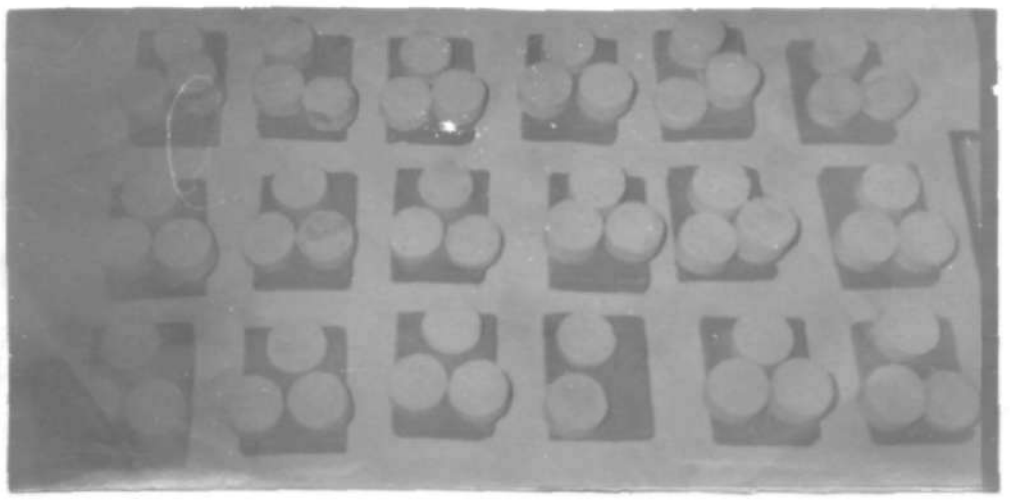


PLATE II: FIRED PRESSED COMPACTS

In the case of the subdivisions (B.T.A and B.T.B), it will be recalled that for every given body the only difference between B.T.A and B.T.B is the clay content: B.T.A 12% Bomo clay; B.T.B 19% Bomo clay.

TABLE 4.3: Result of Property Tests Conducted on Fired Pressed Compacts

BODY	MATERIAL KAOLIN	QUARTZ	FELDS.	% WATER ABSORPTION	BEHAVIOUR AT 1000%	COLOUR INTERNAL	EXTERNAL	REMARK
A	30	20	50	0.20	Friable	White	white	Translucent, selected.
B	40	20	40	0.00	Friable	White	White	Selected
C	30	30	40	0.00	Friable	White	White	Selected
D	50	20	30	1.97	Friable	White	White	Selected
E	40	30	30	1.80	Friable	White	White	Selected
F	60	10	30	2.10	Friable	White	White	-
G	40	40	20	5.77	"	"	"	-
H	50	30	20	7.36	"	"	"	-
I	60	20	20	4.98	"	"	"	-
J	35	15	50	0.00	"	"	"	Translucent, Selected
K	45	15	40	1.14	"	"	"	Selected
L	35	25	40	0.88	"	"	"	Selected
M	55	15	30	1.89	"	"	"	Selected
N	45	25	30	1.78	"	"	"	Selected
O	35	35	30	0.96	"	"	"	Selected
P	55	25	20	4.00	"	"	"	-
Q	45	35	20	4.80	"	"	"	-
R	35	45	20	8.20	"	"	"	-

BODY 1

	B.T.A (%)	B.T.B (%)
Kankara Kaolin	18	11
Bomo Clay	12	19
Okene Quartz	20	20
Okene Feldspar	50	50
Water(ml)	246	246
Solid Ingredients (gm)	500	500

Deflocculated with 3gm sodium silicate and 1gm sodium carbonate and left to stand for 24 hours, B.T.A formed a sludge at the bottom of the container with water gathering at the top hence other property tests were not determined. However, this settling phenomenon was not observed in B.T.B which was deflocculated with 1.5gm sodium silicate and 1.5gm sodium carbonate. After the 24 hour time lapse, rheology of the slip remained good coupled with an early mould release. The fired body was translucent, reminiscent of the original composition (Table 4.3).

TABLE 4.4: PROPERTIES OF BODY 1

Test Conducted	B.T.A	B.T.B
Drying Shrinkage (%)	-	1.63
Firing Shrinkage (%)	-	13.37
Total Shrinkage (%)	-	14.79
Water Absorption (%)	-	0.08
Apparent Porosity (%)	-	0.20
Bulk Density (gm per cc)	-	2.315
Fired Colour: Internal	-	Greyish White
External	-	Tan Tinged

BODY 2

	B.T.A (%)	B.T.B (%)
Kankara Kaolin	28	21
Bomo Clay	12	19
Okene Quartz	20	20
Okene Feldspar	40	40
Water (ml)	246	250
Solid Ingredients (gm)	500	500

The B.T.A. body was deflocculated with 3gm sodium silicate and 1gm sodium carbonate. After 24 hours, setting was observed and when about 2gm sodium hetametaphosphate was introduced, the slip gelled heavily making it impossible to continue further tests on the body.

The modified body (B.T.B) was deflocculated with 2.5gm sodium silicate and 1.5gm sodium carbonate in 250ml water. Rheology of the deflocculated slip remained good after 24 hours. Mould release was early. This body was translucent after firing and its other properties are given in Table 4.5.

TABLE 4.5: Properties of Body 2

Test Conducted	B.T.A.	B.T.B.
Drying Shrinkage (%)	-	2.95
Firing Shrinkage (%)	-	13.20
Total Shrinkage (%)	-	15.78
Water Absorption (%)	-	0.085
Apparent Porosity (%)	-	0.21
Bulk Density (gm per cc.)	-	2.395
Fired Colour: Internal	-	Greyish White
External	-	Tan tinged

BODY 3

	B.T.A (%)	B.T.B (%)
Kankara Kaolin	18	11
Bomo Clay	12	19
Okene Quartz	30	30
Okene Feldspar	40	40
Water (ml)	250	246
Solid Ingredients (gm)	500	500

B.T.A was deflocculated with 2gm sodium silicate and 1.5gm sodium carbonate. After 24 hours no settling was observed but the thixotropy appeared to be high. This was corrected with the addition of .5gm sodium silicate and about 4ml water. Properties of this body are presented in Table 4.6. In the case of B.T.B. 1.5gm sodium silicate and 1.5gm sodium carbonate was enough for effective deflocculation. Rheology of the slip remained good after the 24 hour time lapse. Mould release of B.T.B, as with B.T.A, was early. The B.T.B body was very translucent after firing and its other properties are presented in

Table 4.6.

