# DEVELOPMENT OF DRY PRESSED
# E L E C T R I C A L P O R C E L A I N B O D Y

A THESIS

PRESENTED TO

THE POST GRADUATE SCHOOL

AHMADU BELLO UNIVERSITY, ZARIA

BY

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IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR
THE DEGREE, MASTER OF SCIENCE (CHEMICAL
ENGINEERING)

DEPARTMENT OF CHEMICAL ENGINEERING
AHMADU BELLO UNIVERSITY
ZARIA - NIGERIA.

FEBURARY, 1994

# DECLARATION

I, USMAN UMAR, declare that this Thesis is solely the result of my work and has never been submitted any where for any Degree. All literature cited have been duly acknowledged in the References.

USMAN UMAR

FEBRUARY 1994

## CERTIFICATION

This Thesis entitled DEVELOPMENT OF DRY PRESSED ELECTRICAL PORCELAIN BODY by USMAN UMAR meets the regulation governing the award of the degree of Master of Science (Chemical Engineering) of Ahmadu Bello University, Zaria, and is approved for its contribution to knowledge and literary presentation.

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## DEDICATION

This work is first and foremost dedicated to Allah (Subahanahu-wa-Ta'ala) for the strength, knowledge, and wisdom He bestowed on me that enabled the completion and success of the work. It is also dedicated to my parents, Mallam Usman Adamu and Mallama Abu Usman, my beloved wife, Hajiya Maimuna Yusuf and lastly my first daughter Zainab Ummie Usman (Miss) for their moral support and understanding which were essential for the successful completion of this work.

## ACKNOWLEDGEMENT

I would like to express my sincere gratitude to all those who have assisted in one way or the other, no matter how small, to make this work a success.

I am particularly grateful to Dr. Ir. P.B. Onaji who supervised the work, and offered constructive criticisms to the best of his ability. I am also in-debted for his inexhaustible patience in waiting for so long, for my release by my employer for the work on full time basis.

My gratitude also goes to my second internal examiner, Dr. K.S. Ahmed for the keen interest he showed in the work, and his contributions during its execution.

Special thanks and gratitude go to Nigerian National Petroleum Corporation, Port Harcourt Refining Company, my employer, for granting me eight months study leave for the execution of the research work.

Finally, I would like to thank the entire staff of Chemical Engineering Department who have rendered assistance, and also Alh. Yakubu Usman who typed this thesis.

## ABSTRACT

Electrical porcelains are white vitreous ware insulators made from clay, feldspar, and quartz. There is little or no local production of these porcelains inspite of availability of raw materials and fuels required for their manufacture.

Little work has been done on developing bodies for the porcelains using slip casting. The present work focuses on developing low tension porcelain using dry pressing method.

Nine clays from different sources were studied for important ceramic characteristics, based on which two clays, namely, KanKara and Bida clays, with favourable characteristics for dry pressing electrical porcelains were chosen.

Four porcelain bodies, namely, body A (45% clay, 30% Silica sand, 25% feldspar), body B (45% clay, 25% silica sand, 30% feldspar), body C (50% clay, 20% Silica, sand, 30% feldspar) and body D (40% clay, 25% silica sand, 35% feldspar) were studied. Four different firing temperatures, namely, 1180, 1220, 1260, and 1300$^{o}$C were used. Dry pressing mode of production was employed.

Kano KRD, KanKara, Cross River, and Ikebedi clays are most probably Kaolins while Bomo, Goronyo MWF1and Bida were found to be stone ware clays probably. Kaoje clay showed the highest pfefferkorn plasticity index while Kano KRD showed

the lowest. The minimum viscosity studies showed that cross river had the highest while Bomo had the least.

Bodies 'B', 'C', and 'D' were very adequate for producing dry pressed electrical porcelains using Kankara and Bida clays, Okene feldspar and Danbatta silica sand. No good porcelain was found using a firing temperature of 1180 or 1220°C. Body 'C' showed the most favourable green properties. Firing temperature of 1260°C proved to be the best firing temperature, within the investigated range, and the ranges of the properties developed at this temperature are shown on table 1.

TABLE 1: RANGES OF FIRED PROPERTIES OF THE DEVELOPED PORCELAINS FIRED AT 1260°C.

| Property | Range |
|---|---|
| Linear firing shrinkage (%) | 7.94 - 8.31 |
| Firing Weight loss (%) | 5.41 - 6.77 |
| Fired Density $Kg/m^3$ | 2164 - 2238 |
| Fired MOR $KN/cm^2$ | 2.92 - 3.46 |
| Apparent Porosity (%) | 0.08 - 6.92 |
| Water absorption (%) | 0.04 - 3.21 |

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## LIST OF NOTATIONS

| | |
|---|---|
| R | Radius of Cylinder (ft) |
| r | Average radius of grinding balls (ft) |
| N | Number of revolutions Per minute |
| J | Energy Spent per Unit Volume |
| B | Von Rittinger's Constant of Proportionality |
| D | Particle Size |
| G | Kick's Proportionality Constant |
| F | Bond's Proportionality Constant |
| E | Young's Modulus |
| V | Fracture energy |
| L | Crack length |
| S | Stress |
| 0 | Porosity |
| P | Pressure |
| R′,S′,T | Constants |
| V* | Fraction of Volume decrease to the total initial void volume of the powder |
| a | Maximum Possible Fractional Volume Compaction Associated With one particular |

| | |
|---|---|
| | densification Mechanism, that is, particle slippage or filing of similar holes. |
| X | Approximate Pressure Under which a particular Mechanism, Particle Slippage, or Filling of similar holes by fragementation or plastic flow, are most likely occuring |
| V′ | relative Volume |
| C′ | Constant |
| SG | Relative density |
| K′ | Material Constant |
| B′ | Constant Associated with densification by particle rearrangement. |
| C* | Degree of volume reduction |
| a′,b | Constants (Characteristics of the Powder) |
| V | Volumetric change after time t |
| | Surface tension of the body |
| d | Diameter of Constituent particles |
| F′ | Maximum breaking force for MOR |
| S" | Span (distance between Supports) for MOR |

Subscripts

| | |
|---|---|
| s | Solid |
| v | void |
| L | Liquid |
| 1,0 | Initial |
| 2 | Subsequent |
| PS | Particle Slippage |
| HF | Hole filling |

| | | |
|---|---|---|
| | | Infinite |
| P | | Packing |

Abbreviations

| | |
|---|---|
| SQRT | Square root |
| Log | Logarithms |
| Exp | Exponent |
| In | Natural Logarithms |
| LDS | Linear drying shrinkage |
| LFS | Linear Firing Shrinkage |
| GD | Bulk Green Density |
| FD | Bulk Fired Density |
| GMOR | Green Modulus of Rupture |
| FMOR | Fired Modulus of Rupture |
| FWL | Firing Weight Loss |
| AP | Apparent Porosity |
| WA | Water Absorption |
| FC | fired Colour |
| Ld | Dried length |
| LF | Fired length |
| Wd | Dried Weight |
| WF | Fired Weight |
| Bd | Dried breadth |
| Hd | Dried depth |
| Wsat | Saturated Weight |
| Ws | Suspended Weight |
| Li | Initial length (before drying) |
| BF | Fired breadth |
| HF | Fired depth |
| dd | Dried diameter |
| dF | Fired diameter. |

# CHAPTER ONE

## INTRODUCTION

Electrical porcelains are very important in technological development, and presently, almost all Nigerian requirements are being met through imports, though raw materials needed for their local production are abundantly available.

Previous investigations were in slip casting, which is used to produce high tension insulators, mostly. Lower shrinkages, which lead to ease of shaping intricate and accurate shapes and dimensions are frequently very necessary, and hence the need for dry pressing, though only low tension porcelaine are normally produced with dry pressing.

It is, therefore the objective of this work to employ dry pressing together with local raw materials to develop low tension insulator bodies.

Nine clays from different sources, in Nigeria, were studied for their physical properties, and two clays, namely, Kankara and Bida clays, with favourable properties regarding dry pressing and electrical porcelains were chosen. Four different bodies, A,B,C, and D were used to produce the test specimens for both green and fired properties. Firing was done at four different peak temperatures; 1180, 1220, 1260 and 1300$^{o}$C, and Soaking times. However, duration of heat treatment, in all cases, was kept same and equal to 23.75 hrs.

Test Specimens were tested only for physical properties because according to Johnson and Robinson (1975), electrical properties are not critical, in practice, insulator designs based on mechanical considerations, generally have sufficient electrical strength.

Once a good porcelain body has been developed, commercialisation could be done through scaling-up of the processes of manufacture.

# CHAPTER TWO

## LITERATURE SURVEY

### 2.1 COMPOSITIONS

A ceramic material particularly suitable for electrical insulation is obtained by firing a mixture containing 5-35% beryl in addition to the usual ingredients, such as clay, silica, and feldspar. British - Houston (1921).

Table 2. shows the compositions used, by Watkins (1923-4), in his work on electrical porcelain. Compositions shown in Figure 1 were used for work on high tension insulators. Reichau (1924-5). A wide range of traditional ceramic compositions are mixtures of clay, feldspar, and flint, and these compositions include hard porcelain, table ware, vitreous sanitary ware, hotel china, electrical porcelain, and a host of others. Kingery (1960). The amount and kind of clay used are largely determined by requirements dictated by the forming technique; as more difficult forming techniques are employed when a larger content of China clay is required. Ball clay is more plastic, and hence facilitates use of easier forming techniques, but contains much impurities. Consequently, a balance is struck by using as much China clay as will permit successful forming. Compositions such as low tension electrical porcelains are not critical as regard either forming or firing operatioins.

TABLE 2: Porcelain Compositions with their determined Suitable Firing Temperatures Tried by Watkin (1923-4) in his Work on Electrical Porcelain

| Number | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| China Clay | 30 | 30 | 30 | 30 | 30 | 30 | 15 | - | 30 | 30 |
| Ball Clay | 25 | 25 | 25 | 25 | 15 | 35 | 40 | 20 | 25 | 25 |
| Flint | 30 | 20 | 10 | - | 30 | 10 | - | 15 | - | 20 |
| Feldspar | 15 | 25 | 35 | 45 | 25 | 25 | 45 | 30 | 35 | 10 |
| Red Clay | - | - | - | - | - | - | - | 35 | - | - |
| Zirconium | - | - | - | - | - | - | - | - | 10 | - |
| Steatite | - | - | - | - | - | - | - | - | - | 15 |
| Seger Cone Number | 10+ | 10 | 9 | 8 | 9+ | 9 | 8 | 7 | 9 | 7 |
| Watkin Recorder | 32+ | 32 | 31 | 30 | 31+ | 31 | 30 | 29 | 31 | 29 |
| Approximate Temperature of firing°C | 1310 | 1300 | 1280 | 1250 | 1290 | 1280 | 1250 | 1230 | 1280 | 1230 |

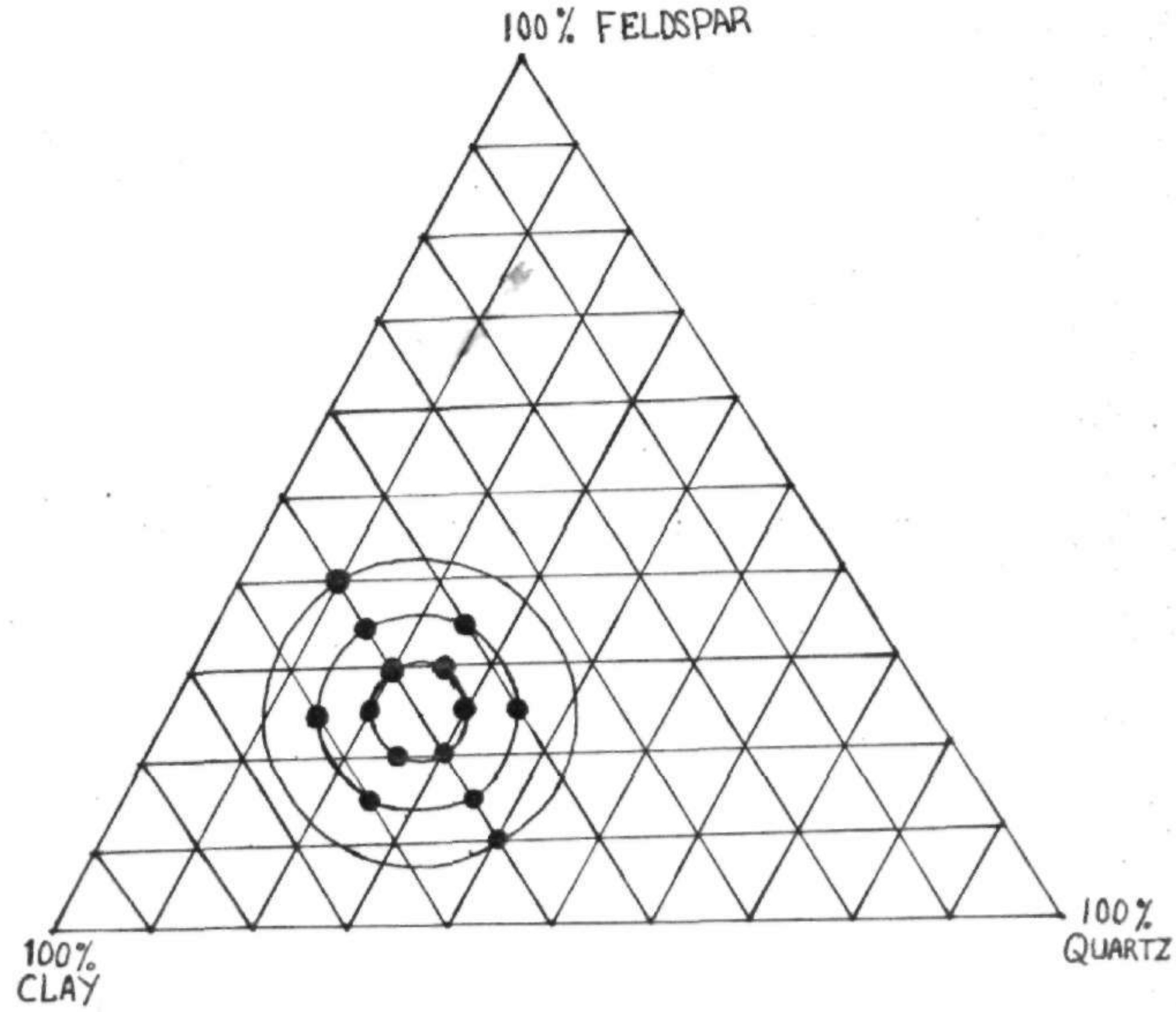


FIGURE 1: ELECTRICAL PORCELAIN COMPOSITIONS (USED BY BEICHAU (1924-5))

However, a balance is desired, which will lead to the most economical ware; easy to form but also fired without difficulty. Kingery (1960).

Bodies suitable for electrical porcelain manufacture must possess these properties:

i) Vitreousness over the required firing range.

ii) Adequate plasticity and workability for the forming processes involved.

iii) Adequate green strength

iv) Reasonable drying and firing shrinkages to enabale close tolerance to be maintained, and to minimise distortion and cracking.

v) Capability of taking glaze, this involves not only a suitable match of body and glaze thermal expansions, but also the absence of undesirable body-glaze reactions.

vi) Adequate firing range to cover the variations in firing obtained in large intermittent kilns, and to enable refiring for repairs of glaze faults, or the glaze-jointing of sections.

vii) Minimum pyroplastic deformation over its firing range

viii) Adequate Mechanical strength, and in some cases, good resistance to impact and thermal shock. All these properties are enhanced when the porcelain is matched with suitable glaze.

ix) Sufficiently low dielectric loss factor and adequate electrical break down strength.

It may seem odd to place the electrical properties last, but in practice this is not usually a critical factor, and insulator designs based on mechanical considerations, generally, have sufficient electrical strength. Johnson and Richardson(1975).

## 2.2 AUXILIARIES

Some additives are normally used in dry pressing to improve flowability, reduce abrasion, improve bonding strength, increase internal lubrication, and aid external or die-wall lubrication. Ideal binder or combination of additives is needed to achieve the aforementioned objectives. *Reed and Runk (1976). Some of the auxiliaries* together with their functions, given by Reed and Runk (1976), are presented in Table 3.