TABLE 4.6: Properties of Body 3

Test Conducted	B.T.A	B.T.B
Drying Shrinkage (%)	1.97	1.63
Firing Shrinkage (%)	15.12	14.35
Total Shrinkage (%)	16.79	15.77
Water Absorption (%)	0.09	0.12
Apparent Porosity (%)	0.21	0.29
Bulk Density (gm per cc.)	2.40	2.32
Fired Colour: Internal	Greyish White	Greyish White
External	Greyish White	Greyish White

BODY 4

	B.T.A (%)	B.T.B (%)
Kankara kaolin	28	21
Bomo Clay	12	19
Okene Quartz	30	30
Okene Feldspar	30	30
Water (ml)	250	250
Solid Ingredients (gm)	500	500

A combination of 1.5gm sodium silicate and 1.5gm sodium carbonate was used to deflocculate B.T.A. After resting for 24 hours, the thixotropy of the slip was observed to be high. Fluidity was therefore enhanced by the addition of .5gm sodium silicate and about 4ml water. The mould release was good and the cast experimental bars were subjected to the specified tests. (Table 4.7).

When Bomo Clay was increased and Kankara Kaolin reduced (B.T.B), 2.5gm sodium silicate and 1.5gm sodium carbonate was needed for effective deflocculation. When observed

after 24 hours rheology remained fine. B.T.B., which was translucent after firing, also had an early mould release.

TABLE 4.7: Properties of Body 4

Test Conducted	B.T.A	B.T.B
Drying Shrinkage (%)	2.95	3.94
Firing Shrinkage (%)	13.55	13.30
Total Shrinkage (%)	16.11	16.78
Water Absorption (%)	0.04	0.05
Apparent Porosity (%)	0.10	0.11
Bulk Density (gm per cc.)	2.405	2.405
Fired Colour: Internal	Greyish White	Greyish White
External	Greyish White	Greyish White

BODY 5

	B.T.A. (%)	B.T.B. (%)
Kankara Kaolin	23	16
Bomo Clay	12	19
Okene Quartz	15	15
Okene Feldspar	50	50
Water (ml)	255	255
Solid Ingredients (gm)	500	500

After deflocculating B.T.A with 2gm sodium silicate and 1.5gm sodium carbonate, the slip was left to stand. After 24 hours, rheology of the slip remained good. When Bomo Clay was increased the (B.T.B), body required the same amount of deflocculants as B.T.A: 2gm sodium silicate and 1.5gm sodium carbonate. In both cases mould release was good and when fired translucency was more pronounced in the B.T.B.

TABLE 4.8: Properties of Body 5

Test Conducted	B.T.A	B.T.B
Drying Shrinkage (%)	1.96	2.95
Firing Shrinkage (%)	12.33	13.85
Total Shrimkage (%)	14.15	16.44
Water Absorption (%)	0.065	0.085
Apparent Porosity (%)	0.16	0.21
Bulk Density (gm per cc.)	2.36	2.32
Fired Colour: Internal	Greyish White	Greyish White
External	Tan tinged	Tan tinged

BODY 6

	B.T.A (%)	B.T.B (%)
Kankara Kaolin	33	26
Bomo Clay	12	19
Okene Quartz	15	15
Okene Feldspar	40	40
Water (ml)	250	246
Solid Ingredients (gm)	500	500

The B.T.A was deflocculated with 2.5gm sodium silicate and 1.5gm sodium carbonate. The B.T.B, on the other hand, required 1.5gm sodium silicate and 1.5gm sodium carbonate to make a good casting slip. Rheology of both slips remained good after the 24 hour time lapse. The mould release of both slips was also good and early. The fired B.T.B was however more translucent than B.T.A. Their other properties are given in Table 4.9.

TABLE 4.9: Properties of Body 6

Test Conducted	B.T.A	B.T.B
Drying Shrinkage (%)	4.28	3.94
Firing Shrinkage (%)	12.50	14.65
Total Shrinkage (%)	17.43	18.00
Water Absorption (%)	0.095	0.16
Apparent Porosity (%)	0.24	0.39
Bulk Density (gm per cc.)	2.425	2.405
Fired Colour: Internal	Greyish White	Greyish White
External	Greyish White	Tan tinged

BODY 7

	B.T.A (%)	B.T.B (%)
Kankara Kaolin	23	16
Bomo Clay	12	19
Okene Quartz	25	25
Okene Feldspar	40	40
Water (ml)	250	246
Solid Ingredients (gm)	500	500

A combination of 2.5gm sodium silicate and 1.5gm sodium carbonate was used to delocculate B.T.A. After the 24 hour observation, slight settling was observed and this situation persisted even after the addition of .5gm sodium silicate. This, evidently, was a case of over deflocculation because when a new batch was weighed out and the proportion of the deflocculants changed to 2gm sodium silicate and 1.5gm /sodium carbonate, there was no settling after leaving for 24 hours.

When deflocculated with 1.5gm sodium silicate and 1.5gm sodium carbonate B.T.B had good rheological properties.

Mould release of B.T.A and B.T.B was good and they both produced firm casts. The fired B.T.B however displayed more translucency.

TABLE 4.10: Properties of Body 7

Test Conducted	B.T.A	B.T.B
Drying Shrinkage (%)	1.97	2.29
Firing Shrinkage (%)	14.43	14.14
Total Shrinkage (%)	16.12	16.11
Water Absorption (%)	0.02	0.09
Apparent Porosity (%)	0.05	0.22
Bulk Density (gm per cc.)	2.385	2.365
Fired Colour: Internal	Greyish White	Greyish White
External	Tan tinged	Tan tinged

BODY 8

	B.T.A (%)	B.T.B (%)
Kankara Kaolin	43	36
Bomo Clay	12	19
Okene Quartz	15	15
Okene Feldspar	30	30
Water (ml)	250	250
Solid Ingredients (gm)	500	500

For effective deflocculation, B.T.A required an equal combination of 2gm sodium silicate and 2gm sodium carbonate. When observed after 24 hours, rheology of the slip remained good. On the other hand, the B.T.B required 2gm sodium silicate and 1.5gm sodium carbonate for optimum deflocculation. As with B.T.A rheology of the slip remained good after the 24 hour rest. Mould release of both bodies was early and good.

However, bodies only B.T.B displayed slight signs of

translucency after firing. Their other properties are given in Table 4.11.

TABLE 4.11: Properties of Body 8

	B.T.A	B.T.B
Drying Shrinkage (%)	3.94	4.60
Firing Shrinkage (%)	11.60	13.79
Total Shrinkage (%)	15.12	17.76
Water Absorption (%)	0.07	0.045
Apparent Porosity (%)	0.17	0.11
Bulk Density (gm per cc.)	2.38	2.43
Fired Colour: Internal	Greyish White	Greyish White
External	Tan tinged	Tan tinged

BODY 9

	B.T.A (%)	B.T.B (%)
Kankara Kaolin	33	26
Bomo Clay	12	19
Okene Quartz	25	25
Okene Feldspar	30	30
Water (ml)	246	246
Solid Ingredients (gm)	500	500

After the 24 hour rest slight settling was observed in B.T.A which was deflocculated with a combination of 2gm sodium silicate and 1gm sodium carbonate. When stirred and screened, the body produced firm casts, and after firing the body was not translucent. B.T.B, which was fairly translucent after firing, was deflocculated with 1.5gm sodium silicate and 1.5gm sodium carbonate. No settling was observed after the 24 hour rest. As with B.T.A mould release was early.