A number of materials are employed as binders or lubricants to assist in dry pressing. In the presence of sufficient clay in the body, other additions are not necessary. However, where insufficient clay is desired in the body, the selection and application of organic or inorganic binders can be classified as the second most critical factor in dry pressing processes. Whittermore(1978)

TABLE 3: Functions of Additives in Ceramic Batches

| Additive | Function |
|---|---|
| Binder | Green strength provision. |
| Lubricant | Provides mold release and interparticle sliding. |
| Plasticizer | Improves flexibility of binder films, allows plastic deformation of granules and gives rheological aids. |
| Chealating or Sequestering agent | Inactivates undesirable ions |
| Fungicide and bactericide | Stabilizes Ceramic mixes against degradation with aging. |

### 2.2.1 BINDERS

In has been, generally, found that better results are obtained by using water soluble binder (three percent) with high melting micro-crystalline waxes (three percent) than by employing only one type of binder. In addition binders should have low melting points. Singer and Singer (1971). Many binders are compatible with others, that is precipitation, gelatinization, or other detrimental effect does not occur as a result of combining two binders together. Onoda(1978)

### 2.2.2 PLASTICIZERS

The function of a plasticizer is to develop the adhesiveness of the binder in the absence of water. Singer and Singer(1971). Plasticizers are the additions that soften the binder in dry or near-dry state. They are lower - molecular-weight organic species that dissolve in the same liquid as the binder. Onoda (1978).

### 2.2.3 ANTISTICKING AGENTS (LUBRICANTS)

Lubricants have three functions according to Singer and Singer (1971).

These are:

i) Aiding flow of the particles of the body in the die while the forming pressure is applied.

ii) Preventing the body from sticking to the punches when the forming pressure is released.

iii) Providing the 'slip' necessary to prevent the pressed pieces from sticking to the walls and core pins when ejected.

Waxes can be combined with fats, fatty acid soaps as antisticking agents, but not with oils. Singer and Singer (1971). Best lubricants, in practice, are those with polar groups, and for oxide powders, oleic acid, stearic acid, zinc strearate, alcohols, and waxes are commonly added in minor amounts. Reed and Runk(1976).

2.2.3.1 INTERNAL LUBRICANTS

Internal lubricants are added to improve the flow properties of the powder mixture and also contribute some strength, but to a lesser degree compared with binders. Materials used include, Poly (ethylene) glycol and paraffin wax. A large amount can be added and is contained in the void volume of the particle mass. Whittermore(1978).

2.2.3.2 MOLD LUBRICANTS

Mold lubricants help in reducing the frictional effects at the mold walls. Lubricants such as colloidal graphite and stearic acid are employed. They may be wiped or sprayed on the mold wall or pumped through portings, Whittermore(1978).

2.3 SHAPING MIXTURE

The requirements that electrotechnical porcelain has to meet standards with respect to properties, such as mechanical strength, breakdown voltage, and dielectric losses, are particularly rigid. These requirements are only satisfied if the porcelain is densely sintered and has a homogeneous structure with a minimum number of pores, consequently, specially prepared mixtures and special production conditions different from those used for household porcelain are required for electrical porcelain. Budnikov(1964).

Dry pressing requires a powder that has uniform flow characteristics or a powder that exhibits good "Pourability". Generally, coarser particles tend to flow more uniformly than finer ones, and an ideal granular size distribution is known to be between 16 and 80 mesh. As little as 10% finer material can impede the free pouring characteristics of the powder. Reed and Runk (1976).

2.3.1 POWDER

A powder consists of individual crystallites bonded together to form a particle, which may be described as an aggregate, an agglomerate, or a granule. Consequently, the characteristics of both crystallite and particle are important. The characteristics of a powder, at any stage of processing, can be described by a large number of parameters, which are summarised in Table 4 Matkin(1973).

Ceramic powders can be prepared by comminution of bulk material, by chemical preparation, or in a few specialised cases, directly by vapour phase reactions.

TABLE 4: Important Powder Characterisation Parameters

| | Parameters |
|---|---|
| Crystallites | 1. Crystallographic form<br>2. Stoichiometry<br>3. Size<br>4. Size distribution<br>5. Shape<br>6. Impurity content and distribution (including adsorbed gases on the surface).<br>7. Activity<br>8. Reactivity of surface energy<br>9. Internal energy. |
| Particles | 1. Size and size distribution<br>2. Shape |
| Agglomerate aggregate and granule | 1. State of agglomeration<br>2. Pore structure<br>3. Impurity content and distribution<br>4. Powder "Stickness" or "glidability |

## 2.3.2 PROCESSING

The treatment of the mixture before molding has considerable effect on the porosity and other properties of the insulators. For instance, aging, that is, allowing a plastic mixture to stand for sometime, has a favourable effect on the molding behaviour, the green strength, and the properties of the final product. Deairing in special pug mills has, nowadays, successfully replaced aging. In electrical porcelain production, dearing of the mixture is very important because it substantially improves the performance of the insulators. Budnikov (1964). It is established that the characteristics of the initial powdered material, determined by the initial powder preparation, and by its subsequent treatment, strongly influence all stages of ceramic fabrication and, therefore, affect both the microstructure and the physical properties of the final ceramic ware. It has been pointed out that, powder processing is an important stage of fabricating ceramic from powder, but it has been found to be the most difficult to characterise and control. The major aim of powder processing is to obtain a reproducible powder with a set of known and required characteristics, in order to predict the behaviour of the material during the subsequent fabrication stages. Matkin (1973).

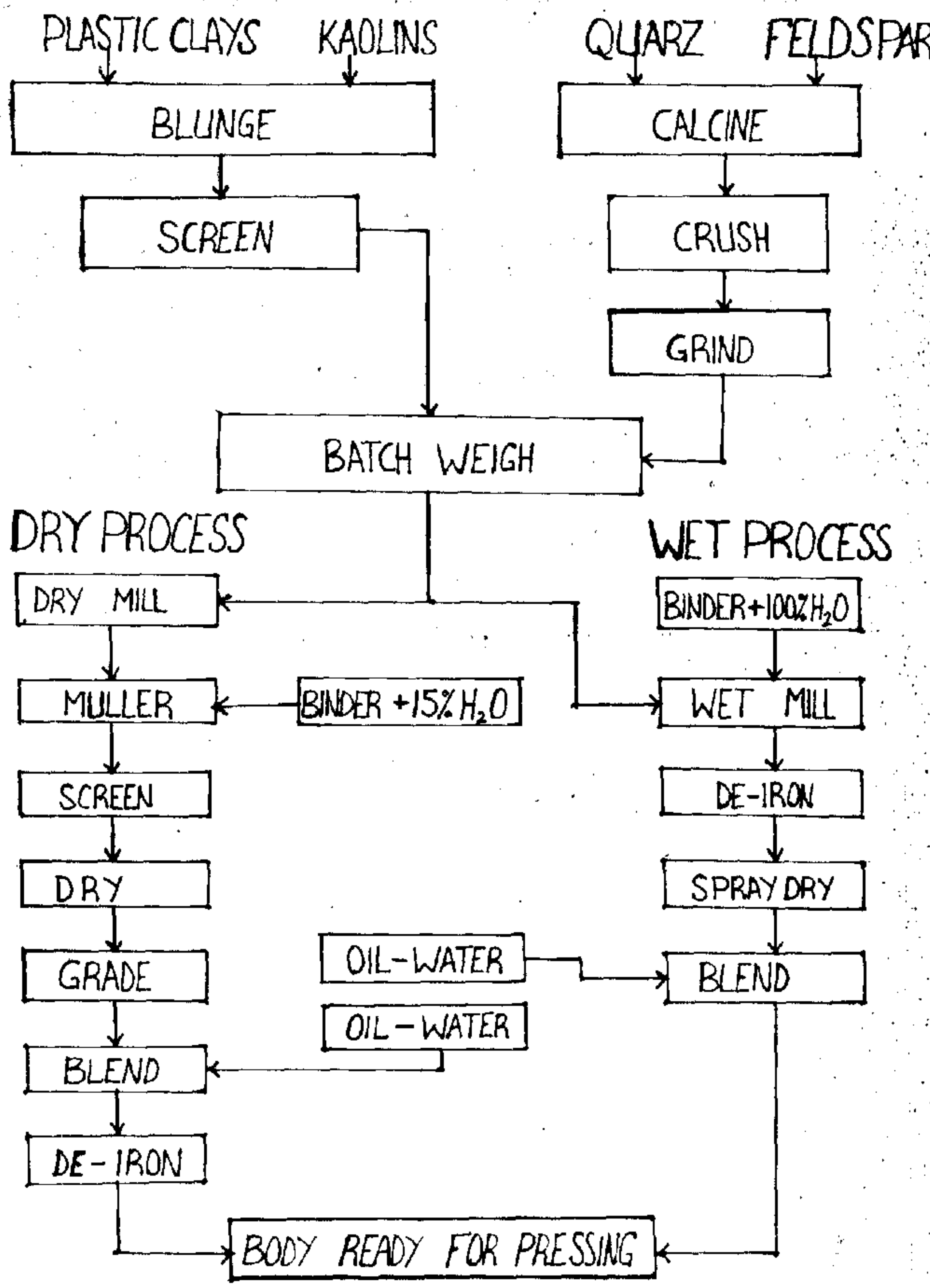


FIGURE 2: BODY PREPARATION FOR DRY PRESSING AND SIMILAR PROCESSES

For processes in which the body is formed by pressing in an essentially dry state, material may be prepared either by wet or dry process. The preparation of the body for shaping is dipicted in Figure 2. However, it is becoming increasingly common to use the wet process in which the materials are milled together with whatever binder or additives are required. The material is then spray dried in order to form small spherical granules which are free flowing and readily fill die cavities. Kingery (1960).

2.3.2.1 CALCINATION

Calcination is said to be the sufficient heating of a material to drive away water and also burn most of the organic impurities in the presence of air. Calcination temperature depends on the material to be treated. Apart from its primary function, it also creates some thermal stresses in the calcined material when it is cooled fast enough. This is being utilised to weaken the rocky materials so as to chip and crush them easily.

2.3.2.2 CRUSHING AND GRINDING

Crushing is the reduction of large lumps of materials to convenient sizes for secondary reduction. There is primary and secondary crushing. Maximum sizes of materials from primary crushing are 7.62 - 15.24 cm. In secondary crushing, sizes from primary crushers are reduced to sizes suitable for grinding or milling.

Grinding is usually done by means of ball mills or edge runners, and could be wet or dry. In wet grinding, the quality and fineness of the powder is governed by the weight of the stones and the length of the time allowed. Hill (1914-5). The variables known to have influence over the wet grinding in cylinder mills, according, to Creyke and Webb (1940-1), are:

i) Speed of rotation, diameter and length, nature and shape of the interior surface of the cylinder. Speed of rotation is fixed by equation (1)

N = 32/SQRT (R-r)----------------------------(1)

ii) Total weight, shape, and size grading of grinding Media.

iii) Weight of charge, and its ratio to the weight of grinding media, and also the ratio of the solid charge to water.

Cylinders were found to be more efficient in grinding than balls. Zeemann etal (1969). Grinding is a particle size reduction process by means of mechanical forces. Greskovich (1976). Ball milling, according to Matkin (1973), consists of three stages, namely:

i) Marked reductioin in the sizes of aggregate by light milling.

ii) Fracture of individual particles and formation of defects and changes within the particles during longer milling.

iii) Re-agglomeration at Prolonged periods of grinding.

Grinding is normally dependent on the viscosity of the medium up to about 20,000 mill revolution, and is then independent. Somasundaran (1978)

2.3.2.2.1 MILL CHARGING

Ball mills are generally filled with grinding media to, approximately, 50 percent of their total volume, while the powder charge is usually 25 percent of the total volume for dry milling and is 30 to 40 percent of the total volume for wet milling operations. A grinding aid, such as stearic acid or oleic acid, is usually added in small emounts (about one percent weight) to powders under going dry milling in order to enhance efficiency. Greskovich(1976).

2.3.2.2.2 GRINDING AIDS

Grinding aids, in vapour forms, such as ethylene glycol, propylene glycol, and butylene glycol were said to be used commercially, in Germany and Yugoslavia, to improve the efficiency of cement grinding. Amine acetates and diethylene glycol were reported to be in use, in Japan, as grinding aids in plant size reduction. Somasundaran(1978).

Some of the grinding aids currently in use are:

a) WATER

Grinding in water is more efficient than dry grinding. This favourable effect of water has been ascribed to a reversible reaction between unsatisfied surface bonds and water molecules. Cushioning effects due to the presence of fines, also, will be less during wet grinding, since fines tend to remain suspended in the water, than during dry grinding.

b) ORGANIC LIQUIDS

Grinding in organic liquid is noted to have more efficiency than in water. Samasundaran(1978) reported a 12-fold higher production of surface area for grinding in organic liquids, such as isoamyl alcohol compared to that in water. Carbon tetrachloride and Methylcyclohexane were reported to give higher grinding rates than Nitrogen. However, grinding efficiency was lower in the two organic liquids than in water but became the same when small amount of water were present in the organic liquids in dissolved form.

c) SUSPENSION AIDS

Some of the dispersing agents commonly used are sodium silicate and tetrasodium pyrophosphate. Greskovich(1976).

2.3.2.2.3 THEORY OF GRINDING

Von Rittinger (1867) has proposed that the energy spent in milling is proportional to the new surface formed. This relation is given by equation (2)

$$J = B(1/D_2 - 1/D_1) \text{---------------} \quad (2)$$

Kick(1885) applied the classical theory of elasticity and strength to grinding process. Kick stated that a critical stress is required for fracture and that the same energy is required to break up a large particle into an infinite number of smaller particles, a step at a time. The relation he gave is given by equation (3)

$$J = G \log(D_1/D_2) \text{ ---------------- } (3)$$

Bond(1952) stated that, the total work useful in particle breakage is essentially inversely proportional to the square root of the diameter of the product particles. Bond's mathematical expression, given by equation (4), is actually empirical because it was formulated on laboratory tests.

$$J = F \text{ SQRT } (1/D_2 - 1/D_1) \text{----------} (4)$$

The above "laws" are employed qualitatively because of the many oversimplificatioins and assumptions used to derive them. Rottinger's law was said to be often applicable, qualitatively, to the reduction of coarse particles, whereas Kick's hypothesis was said to be applicable to fine grinding of particles, as revealed by empirical observations. However, there are several objections to the use of these laws.

Firstly, there is usually a big discrepancy between the energy spent in milling and the surface energy of the newly formed surfaces. Theoretical grinding effciencies are usually low, of the order of 1%, with most kinetic energy converted into heat.

Secondly, it is doubtful that large particles have the same critical stress for fracture as small particles.

Thirdly, it is obviously very difficult to disintegrate large uniform particles into smaller particles of uniform size. This is implied in these postulated hypothesis of milling since the effect of particle size distribution in the feed or product material are not directly taken into account.

Several attempts have been made to improve these theories. However, the development of an edequately unified theory of milling from first principles appeals to be extremely difficult.

There are a variety of complex comminution events occuring that depend greatly on the nature of the material and the type of milling device employed. It is most reasonable then, to make use of empirical observations and relations which, if carefully interpreted in the light of modern theories of comminution can give a better understanding of the milling process and perhaps lead to a rational theoretical treatment. Greskovich(1976).

The fracture of particle involves the propagation of cracks that are present or initiated in the particle.
The stress required for fracture is given by Griffith relationship. Samasundaran(1978)

S = SQRT (2E Y/L) --------------------(5)

For brittle materials, $10^3 < V < 10^4$ erg/sq.cm. However, V is much greater than $10^4$ erg/sq.cm for plastic deformation. When a particle is repeatedly fractured, each new fragment tends to be stronger. This happens because the larger cracks in the original particle propagate first, leaving the finer cracks in the new particles and therefore, the probability of finding a flaw of a given minimum fracture stress decreases. As fragmentation continues, eventually the fracture stress required may increase to the extent that some plastic deformation is possible. With plastic deformation occuring, the particles can not be ground further, consequently, a limit of fineness in grinding exists. The limit, he said, is reported to be 1UM for quartz, and 3 to 5UM for limestone. Fracture stress was reported to have a gradual increase intially when the rate of stress application is increased from slow compressions to fast compressions. Fracture stress further increase with low-velocity impact, but then decreases when the impact velocity is further increased.

## 2.3.3. BLENDING

*Having been ground proportioned accordingly,* the materials can either be tempered in form of a liquid suspension or in the dry state. Both methods are said to have their advantages and disadvantages.

When a suspension is mixed, the mixture attains homogeneity more quickly, but more often than not, on account of large difference in particle size, and sometimes in the specific gravity of the particles, separation into *layers occurs. This results in separating the non* plastic materials and their accumulation at the bottom, while the top layers remain plastic. Hence, it is essential to keep stirring the liquid mixture in the tanks.

Making a mixture from dry powders is said to require more intensive and prolonged stirring than making a suspension. The manner of mixing determines the method by which the mixture is brought to the desired working moisture content. For dry preblending of the components in powder form, small quantities of water is usually added. This causes the formation of lumps in the mixture, and necessitates the use of a tempering Mill to break the bond. The water is added to minimise the entrapped air because the finely porous structure of a powder mixture obtained by preliminary dry mixing prevents air from being removed from the powder during pressing, and this promotes the formation of layer cracks ("overpressing"). Budnikov(1964).