TABLE 4.12: Properties of Body 9

Test Conducted	B.T.A	B.T.B
Drying Shrinkage (%)	2.29	4.27
Firing Shrinkage (%)	11.77	13.30
Total Shrinkage (%)	13.80	17.09
Water Absorption (%)	0.13	0.03
Apparent Porosity (%)	0.31	0.065
Bulk Density (gm per cc.)	2.395	2.41
Fired Colour: Internal	Greyish White	Greyish White
External	Greyish White	Tan tinged

BODY 10

	B.T.A (%)	B.T.B (%)
Kankara Kaolin	23	16
Bomo Clay	12	19
Okene Quartz	35	35
Okene Feldspar	30	30
Water (ml)	250	250
Solid Ingredients (gm)	500	500

Deflocculated with 2.6gm sodium silicate and 1gm sodium carbonate, slight settling was observed in B.T.A after the 24 hour lapse. When stirred and screened bars were cast from this body which, undoubtedly, has the lowest wet to dry shrinkage of 0.97% (Table 4.13) out of all the bodies investigated in the study. With a wet to dry shrinkage of 2.29% B.T.B was deflocculated with 1.5gm sodium silicate and 1.5gm sodium carbonate. There was no settling after the observation period of 24 hours. With an early mould release as in B.T.A, B.T.B. was slightly translucent after firing.

TABLE 4.13: Properties of Body 10

Test Conducted	B.T.A	B.T.B
Drying Shrinkage (%)	0.97	2.29
Firing Shrinkage (%)	12.60	13.80
Total Shrinkage (%)	13.48	15.78
Water Absorption (%)	0.08	0.085
Apparent Porosity (%)	0.20	0.21
Bulk Density (gm per cc.)	2.395	2.40
Fired Colour: Internal	Greyish White	Greyish White
External	Greyish White	Tan tinged

BODY 11

	B.T.A (%)	B.T.B (%)
Kankara Kaolin	38	31
Bomo Clay	12	19
Okene Quartz	20	20
Okene Feldspar	30	30
Water (ml)	250	253
Solid Ingredients (gm)	500	500

Both B.T.A and B.T.B were deflocculated with the same amount of deflocculants 1.5gm sodium silicate and 1.5 sodium carbonate. No settling was observed after leaving to rest for 24 hours. Both bodies had an early and good mould release. B.T.B showed signs of translucency after firing. The other properties are presented in Table 4.14.

TABLE 4.14: Properties of Body 11

Test Conducted	B.T.A	B.T.B
Drying Shrinkage (%)	4.27	4.27
Firing Shrinkage (%)	11.68	13.70
Total Shrinkage (%)	15.45	17.43
Water Absorption (%)	0.095	0.125
Apparent Porosity (%)	0.23	0.32
Bulk Density (gm per cc.)	2.37	2.425
Fired Colour: Internal	Greyish White	Greyish White
External	Greyish White	Tan tinged

Materials Substitution Test

M.S.T

	%	Batch (gm)
Kankara Kaolin	19	190
Rakwamni Kaolin	19	190
Bomo Clay	12	120
Okene Quartz	10	100
Baure Quartz Sand	10	100
Okene Feldspar	30	300
Water (ml)		500

Basically, the M.S.T. composition is the same as that of 11 B.T.A which therefore, constituted the "control" for the M.S.T test. With a batch weight (solid ingredients) of 1000gm, a combination of 3gm sodium silicate and 3gm sodium carbonate was needed to provide an excellent slip which did not settle after the 24 hour observation period. After stirring and screening, test bars were cast and there was an early mould release. Like the control with which it is compared in Table 4.15, the fired M.S.T did not show any sign of translucency. It however had a high wet to dry shrinkage (5.0%) which is the highest drying shrinkage recorded by the study, in effect its percentage total

shrinkage Table 4.15) is higher than that of the control by as much as 1.05%. This high shrinkage finds expression in its relatively low porosity and higher bulk density. Since the introduction of the "new" materials was not inimical to either the bulk density or the apparent porosity, comparatively speaking now, the test can be said to be successful. Further seen from the viewpoint of reproducibility, the deviation from the control is infinitesimal thus confirming that it is possible to achieve "consistent" results if the experimental quantity of 500gm used in the control is increased to 1000gm or even more.

TABLE 4.15: Properties of M.S.T

TEST CONDUCTED	M.S.T	CONTROL
Drying Shrinkage (%)	5.00	4.27
Firing Shrinkage (%)	12.10	11.60
Total Shrinkage (%)	16.50	15.45
Water Absorption (%)	0.04	0.095
Apparent Porosity (%)	0.10	0.23
Bulk Density (gm per cc.)	2.38	2.37
Fired Colour: Internal	Greyish White	Greyish White
External	Greyish White	Greyish White

Talc Addition Test

T.A.T

	%	Batch (gm)
Kankara Kaolin	19	190
Rakwamni Kaolin	19	190
Bomo Clay	12	120
Okene Quartz	10	100
Baure Quartz Sand	10	100
Okene Feldspar	30	300
Isanlu Talc	2	20
Water (ml)		510

For the Talc Addition Test, the "control" is M.S.T because its composition was retained and 2% Isanlu Talc added to it for reasons earlier stated. The 2% was calculated as a percentage of the 1000gm solid ingredients.

For effective deflocculation, T.A.T required 3gm sodium silicate and 3gm sodium carbonate. After the 24 hour time lapse, fluidity and thixotropy remained good. When stirred and screened nice firm casts were produced. The wet to dry shrinkage of T.A.T was 2.0% lower than that of the control, 0.4% less than the dry to fired shrinkage of the control (Table 4.16) and with a total shrinkage which is 1.5% less than that of the control. The porosity of T.A.T (even though its bulk density is higher by 0.003gm per cc.), is however not as favourable as that of the control. Like the control, there was no discernible sign of translucency.

TABLE 4.16: Properties of T.A.T

Test Conducted	T.A.T	CONTROL
Drying Shrinkage (%)	3.0	5.0
Firing Shrinkage (%)	12.50	12.10
Total Shrinkage (%)	15.00	16.50
Water Absorption (%)	0.053	0.040
Apparent Porosity (%)	0.14	0.10
Bulk Density (gm per cc.)	2.383	2.380
Fired Colour: Internal	Grey	Greyish White
External	Grey	Greyish White

4.4. Discussion

By the postulation of Hamer (1975), "Highly plastic clays (such as ball clays) require more water than low plasticity clays to bring them to the plastic state." If

"plastic state" may be taken to mean optimum workable consistency then the view of Hamer (1975) is at variance with the calculated percentage water of plasticity as it affects Kankara Kaolin (not "plastic"), Rakwamni kaolin (also not "plastic"), and Bomo Clay which is "plastic".