The main essence of blending is to ensure uniform distribution of the additives required to aid the fabrication process of a ceramic ware from a ceramic powder. Matkin (1973).

## 2.3.4 GRANULATION

Granulation techniques, to improve flowability and packing efficiency of powders, must be applied in order to facilitate pressing of fine powders because of their poor flow properties. Odemans(1965), Reed and Runk (1976).

Binders are employed in most granulation techniques. A number of parameters which include granule size and shape, and inter-granular cohesion and friction are used to determine the flow properties and packing efficiency of the granulated powder. Strength of the powder granules is also important because they have to be strong enough to withstand handling yet, if the granule structure is not broken under the compaction pressure during cold forming, then non-uniform microstructure and low green densities result. Since the flow properties and packing efficiency are markedly influence by granule size distribution, the classification of granule size is important in ceramic processing. The various classification techniques include screening and air classification. Matkin(1973).

The following granulation techniques are used according to Reed and Runk (1976), Whittermore(1978).

i) Compaction followed by crushing and screening

ii) Ball-milling and screening

iii) Spray drying.

Binders could be used for granulation process. In most cases, the three techniques enumerated are employed for small, intermediate, and large quantities of powders respectively. Spray drying permits close automatic control over the final moisture, binder, and lubricant content to within less than ± 0.5%. It permits more precise control over the powder characteristics by having all the process parameters under high control. This ensures consistently good flow properties. Reed and Runk (1976). Granulation, usually in form of compaction, is often employed to alleviate problems, such as low bulk densities, and high compaction ratios (ratio of compact density to powder bulk density), associated with fine powders. Precompaction gives rise to a large reduction in the compaction ratio and a more uniform final product. However, although high granule density will reduce compaction ratio, coarse weak bodies may result, with greater density variations within the compact. Whittermore (1978).

## 2.4 DRY PRESSING

Dry pressed electrical porcelain served well for low frequency, low voltage insulation although, wet processed electrical porcelains have somewhat finer microstructure which results in improvement of properties, particularly in dielectric strength.

The rising demands on the size tolerance of ceramic products used in electronic and nuclear application is the factor which brought the importance of shaping ceramic articles by pressure application into picture. However, even with drypressing, it is difficult to fulfill this demand, though easier than wet methods of forming, because of the linear firing shrinkage which ranges from 10% to 30%. This shrinkage causes discrepancies between the compact and fired product in size as well as in shape. Oudemans (1965). The final product precision depends on many factors, including powder mixing and sintering control. Whittermore(1978). Dry pressing is a process of shaping granular ceramic bodies in dies by application of pressure. Singer and Singer(1971). Dry pressing is simultaneous uniaxial compaction and shaping of a granular powder with small amount of water and/or binders during confined compression in a die. Reed and Runk(1976). The main objective of pressing is to turn a loose, unbounded powder mixture into a firm product of maximum uniformity and density, of definite shape, size and hollowness. Budnikov(1964). Reduced cost of production, lessened cost of installing expensive dryers or dry floors, more uniformity and precision in size, improved refractoriness for some clays, better thermal shock resistance of large refractory shapes, and poorer heat conductivity of dry pressed ware are some of the advantages claimed in making large shapes by dry pressing. Flood (1922). The greater the pressure used during molding, the smaller the firing and total shrinkages. It was observed that the total shrinkage of pressed ware are much lower than those of cast and molded bodies. Rieke and Keeding (1930).

The advantages of pressing are the rapid molding rates (up to 5000 pieces per minute in rotary presses) and precision (molded tolerance on the order of 0.1mm). Reed and Runk (1976) and Whittermore (1978).

2.4.1 PRESSING

The shape, size, and hollowness of the compact are determined by the structure, shape, and size of the press dies. Strong green ware (compact) is obtained by densifying the powder; the densification brings the particles close together, increase their contact area and thereby produces considerable mechanical adhesion. Due to the internal friction which accompanies powder compression, owing to its low moisture content and poor slippage of the particles with respect to one another, considerable pressure (100-150kg per sq.cm) or more is required to make the materials strong. Budnikov(1964). Porosity diminishes as the pressure is increased from 25KN/Sq.m to 200KN/Sq.m. Foster and Emery(1915-6). The greater the strength and therefore, the density of the green ware, the higher the strength of the fired parts, all other factors being equal. The volume of air in the loose powder may attain 50% of the volume of the mixture. This air fills the spaces between the grains and is patially adsorbed on the surface of the particles weakenning their cohesion. For dense compact to result, particular attention must be given to the grain composition. Densification of powdered mixture is accompanied by expulsion of the air, which escapes through the gaps between the die and the mold. The air is partially forced inside the compact and, it finds its way between the grains in the pressed state, thereby lowering the cohesion and increasing the amount of elastic deformation occuring during pressing, and also the elastic after effet. However, larger amount of dust and clay with a greater moisture content makes it difficult for the air to espace, and may cause lamination of the compact (formation of cracks parallel to the pressing plane). Application of pressure for a longer time and in a staggered fashion makes it easier for the air to escape. Moisture enveloping the particles of the mixture helps the clay particles to adhere to one another and reduces the internal friction. The higher the moisture content of the mixture, the lesser pressure required. Budnikov (1964).

2.4.1.1 PROCESS

Densification requires an increase in pressure and is accompanied by a decrease in volume (Shrinkage) of the mixture. The shrinkage depends on the properties of the mixture and the extent of the pressure, and is evaluated by the compression coefficient ($K_c$), that is, the ratio of the height of the powder when poured into the mold ($h_i$) to the height of the compact ($h_2$). The value of $K_c$ is reported to be usually within the range 1.6 - 2.0. As the pressure builds up, the shrinkage rate of the mixture is uneven. Initially, there is more shrinkage principally due to wedging and mixing of the particles in the direction of application of the force. On further pressure increase, less shrinkage together with plastic and brittle deformation of the particles result. Further pressure increase makes plastic deformation more and more important, and increases to such an extent that it results in a waste of energy and demixing of the compact (over pressing) and this is further aggravated by the air pressed into the compact. Relaxation of the load from the compact clamped by the sides of the mold causes expansion due to elastic deformation, but only in the direction of the receding die. This is why the lamination cracks lie parallel to the plane of pressure application. While higher pressure than necessary causes overpressing, reduced pressure (under-pressing) produces flimsy compact and fragile fired material. Budnikov (1964). The moisture content at which maximum consolidation occurs (critical moisture content) decreases as the forming pressure is increased. The maximum amount of consolidation produced at the critical moisture content increases with increase in the forming pressure. For a particular pressure, the critical moisture content is higher for fast than for slow rates of loading. It was observed that, at similar pressures, the critical moisture content for the fine-grained clay is higher than for the coarse grained clay. The critical moisture range is narrower for the higher pressures and for the coarser grained material. Noble etal(1958). Prolonged or repeated effect of a load makes it possible to reduce considerably the elastic forces created during pressing and to change elastic to residual deformation. However, high productivity required from presses limit the duration of the pressure to 2-3 seconds, hence multistamp pressing is employed in practice. Several stages helps to increase the density of the compact and also to decrease the deformation. Two stage pressing, with ratio of primary to secondary pressure usually within the range of 1:3 to 1:4, is usual. Budnikov (1964). Also bumping has a marked effect on the porosity of the compact. Foster and Emery (1915-6).

During pressing, some of the force transmitted by the die is spent in overcoming friction inside the mixture itself and against the walls of the mold. Hence layers further away from the die have reduced density. Consequently, the thicker the layer of the mixture, the greater the differential in the density of the compact - the so-called nonequidensity. This is why articles are pressed in the direction of their smallest dimension for bilateral pressing, which is equivalent to halving the thickness of the layer. Increase in moisture content of the mixture decreases nonequidensity tendency. The quality of the compact, therefore, depends not only on the properties and preparation mixture, but also on the pressing conditions, including the final pressure and the load ratio, in step-by-step- pressing. Budnikov (1964).

### 2.4.2 <u>CLASSIFICATION</u>

There are two extreme forms of dry pressing namely, dust pressing and true dry pressing. Singer and Singer (1971). Pressing processes include dry pressing, which uses up to 4% water, dust pressing or semi-dry pressing, which utilises up to 20% water, and isostatic pressing, which uses less than 2% water. Reed and Runk (1976).