With reference to Table 4.1, Kankara and Rakwamni kaolins which could not withstand plastic deformation without cracking clearly required more water than the "very plastic" Bomo clay which was able to withstand plastic deformation without cracking. The index for the clays being 0.55; 0.39; 0.36 for Kankara, Rakwamni, and Bomo respectively. This ordering persisted even for samples that had been dried to constant weights at 110°C before carrying out the test.

Understandably, the view of Hamer (1975), like those of Rhodes (1973) and Worrall (1982), is predicated on the premise that since most plastic clays have smaller sized particles, they (i.e the smaller sized particles) offer more surface area per given weight of clay than do the relatively larger particles. Invariably, there is a greater surface area to be surrounded by water in fine clays. If this be the case then one would be inclined to think that the agglomerates, of say Kankara kaolin, have a corrugated surface of larger area.

Also in line with the surface area requirements is the view of Adekale (1983) that maximum plasticity increases linearly with increasing proportion of fine particles while solid concentrations for maximum plasticity is influenced by the proportion of coarse particles.

Various investigators have established a relationship

between the grain size distribution of a clay, its water of plasticity, duration of drying, plastic to dry shrinkage, and loss in weight of specimens where the physically added water had been removed by evaporation.

While indicating that shrinkage increases with the amount of colloidal material (i.e. particles of submicron size), Worrall (1982) pointed out that Ca^{2+}, Mg^{2+}, and H^{-} clays shrink more than the corresponding Na^{+}, or K^{+} clays. Further to drying shrinkage, Griffiths and Radford (1965) observed that, depending on the mix and firing schedule employed, the "melting of the more fusible components which occurs above 900°C during heat treatment" causes a further shrinkage.

The shrinkage determined in this study was the linear shrinkage and differs from the dimensional change in volume which is estimated by Griffiths and Radford (1965) to be approximately three times the linear shrinkage of the same material. Cardew (1969) contends that "Since it is not possible to make clay test pieces which do not have some alignment of the clay particles, shrinkage figures, however carefully measured, will not represent the 'true' linear shrinkage but only that measured parallel to the horizontal axes of the plate-shaped clay particles. The shrinkage parallel to their vertical axes will be greater." This view has since been vindicated by the work of Robinson (1978).

It can be taken for granted that the further milling of the materials (as part of a batch in this study) for 8 hours produced additional reduction in particle sizes thus altering the grain distribution from that presented by the sieve

analyses and the cummulative distribution curves presented in 4.2. Since further particle reduction increases the surface area, thus the surface energy, it can be argued that the sinterability of the powders had been further enhanced.

The settling phenomenon observed in some slips which had been treated with deflocculants is not acceptable for several reasons. First, in high-volume industries where slip is stored in silos and moved around by piping system this poses a problem of blockage. Secondly, if such a slip is used for casting purposes, there is the tendency for the ingredients to sediment according to their weights giving rise to problems associated with segregation: homogeneity of the mix is upset, resulting in strength loss.

Settling of slips denotes a situation where the slip has either been overdeflocculated or underdeflocculated. A case in point is that of 7 B.T.B which settled after it was deflocculated with 3 gm 75^{o}Tw sodium silicate and 1.5gm sodium carbonate. When a new batch was deflocculated with 2 gm 75^{o}Tw sodium silicate and 1.5gm sodium carbonate, there was no settling.

The idea behind deflocculation of slips meant for casting is not really to raise the zeta potential so high that deflocculation is at its peak. In a situation like this the particles will continue repelling each other in the mould while local drying is slowly being effected by the "neutralization" of the zeta potential brought about by flocculation introduced by the calcium sulphate mould acting in conjunction with the natural forces of attraction. Increased local drying of this nature produces

an impervious layer which makes the slip "impermeable" resulting in prolonged casting time and flabby casts.

The lower limit of 3gm of deflocculants to every 500gm solid ingredients sufficed in most cases except in isolated cases where this was exceeded to give an upper limit of 4gm deflocculants to every 500gm solid ingredients.

Some properties of the investigated compositions were presented in the previous section. To fully comprehend the trend it will be necessary to consider the P.T.P of every given body as an extension of the P.T.A of the same body, with the "melting" point of the latter batch now lowered as a result of the new eutectic points introduced by the increase in Bomo with every reduction made to Kankara kaolin. For every given body, therefore, there is a point about the vitrification range where bulk density will reach a maximum then a little above this point, say 10°C higher, the point of minimum apparent porosity is reached; For an english-type electrical porcelain investigated by Waye et al (1963) maximum bulk density and strength occured at the same temperature of 1250°C before minimum apparent porosity of 0.06% was attained at 1260°C.

In this model it is granted that maximum bulk density and strength are achieved before minimum apparent porosity is attained. Beyond the point of minimum apparent porosity, more of the high-density quartz will be taken into the glassy solution to give a glass phase of lower density (and probably lower linear expansion coefficient). The cumulative effect of this and the gradual expansion of sealed pores is that bulk density starts to fall followed by

an increase in the apparent porosity. Rapid escape of gases would probably account for the increase in apparent porosity.

For the purpose of this discourse, the study will append the terms "Up traits" and "Return syndrome" in the classification and analyses of data collected.

Under the "Up traits" category are those bodies which are "yet" to attain, or rather yet to exceed, their point of either maximum bulk density or minimum porosity hence, there is a reduction in apparent porosity and an increase in both fired shrinkage and bulk density of the B.T.B of such bodies relative to their B.T.A with increase in heatwork. Also, the tan tinge is more pronounced in the external colour of the B.T.B of these bodies than their B.T.A. Included here are bodies 8 and 9.

The bodies exhibiting the "Return syndrome" are 3, 4, 5, 6, 7, 10, 11. Having "exceeded" their points of maximum bulk density and minimum porosity, there is a "return" effect in the B.T.B of these bodies: bulk density drops and apparent porosity increases. Fired shrinkages of the bodies fluctuate so that it is possible to have a "Return syndrome" body where the difference between apparent porosity, bulk density and fired shrinkages between its B.T.A and B.T.B is infinitesimal suggesting that the body has just started to "return" as in the case of body 4.

What is worthy of note at this point is that the sintering process involves the consolidation of particles at high temperatures meaning that surface area and surface

energy are reduced so that the "return" rate is not expected to be as rapid as the rising "up" rate where surface energy is higher.

In addition to being tan tinged externally, the B.T.B of bodies in the "Return syndrome" category also displayed a high degree of translucency (with the exception of body C which was not as translucent as the others). Bodies 1B.T.A and 2B.T.A were not fired for reasons already given but the fired B.T.B of both bodies were very translucent. The absence of their B.T.A. therefore, makes comparative analyses difficult as regard their classification.

The ongoing would then seem to suggest that translucency is accompanied by a reduction in bulk density and a rise in apparent porosity. It is also note-worthy, the fact that very translucent bodies - falling under the "Return syndrome" category - have similar fired shrinkage values with a calculated dispersion range of 1.65, with no bloating tendencies obvious to the naked eye.