#### 2.4.2.1..<u>SEMI-DRY PRESSING</u>

Semi-dry pressing is the end point of the clay-water combinations requiring more pressure, the lower the water content.

These bodies contain clay and have about 10-15% moisture, although Reed and Runk(1976) have reported the moisture content required for semi-dry pressing to have extended up to 20%. Under sufficient pressure plastic flow into the die cavities occurs. In fact, some of these bodies, although appearing granular, will form a compact when pressed in the hand. Semi pressed pieces are rather fragile.

Vitrified bodies, shaped in this way, do not attain the density of a truly dry pressed piece or one made by a wet process. Therefore, electrical porcelain fabricated in this way can only be for low-frequency, low-voltage applications. However the universal usage of dust pressing is for wall tile production. Singer and Singer(1971).

2.4.2.2 TRUE DRY PRESSING

Although true dry pressing may contain up to 4% water, it makes no use of the natural plasticity that could be developed in any clay by the water that is present. Dry pressed ware that will become hard on firing can be produced from any raw materials, whether they possess any pasticity or not, by suitable additions of lubricants and binders. Dry pressed wares are known to have little or no drying shrinkage and so cut out drying time. They can be made with high dimensional accuracy, completely vitrified, with high dielectric strength, and with special precautions, vacuum-tight ware can be produced. In this case, no flow of the body will occur under pressure, so the granules must be correctly distributed in the die before pressing starts. Means of over-coming the flow problems associated with the production of suitable electrical porcelain include proper design of shapes with regard to the method by which they are made, avoiding unnecessary sharp corners, excrescenses, weakness etc, and addition of small quantities of binders, lubricants and anti-sticking agents and plasticizers. Singer and sunger (1971).

2.4.3 COMPACTION BEHAVIOUR

During compaction, the dispersed powder becomes a cohesive unit, the porosity decreases, single crystal or aggregated particles fracture, and the number of interparticle contacts and degree of particle interlocking increase.

The force applied by the moving punch is transmitted through the compact via interaction forces at particle contacts. An unbalanced system of normal and shear stresses at contacts can lead to particle deformation and sliding and multiple particle rearrangements. The reaction forces of neighbouring particles in contact restrict sliding and translation. It was said that, greater displacement of particles in line with the applied pressure are expected, but lateral displacements are also possible, especially initally, due to the random blocking of neighbouring particles and the random availability of nearby pores. As the force applied, increases, contact stresses increase and therewith both normal and frictional forces are increased. The contact stresses can cause elastic deformation and perhaps plastic flow or fracture at local contacts for relatively low applied force because the sum of the contact area is only a small fraction (as little as $10^{-4}$) of the total cross section. The increase in the number of contacts per unit volume and a greater deformation at contacts would tend to diminish the stress multiplication at contact points. Reed and Runk (1976)

2.4.4. <u>STAGES OF PRESSING</u>

Knowledge of how compaction proceeds within, the die, and what forces operate on the individual powder particles and determine their movements individually or in small assemblages is too scanty. This is attributed to the complexities of the experimental problems which tied down research publications on compaction of dry ceramic powders to only a few. Oudemans(1965).

Reed and Runk (1976) conveniently considered compaction behaviour in four stages distinguished in terms of a range of pressure and predominant mode of densification. These stages are:

i) Particle sliding and rearrangement without fracture

This is the initial compaction characterised by irreversible flow at applied pressures of less than 68.95 N/Sq.cm for fine powders containing weakly agglomerated particles. Punch travel, within this stage, ranges between 0.25 - 0.50 of the total, depending on the porosity of the feed material. Powder porosities on filling range typically between 0.80 - 0.95 for bulky powder feed and between 0.60 - 0.75 for spray dried feed. Gas escaping between the die and the puch causes "flashing" of the powder there, and compression of air in the pore phase can occur if the strain rate is high. The microstructure of the granulated (Spray dried) powders is quite different from that of the bulky powders. The particle packing in the granules and the cohesive strength of the granules can be quite important in controlling the pore structure and the degree of preferred orientation of particles at this stage in the compact.

ii) Onset of local deformation and fracturing of particles at points of contact

As the applied pressure continued to increase, more intimate particle configurations are formed and can only collapse if local fractures occur at points of concentrated stress. In this state, the grain size/particle size, intergranular strength, particle shape, and the modulus of elasticity (hardness) of the particle phase are quite important. Densification, within this stage, exhibits a linear relationship betwen relative density and log pressure followed by another linear dependence of density on log pressure but of greater slope. The critical stress corresponding to the change in slope is interpreted as a qualitative index of the fracture stress of the particle leading to continued sliding and rearrangement. Compaction slopes for applied pressures, exceeding the critical pressure are similar for specimens of the same material phase with different powder character, whereas the initial compaction density is very sensitive to the powder character. Several compaction studies indicated that the relative densities at an equivalent applied pressure are lower for powders containing particles with a higher Young's modulus of elasticity and Moh's hardness.

iii) <u>Elastic Compression</u>

In the pressure range between 6.9-20.7KN/Sq.cm, the rate of particle fracturing and rearrangement drops significantly and elastic strain energy density in the compact rises at an increasing rate. The use of pressures in excess of some practical limit of approximiately 20.7KN/sq.cm for typical ceramics greatly maximises the difficulty in realising satisfactory compact and increases die wear at an accelerated rate. This is because differential elastic moduli for the powder and admixed phases and elastic anisotropy of "brittle" powder particles will cause inhomogeneous strain gradients with increasing applied pressure and these elastic strains are largely reversible and can lead to complications in controlling compact defects.

iv) Ejection

On ejecting the compact from the die, residual elastic strains are relieved (poisson effect) and the dimensions of the compact increase by as much as 0.5%. Pore pressures are also relieved on ejection, and entrapped gases must be vented slowly if the permeability of the compact is low. Lubricants can effectively drop ejection pressures from several thousand to several hundred N/Sq.cm. It was found that relatively small stresses can cause fracture of the compact on ejection because it is relatively weak in tension.

Where the stages of ceramic powder compaction are die filling, particle rearragement, and particle fracture, the mechanisms are considered to be particle sliding, elastic deformation, and fragmentation. However, it was noted that description of compaction can be difficult because two stages may be occuring simultaneously and mechanisms predominating during this stage cannot be clearly defined. Whittermore (1978).

2.4.5 COMPACTION MODELS

Seeling suggested that compaction behaviour may be described by expressions presented in equations 6 - 8 Cooper and Eaton (1962).

$$0/0_{.} = Exp\ (-P/R')\text{------------------}(6)$$

$$= S'P^2\text{------------------------}(7)$$

$$= T\ P^{1/3}\text{---------------------}(8)$$

These expressions were found to be only meaningful physically, if the compaction is achieved by a single process, and there were several published sets of data that do not fit them. It was concluded that the behaviour of the four ceramic powders, used, depends markedly on their hardness. Work on model for compaction of ceramic powders led to the conclusion that the coulomb yield criterion may be used to compute load and stress distribution in the compaction of ceramic powders. Schwartz and Weintein (1965) Cooper and Eaton have developed a formula that expresses compaction as the sum of two distinct effects. Each effect corresponds to a particular compaction mechanism, which is allowed to occur according to a statistical probability dependent on the applied compaction pressure only. Oudemans (1965).

$$V_o - V/V_o - V_{oo} = V^* \text{-----------}(9)$$

$$V^* = a_{ps}Exp\ (-X_{ps}/P) + a_{Hf}Exp(-X_{Hf}/P)\text{----}(10)$$

However, $a_{ps}$, the constants associated with particles slippage, differ considerably at lower compaction pressures.

Analytical equation of state would be desirable for basic understanding of powder compaction. However, information which includes flow properties of powders under stress, stress or pressure distribution within compact, particle - to - particle stress distribution, and the stress distribution of ceramic particles would be required. Due to the unsolved analytical problems, several empirical equations have been proposed. These empirical correlations have value in understanding compaction. These equations, include previous researches carried out to show the pressure - volume relationship. Assuming that incremental pressure is proportional to incremental energy expended, Balshin developed the relation shown by equation------(11).

$$\text{Log } P = V'L + C' \quad \text{---------------------(11)}$$

The pressing modulus, L, is a function of the initial material properties but was observed to vary during pressing.