At this juncture there is the need to resort to literature if extrapolations are to be made in the absence of facilities needed for further tests.

According to Glenny (1963), the observed strength at fracture of brittle materials is dependent on chance "Simply because such materials contain defects or regions of weakness that act as stress concentrations" failing at its weakest link. He referred to the study of Duckworth (1961) who, using Weibulls statistical theory of failure, found that the material constant "m" for steel at liquid-air

temperature was no higher than that for ceramics. The material constant "m" reflects the degree of homogeneity of material; a high "m" value in a material indicates a large number of flaws per unit volume with the probability of failure increasing with the number of flaws.

In a related view Waye (1967) argued that in the case of electrical porcelain insulator an unusually high power factor might cause the insulator to heat up which could result in instability and even breakdown in severe cases. He further noted that in porcelain this breakdown would "indicate a serious fault in manufacture such as gross contamination, or open porosity." The latter would "put up the power factor due to moisture absorption." Developing the argument further Burmeister et al (1978) opined that "moisture is by far the greatest enemy of insulation; consequently the absence of hygroscopic quality is desirable." Table 4.17 is self explanatory and the only addition to it would be the view of Kingery (1976) that "failure events are almost always initiated at regions of physical or chemical inhomogeneity. This is also true for other properties dependent on failure events, such as thermal shock resistance, dielectric strength, and high temperature deformation. Defects can also be introduced by inadequate firing processes - warping, bloating, overfiring, black core, dunting and so forth."

The work of Waye et al (1963) has shown that the maximum strength, though obtainable at the point of maximum bulk density, falls off gradually before the attainment of minimum apparent porosity. Invariably the maximum

TABLE 4.17

Some Typical Fired Properties of Porcelain And Factors Responsible

PROPERTY	UNIT	VALUE	FACTORS AFFECTING VALUE
Apparent Porosity	-	0.1%(pl)	Composition; firing
Apparent Specific Gravity	-	2.3-2.5	Composition; firing
True Specific Gravity	-	2.4-2.5	Composition; firing
Volume Resistivity	ohm.cm	10^{12}-10^{14}	Lowered by high Na; falls rapidly with rising temperature
Te	°C	300-500	Lowered by high Na
Dielectric Strength	KV/mm	25-35 at	Lowered by inhomogeneities; affected by conditions of measurement e.g falls with increasing thickness of test piece and temperature.
Dielectric Constant	-	5.5-6	Lowered by (sealed) porosity.
Tensile Strength	$lb/in^2 x10^3$	3-6(u,pl) 1.5-3(u,pl) 4-8(g,pl) 2-4(g,pr)	Decreased by porosity or flaws especially on surface, affected by method of measurement, e.g shape of test piece.
Coeff. of Thermal Expansion	$x10^{-6}/°C$	4.5-6.5(s) 3.5-5.5(h) (20-700°C)	Composition-decreases with increase of SiO_2 dissolved in glassy phase

Key:

pl - Plastically made (not pressed), pr - pressed

s - 'soft' porcelain, h - 'hard' porcelain

g - glazed, u - unglazed

Source: Waye (1967)

attainable strength and some other desired properties would require a compromise between the apparent porosity and the bulk density. These considerations were instrumental in the selection of likely bodies for electrical insulation (Table 4.18).

In Table 4.18, the 0.05% apparent porosity calculated for body 7 B.T.A is even lower than the minimum apparent porosity of 0.06% which Waye et al (1963) got for an english electrical porcelain body. The bulk densities of the selected bodies, and in fact all the compositions investigated in the study, fall within the 2.3 - 2.6 range given by Waye (1967) and Wiegmann (1980). Also the 14.2 - 17.8% fired shrinkages of the given bodies/similar to the 15% given by Singer and Singer (1963).

It will be discovered that, in line with the "Up traits" and "Return syndrome" which this study is proffering, the only compositions selected from the "Up traits" are from their B.T.B. Converesely, those from the "Return syndrome" all come from their B.T.A with the exception of body 7 which, for reasons already given, is represented by its B.T.A and B.T.B.

As far as the property tests conducted in this study are concerned, the substitution of Rakwamni kaolin for Kankara kaolin and Baure quartz sand for Okene quartz - within limits defined by the study - will not produce an adverse effect. The effect of the substitution and the addition of Isanlu talc (T.A.T) on mechanical and electrical properties could, however, not be determined quantitatively

TABLE 4:18: Properties of Selected Compositions

BODY	% Linear Shrinkage			% Water Absorption	Apparent Porosity(%)	Bulk Density (gm. per c.c)	Fired Colour	
	Drying	Firing	Total				Internal	External
4 B.T.A	2.95	13.55	16.11	0.04	0.10	2.405	Greyish White	Greyish White: Tan tinged.
4 B.T.B	3.94	13.30	16.78	0.05	0.11	2.405	Greyish White	Greyish White: Tan tinged.
5 B.T.A	1.96	12.33	14.15	0.065	0.16	2.36	Greyish White	Tan tinged
7 B.T.A	1.97	14.43	16.12	0.02	0.05	2.385	Greyish White	Greyish White
8 B.T.B	4.60	13.79	17.76	0.045	0.11	2.43	Greyish White	Tan tinged
9 B.T.B	4.27	13.30	17.09	0.03	0.065	2.41	Greyish White	Tan tinged
T.A.T	3.00	12.50	15.00	0.053	0.14	2.383	Greyish White	Greyish White
M.S.T	5.00	12.10	16.5	0.04	0.10	2.38	Greyish White	Greyish White

KEY: B.T.A = Bomo Test "A"
B.T.B = Bomo Test "B"
T.A.T = Talc Addition Test
M.S.T = Materials Substitution Test

due to limitations already acknowledged at the beginning of the study.

4.5. Findings

As a result of the tests conducted in this investigation, the following observations were made:

1. Kankara kaolin, with reference to its observed behaviour, is a highly adsorptive clay.
2. The nature and colour of the melt produced by Zaria feldspar is not the same as that associated with "good" potash feldspar - needed in porcelains in general and electrical porcelains in particular.
3. Petramin talc would appear to be a poor quality talc which is not suitable for the type of pottery envisaged here.
4. Talc is not easily wetted so it is better to dry-mix with the batch before wet milling or, alternatively, overcome the problem by the use of fairly hot water.
5. The addition of 12% Bomo clay is enough to enhance castability and dry strength.
6. The method of milling the batch before treating with deflocculants is a convenient check on the problem of specific gravity fluctuation observed in the study of Okoruwa (1986).
7. A lower limit of 3gm and an upper limit of 4gm deflocculants to every 500gm solid ingredients was found to be adequate for most applications.
8. Under-deflocculation or over-deflocculation are likely causes of the settling phenomenon observed in the study.

9. The tan tinge observed on the surface of some of the specimens was caused by the Pomo clay at a certain point in the course of Heat treatment, probably acting in conjunction with the glaze containing atmosphere and to a lesser extent, the firing schedule employed in the study since the internal colour of specimens was not affected at all.

10. Percentage apparent porosity, by approximation, is twice the value of the percentage water absorption. By definition the former gives the ratio of open pore volume relative to total volume of the test piece while the latter gives the ratio of open pore volume relative to the weight of the specimen in question.

11. The high-clay bodies had a high wet to dry shrinkage.

12. Translucency is almost always accompanied by a lowering of bulk density and increase in both apparent porosity and fired shrinkage.

13. For each composition there is a minimum attainable apparent porosity and a maximum bulk density and, by necessary implication, each body has its own maximum attainable strength.

14. From the apparent porosity point of view, the 0.05% achieved in 7 B.T.B is an improvement on the 0.06% which Waye et al (1963) got for an english - type electrical porcelain; the bulk densities and shrinkages recorded by the study accord with the ranges given by Singer and Singer (1963), Waye (1967) and Wiegmann (1980). Based on the above and the discussion in the previous section, the conclusion can be drawn that the area from which the

compsitions were drawn (i.e. from the clay-Feldspar-Quartz triaxial blend) if anything, was well pin-pointed.

15. Kankara kaolin, Rakwamni kaolin, Bomo clay, Okene quartz, Baure quartz sand, Okene Feldspar, and Isanlu talc are suitable for porcelain production.

16. Translucency is more pronounced in the relatively high-feldspar bodies.

CHAPTER FIVE

SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

5.1. Introduction:

Given the observed limitations, the purposes of this study were the formulation of a porcelain body suitable for electrical insulation from local sources and to determine the extent to which the locally formulated body will compare with the imported one. This chapter is a synopsis of the observations based on the various tests conducted in the course of the study with a view to achieving the stated purposes.

5.2. Summary:

Prior to compounding the different compositions investigated in the study, attempts were made to characterize the procured materials. This was necessary to: identify and, possibly reject any material which lacked the desired characteristics as in the case of Zaria feldspar and Petramin talc; to gather information for future references; ensure quality and, on a more practical note, make predictions and adjustments possible.

An alternative to material characterization, though a costly one, was offered by Flock (1978) who suggested transferring the problem of characterization to process control which places production within the control of the investigator. This, obviously, is better suited for situations such as ours where powerful characterization

tools are not available.

Some of the procured materials, namely Okene feldspar, Okene quartz, Baure quartz sand, Rakwamni and Kankara kaolins, and, to some extent, Bomo clay, have all proven to be very good materials for porcelain production. It should however, be noted in passing that these minerals are not the only ones available in the country.

The specific gravity and casting time of the porcelain slips, setting time of the plaster of paris for the casting moulds are variates that are determined by the results desired.

5.3. Conclusions:

The results obtained from the various tests conducted in the study have made it possible to draw the following conclusions:

1. The materials used in the formulation of the selected bodies, if processed and formulated according to the procedure in this study, will give consistent results in both high - and low-volume applications.
2. Zaria feldspar and Petramin talc are not suitable for porcelain production.
3. Addition of Bomo plastic clay, within defined limits, enhanced castability, dry strength, and densification as noted by Rhodes (1960).
4. Problem of specific gravity fluctuation can be overcome by milling and drying the batch before treating with deflocculants.
5. A lower limit of 3gm and an upper limit of 4gm deflocculants (combination of 75°Tw sodium silicate

and sodium carbonate) to every 500gm of dry solid ingredients was found to be adequate in most cases. This accords with the views of Worrall (1982) and Okoruwa (1986).

6. A wet-ball milling time of 6 hours with a 1:1:1 ratio of grinding balls to batch to water is enough to give the batch a homogeneous mix.
7. Raw materials required for porcelain production are locally available.

5.4. General Recommendations:

The introduction of technical ceramics into our hitherto primordial ceramic culture will set a "production possibility frontier" in motion in which case alternative sources of materials would have to be sought, suffice it to say that there is the need to discover other sources of ceramic raw materials so that in the long run, production of technical ceramics will not be inimical to the production of other more mundane wares moreso when about the same materials and facilities are required in the production of the two categories. Other recommendations which this study has to make are:

1. That the University makes available more books on technical ceramics, as relevant literature in this area is minimal. An extension to this would be to further implore the authorities to put more materials and equipment at the disposal of researchers.
2. There should be a more intimate interaction between ceramics and pure science with a view to broadening the

present narrow and empirical sphere. Further to this is the need to have a greater flow of ideas between ceramic schools, the ceramic industry, ceramic societies, and allied associations.

3. Researches of this nature should be sponsored by individuals, institutions or government establishments like National Electric Power Authority (N.E.P.A.), and the Directorate of Food, Roads and Rural Infrastructure (D.F.R.R.I.) which are currently involved in the rural electrification scheme. The Raw Materials Research and Development Council is also expected to play an active role in this regard.
4. There is the need to conduct more researches in the area of processing so as to cut down on the costs of production while quality is improved upon.

5.5. Recommendations for Further Research:

The study is recommending the following for further research:-

1. The determination of the base exchange values of Kankara, Rakwamni, and Bomo clays;
2. Determination of the chemical compositions of Rakwamni kaolin, Bomo clay, Okene quartz and feldspar, and Toanla talc;
3. The possibility of developing deflocculants from local sources;
4. Development, from local raw materials, of semi conducting glazes which can control flashover on high tension insulators under pollution conditions;

5. There is the need to assess the mechanical and electrical properties of the selected compositions;
6. The possibility of substituting some of the materials, piece meal or wholly, with other materials not investigated in this study.