An equation developed by Heckel presented as equation (12), was proposed to govern the compaction of metals.

$$\text{In } (1/(1-SG) = K'P + A \quad \text{--------------------(12)}$$

$$A = \text{In } [\ (1/1-SG_o) + B'] \text{-----------------(13)}$$

Equation (12) describes three compaction stages: densificatioin by die filling, In $(1/1-SG_o)$: densification by particle rearrangement, B′; and densification by particle deformation. The foregoing expression was found to be valid for fused pure Magnesia but not for alumina, mullite and glass. Since fused pure Magnesia has some ductility, the expression, he said, appears to be valid only for ductile materials.

KawaKita and Ludde developed an equation relating degree of volume reduction, C′, to the applied pressure P.

$$C^* = a'bP/1+bP \text{ ---------------------(14)}$$

$$P/C^* = 1/a'b + P/a' \text{--------------------(15)}$$

$$C^* = (V_o - V)/ V_o \text{-----------------(16)}$$

This relation was found to be valid for soft, fluffy, and pharmaceutical powders and also stainless steel and copper oxide powders except for deviation at the lower pressures. KowaKita's equation has been applied to tapping compaction and vibratory compaction, replacing pressure with tapping number and vibrating time respectively. Whittermore(1978).

## 2.5 FIRING

During firing, at about 500°C, China Clay gives up its water content, and breaks up molecularly into silicic acid and alumina at about 900°C. Portion of the free silicic acid unites again with alumina, causing the development of heat and forming an amorphous silicate with a composition $Al_2O_3S_iO_2$, an amorphous sillimanite. At about 1100-1200°C, the feldspar in the body melts to form a very viscous glass and as the temperature rises further the glass acts on other components, namely, silicic acid, alumina and sillimanite. Consequently, the components must possess the right form and the right grain size, so that the particles would have their molecular surface for the attack of the feldspar melt as high as possible, but on the other hand, the material must be able to withstand both a high and lengthy firing. It was pointed that the higher the firing temperature rises and the longer it can be maintained, the better the quality of the body , since it is only in a body of this kind can the feldspar work intensively on all the individual components of the mass. The feldspar melt dissolves the molecular residue of silicic acid left from the breaking up of the China Clay and then attacks the free silica contained in the material. It more or less dissolves it completely, depending on the size of the surface of the silica, and it changes the impervious silicate into desmotrope crystalline form by mass action. Reichau (1924-5).

The temperature at which hard porcelain must be fired to give good results is not so rigorously defined as is generally believed. Bodin and Gaillard (1929-30). During firing, the behaviour of ceramic bodies is largely determined by the mineralogical composition rather than the percent chemical composition, which is merely the sum of the various mineralogical compositions. Ceramic firing includes three essential factors. These are the temperature, the duration of firing, and the chemical nature of the Kiln atmosphere. Bremond (1929-30). Previous research with a porcelain body showed that while the dielectric strength increase from cone 10 to cone 15, Porosity remained essentially zero over the range. Higher temperatures were noted for their considerable reduction in the mechanical strength. Also repeated firing has been found to increase mullite formation but could also decrease the MOR and the linear thermal expansion. Harris (1938-9). The final temperature to which a body is to be fired must be from 150 to 200°C below the melting point of the body, so that the temperature can be maintain for a sufficient length of time. Reichau (1924-5). Firing at 1280°C gave results that were in general, as good as 1320°C and in some cases even better with regards to whiteness and translucence. He noted that firing at 1410°C gave maximum whiteness. Berkman (1953). Electric porcelains are fired in periodic furnace or non periodic tunnel kilns.

The conditions and duration of firing are determined by whether or not high-grade porcelain is required, as well as by the size of the insulators fired and the volume of the compartment or section of the tunnel. Bundnikov (1964). Most deposits of clay and china clay contain iron compounds in greater or smaller proportions, which may give the fired material a more or less distinct colouring. Firing is controlled, consequently, so that iron oxides in the body of a porcelain artware or tableware are reduced to ferrosilicates, the colour of which is near-white with only a faint tinge of blue.

Firing porcelain in a reducing (bloody smoke) atmosphere, unlike oxidising atmosphere, solves the problem of ware blistering caused probably by ferric oxide dissociation, and also gives the ware a delightful bluish white tint instead of the greyish yellowish off-white obtained by using oxidising atmosphere. Rado(1971). Bulk density and firing shrinkage, increase while the apparent porosity decreases as a ceramic ware is fired. On reaching zero apparent porosity, the vitrification point, further heating increases the bulk density to a maximum, and then decreases as the closed porosity increases because of bloating. Norris et al (1979). The temperature range for firing electrical porcelain is 1260 - 1310$^{o}$C. Ware may be fired before glazing or glazed then fired. During firing processes, either a viscous liquid or sufficient atomic mobility in the solid is developed to allow the forces of surface tension to consolidate the ware and reduce porosity. The shrinkage which occurs, in this process, is equivalent to the porosity decrease and varies from a few volume percent to 30-40 volume percent depending on the fabrication process and the ultimate density of the ware. Uneven shrinkage rate can lead to the development of stresses, warping and even cracks. Consequently, it is necessary to avoid excessive friction when setting ware for firing, control temperature uniformity, control the rate of firing, and in addition to that, control the time and temperature level. Particle size and size distribution of the raw materials in the ceramic shape before firing are of particular importance. Composition, firing time, viscosity of liquid formed during firing, desired properties of the product, and kiln atmosphere are the factors affecting firing.

2.5.1 DENSIFICATION

Densification of ceramic wares on firing proceeds mainly via two processes, namely, sintering and vitrification.

Densification before vitrification is governed by equation (17) according to Norris etal (1979).

$$DV/V_o = 9Vt/2ud \text{-------------------} (17)$$

Densification is not complete after vitrification because the surface tension forces exert a negative pressure, on the closed pores, equal to 2V/r. Where r is the pore radius. Attempts to define a rate of densification for this stage were made by some workers, however, the process is complicated by the presence of pores of different sizes and that of soluble gases in the pores. The net effect is that the pore volume continues to decrease at constant temperature after vitrification. However, when the temperature passes that necessary for vitrification, the closed porosity increases because of the effect of temperature on the residual gas in the pore, and the occurence of decomposition reactions such as the high temperature conversion of $Fe_2O_3$ to $Fe_3O_4$ with the release of oxygen. In a range curve firing, the competing processes of final-stage densification and bloating (tendencies for the closed porosity to increase) cause the closed porosity to attain a minimum at some temperature above the vitrification temperature.

2.5.1.1 SINTERING

Sintering is a term which connotes either individual or compound processes of bonding, densification, and/or recrystallization of powder compacts. Some researchers in 1955 defined sintering as the extension of the contact area between powder particles in the solid state, by the transport of material across or around pores, under appropriate conditions of time, temperature, pressure, and atmosphere. Sintering occurs due to the reduction in surface energy which is associated with decreasing surface area. According to the researchers, sintering occurs through three stages and several mechanisms, whose relative occurence is largely influenced by fabrication variables, governing the rate of the sintering.

2.5.1.2 VITRIFICATION

According to Harris (1938-9), vitrification is the progressive formation in a body during heating, of a liquid phase consisting of a molten glass, which is, in fact, the bond existing in all pottery bodies.

A completely vitreous body is not completely deficient of crystallization, but it is impermeable to even alcohol solution of a dye, like analine, when subjected to a pressure of 1.38KN/Sq.cm in its unglazed condition. The process of vitrification was influenced by many things, among which the time, temperature, and atmosphere of the firing, the composition of the body, and the fineness of the constituent materials may be cited.

A researcher noted that water vapour is one of the principal agent accelerating the process. Also, it is well known that a reducing atmosphere hastens the process, and water vapour is known to be a powerful reducing agent. Vitrification is reached at a temperature at least 50°C lower than in an oxidising atmosphere, where time of firing and body composition remain constant.

It was found that hard porcelains rich in Kaolin and lime, but with low feldspar content are not readily vitrified until about 1200°C, whereas the feldspathic porcelains begin to vitrify at about 1050 -1100°C. Bremond(1929)-30).

As the temperature rises, the viscosity of the feldspar silica glass falls and consequently, the wetting power of this glass in contact with the surrounding crystals is increased, resulting in more intimate contact and more rapid solutions. Harris(1938-9).