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APPENDIX I

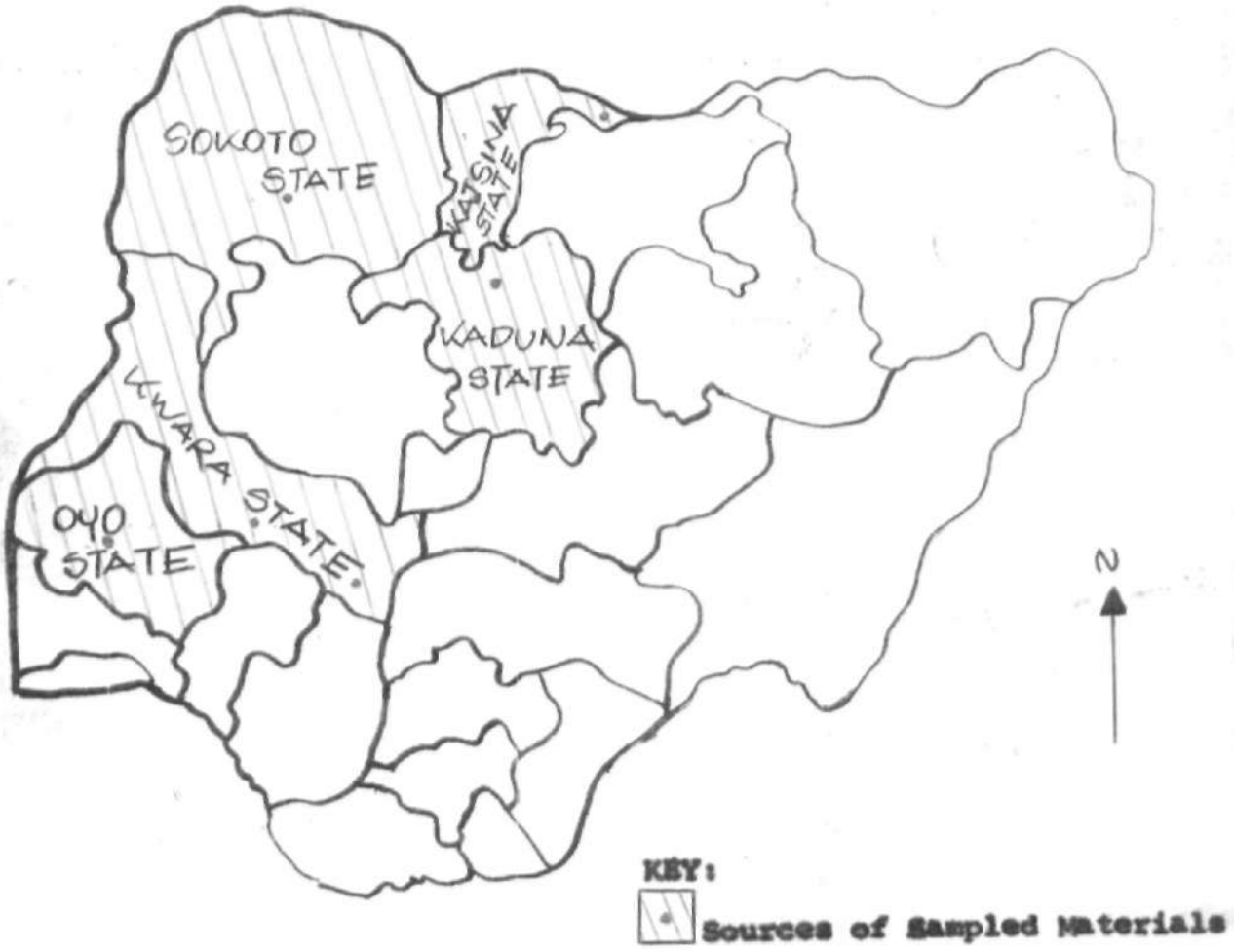


Fig. 9: Map of Nigeria Indicating Raw Material Sources

APPENDIX II

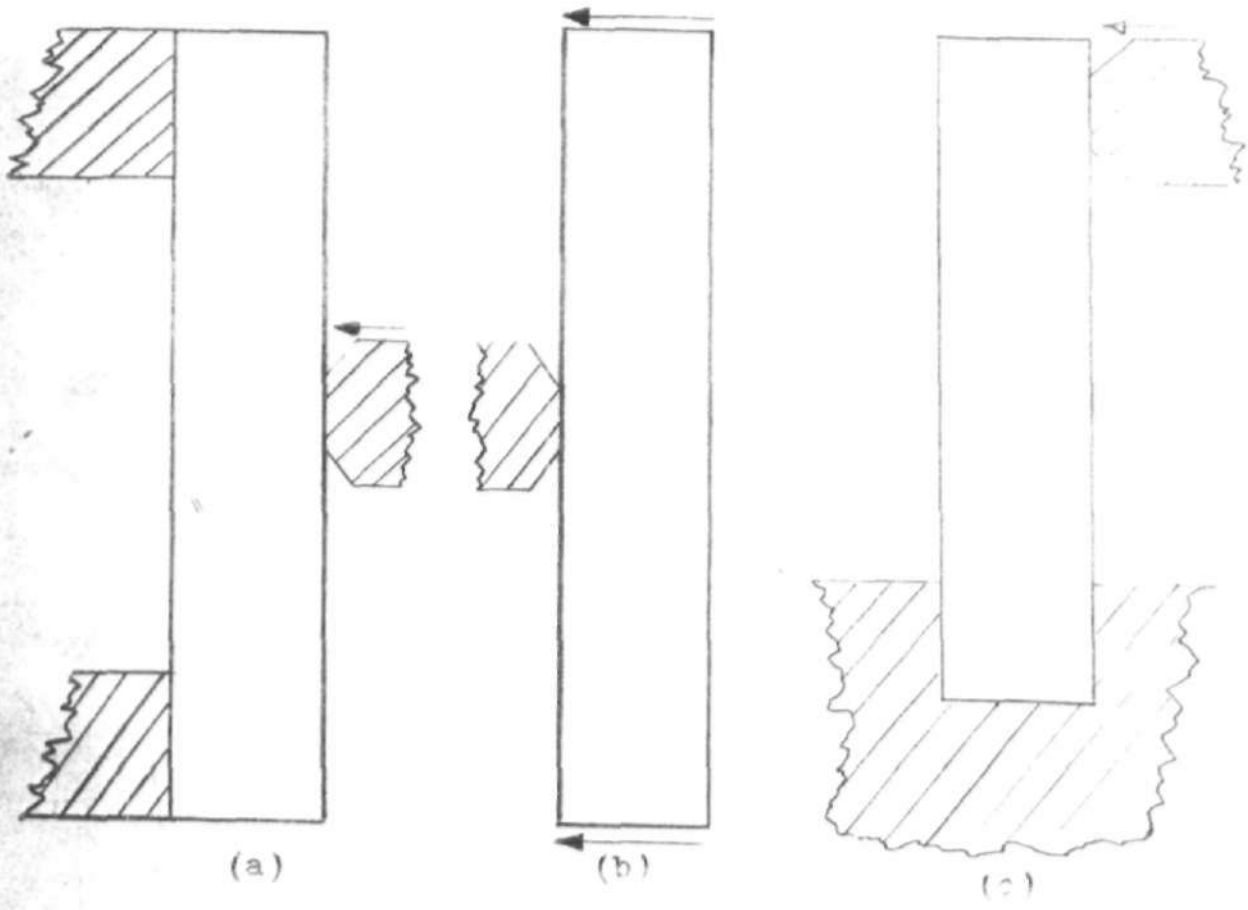


Fig. 10

Methods for determining the impact resistance of Ceramics: (a) Charpy-type test (top view), the specimen is supported at its ends and struck at its centre by a pendulum pivoted above the specimen; (b) Modified Charpy-type test (top view), the specimen acts as the pendulum and strikes a fixed anvil; (c) Izod-type test (side view), the specimen is clamped vertically and struck by a moving pendulum pivoted above the specimen. (After Davidge, 1979).

APPENDIX III

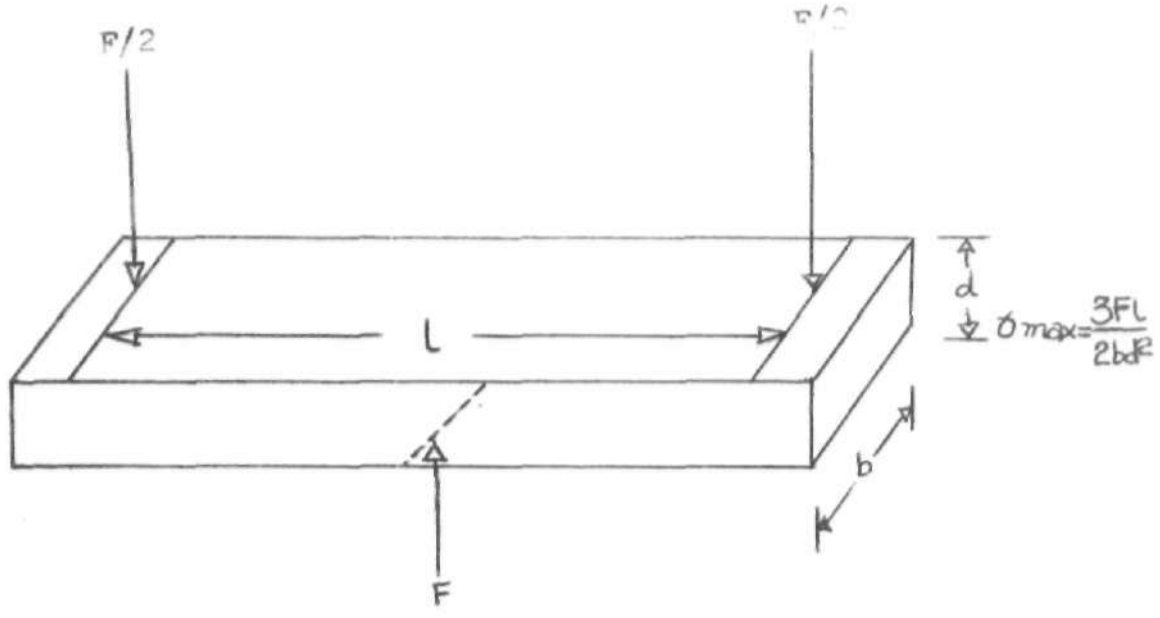


Fig. 11

Three-point bend test and the relationship between maximum stress, applied force and specimen dimensions. The maximum stress occurs along a line on the specimen face opposite the central knife-edge. The measured fracture stress is often referred to as the modulus of rupture. The disadvantages of the test are that only a small area of the specimen surface is subjected to the maximum stress and stress gradients exist both along the bar and through the thickness of the material. (After Davidge, 1979).

APPENDIX IV

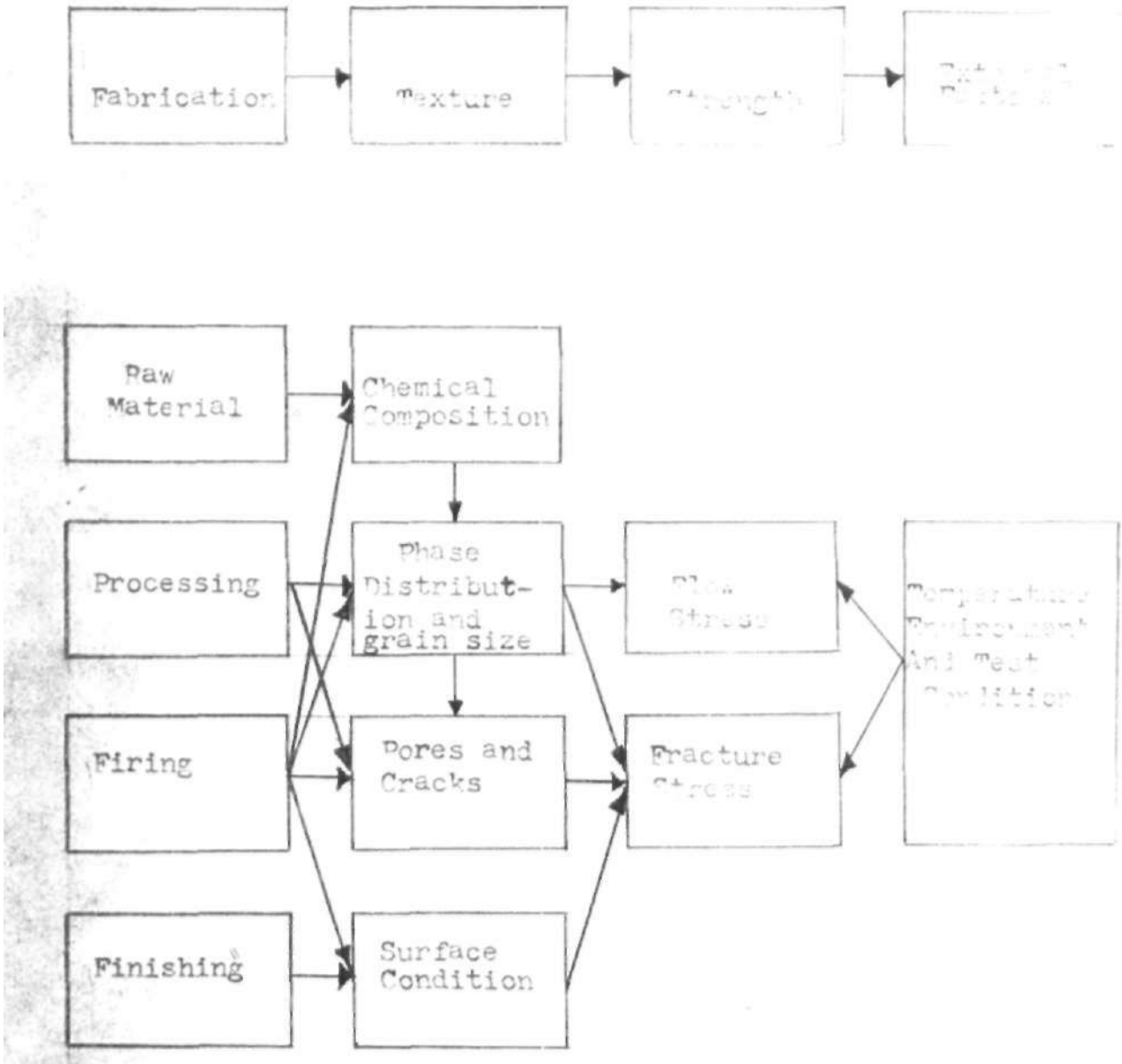


Fig. 12: The Major Fabrication And Textural Factors Affecting the Strength of Ceramics (After Davidge, 1979).

APPENDIX V



PLATE III: Grain Size Determination: Sieving.



PLATE IV: Grain Size Determination: Hydrometer

APPENDIX VI



PLATE V: Fired Specimens: Pressed Compacts And Slip-Cast Bars



PLATE VI: Slip-Cast Experimental Samples

APPENDIX VII



PLATE VII: Water Distiller