Vitrification is a time reaction, and the velocity of the reaction is accelerated by raising the temperature. The effects, contraction, transluency, and so on, produced during prolonged low temperature firing (easy firing) can be achieved in a shorter time by firing at higher temperature (hard firing). Experimental findings show that the vitrification of a body progresses more when heated to the higher temperature for a short time than when heated for a long time at the lower temperature under conditions where the work done on seger's cones is apparently the same. Every condition which favours the heterogeous reaction, in the constitutent mixture, will favour rapid vitrification. The finer the textures of a body, the more rapidly will the reaction and hence, the vitrification occurs. However, certain amount of irregularity in the sizes of the grains facilitates close packing which in turn, facilitates rapid vitrification. Overgrinding leads to rapid vitrification, and by so producing a body of unusually close texture leading to "dunting" as well as other defects. Contraction and porosity of a fired body may be used as a measure of the degree of vitrification, time being constant. Effect of varying fineness become more apparent if the firing temperature is reduced. Finer grained particles get through their work in the earliers of the firing much quickly than the larger grained particles. At higher temperature, the larger grained particles have more opportunity to make-up for their slower speed at the beginning of the reaction. Mellor (1990-10).

### 2.5.2 FIRING SCHEDULE

Bodin and Gaillard (1929-30) suggested the following schedule as an optimum satisfaction of the conditions of modern kilns. They divided the firing time into four periods. First period: Rapid temperature rise at about 85°C/hr up to 1000 - 1050°C in the hottest parts, with an oxidising atmosphere after baiting the fires. Second period. From 1000 - 1050°C to about 1100 - 1150°C in the hottest parts, slow temperature rise of about 9°C/hr, and strongly oxidising atmosphere of about 7 - 11% Oxygen. This period has a double object of equalising the temperatures as much as possible and of completely decarburising the body and glaze. Third period: From 1100 - 1150°C to 1300 - 1350°C, temperature rise should be about 22°C/hr. Fires are banked up at the beginning and constantly reducing atmosphere is maintained, with about 10-6% CO in the flue gases.

Fourth period: From 1325 to 1400°C, heating rate should be about 6°C/hr and the atmosphere neutral or slightly reducing.

In firing - schedule of hard porcelain, the initial conditions must be oxidising so as to burn off organic matter. They said at a temperature of about 1050 - 1100°C just before sealing of the body or glaze takes place, the atmosphere is adjusted to reducing, and ferric iron is reduced to ferrous state. Formation of ferrous iron contributes to the fluxing of the body, and sealing takes place at a lower temperature than would be possible with ferric present, so the body becomes impermeable at about 1200°C. At higher temperatures, the atmosphere can be allowed to become oxidising, which is, in fact, necessary to produce satisfactory glaze colour. Williams etal (1963)

### 2.5.3 SOAKING AND FIRING RANGE

Body and therefore, a porcelain also, is of better quality the longer it is able to withstand the maximum temperature of the kiln. Soaking means the maintainance of the highest possible temperature for some time, the length of which depends on the type of the body being fired, properties desired from the fired body, and of course, economics. Beech et al (1961) employed a firing rate of 40°C/hr and a soaking time of two hours to fire a body which normally vitrifies at about 1180 - 1200°C, and could be used for insulator, sanitary, and vitreous china. Reichau(1924-5).

According to Beech et al (1961), firing range is the temperature range over which the bulk density is within 0.025g/cc of the maximum, which is equivalent to the range over which apparent porosity is less than one percent the closed-pore system being discounted.

Properties for which each value can occur at two different temperatures can be plotted as a function of firing temperature, for otherwise identical firing schedules in terms of heating rates, cooling rates, and soaking times, and these plots or curves known as range curves could be used to obtain the optimum firing range. Norris et al (1979)

2.5.4 CRYSTAL FORMATION

High temperature seems to be necessary for proper development of crystals in feldspar, clay and quartz mixtures. At high firing temperature, the glaze reacts with the china clay, in hard porcelain body, and on cooling, a fringe of crystals grows in the zone between the body and the glaze. These crystals bear a close resemblance to the needles of sillimanite- an aluminum monosilicate $Al_2O_3.SiO_2$- but for the absence of cleavage (Secondary) lines, which was attributed to the smaller size of the crystals. Later, the crystals were identified as identical to sillimanite and it was also observed that both kyanite and andalusite (isomeric forms of sillimanite) appear to pass into sillimanite at about 1350°C Mellor (1905-6). Crystals formation in a ceramic body may be good or bad, dependiang upon the use of the material and upon the nature of the crystals. However, mullite formation, in electrical porcelain, is desirable because it increases the strength and resistance to sudden temperature changes but for translucent porcelain, it is undesirable since it reduces the translucency.

Amorphous mullite forms at temperatures as low as 1000°C but does not become crystal until fired to higher temperatures. Fluxes which lower the viscosity of the molten part of the body such as CaO, MgO or ZnO, facilitate the formation of crystals in the body. Likewise low viscosity glazes may develop cystals, whereas viscous glazes may hinder their growth. Rieke (1928).

The process of mullitization coincides with the second exothermal effect in the heating curve of Kaolinite. This, also concides with the chemical reaction of $Al_2O_3$and $S_iO_2$ to form mullite. Above 100$^oC$ (after the first exothermal effect) and up to 1250$^oC$, the $Al_2O_3$and $S_iO_2$ are in an active state which helps mullitization. Budnikov and Gevorkyan (1951).

2.5.5 COMPOSITIONAL EFFECT

For all bodies, depicted on Figure 1, except the two with 45 percent clay, the maximum temperature is at least as high as seger cones 14 and 16. Reichau (1924-5) High feldspar bodies start to vitrify about 1050$^oC$ and then vitrify very rapidly, becoming thoroughly vitrified at 1250$^oC$.

Johnson and Robinson (1975) observed that, lack of fine particles can seriously retard vitrification, requiring an increase of feldspar to give the same vitrification point as the standard body.

# CHAPTER THREE

## 3. MATERIALS AND EQUIPMENT

The list of the various materials and equipment employed in this research is presented below:

### 3.1 MATERIALS

a) Kaolin from Kankara, known as Kankara clay, of Katsina State.

b) Ground feldspar from OKene of Kogi state

c) Quartz, inform of Silica sand, from kurnar Danjibga of Dabatta in Kano State.

d) Binding Clay from Bida of Niger State.

e) Various type of clays from Kano, Goronyo of SoKoto State, Bomo of Kaduna State, Ikebedi of Kwara State, Cross River State, Kaoje of Sokoto State, Bukuru of Plateau State.

f) Sodium Silicate (1.5g/cm$^3$)

g) Hydraulic oil

h) Copper (ii) oxide

## 3.2. EQUIPMENT

a) Flat bottom flasks, Measuring Cylinders, Beakers, and pipettes of various capacities.

b) 2 Weighing Machines, of 1000g and 100Kg Capacities.

c) Pfefferkorn plasticity defermination set.

d) Brook-field synchro-lectric viscometer, Model RVT, Mfd by Brook-field Engineering laboratory Inc. Stoughton, Mas. USA.

e) Ball mill (Medium, and small size cylinders), Mfd by The pascal Engineering Company Ltd., Gatwick Road, Crawley, Sussex, England.

f) Pebble mill, type 2x2, Mill rate 30.4.61 Wm Boulton Ltd.

g) Stop clock and stop watch

h) Plaster of paris Molds for Cylinderical solid casting.

i) Gallenkamp Oven, Model OV-420, App. No. 7B-95800, England.

j) 2 Sieves of 500Um and 75Um sizes

k) 1 Vernier calipers

l) 1 Micro-Meter Screw Gauge (0-25mm)

m) Earthen Ware containers

n) Filter Cloth

p) Silica Troughs

q) Grinder (Electric), Type Sk1, retsch KG, 5657 HAAN W/Germany.

r) Steel (Hardened) press Mold (Die and punches) for pressing rectangular test specimens.

s) Hydraulic press, Model M SN.23505-208 Carver laboratory press, Mfd by Fred S. Carver Inc. Subsidiary of Sterling Inc., Hydraulic equipment, W142 N9050 Fountain BLVD Mono Monee Falls, WIS 53051 USA.

t) Heraeus Hanau Furnace, Type T1K/R 11/12, Tanuson P.O. Box 208, Zoetermeter, Holland.

u) Water Heater

v) Denison Strength Testing Machine, Model No.T.42.C.2, Load Capacity 44800Ib, Machine no.29601, Pat. No.571765 551722m Mfd by Saml. Denison and son Ltd, moor Road, Leeds 10, England.

# CHAPTER FOUR

## 4. EXPERIMENTAL DESIGN AND EXECUTION

The design and execution of this research work have been summarized in Figures 3 and 4.

A detailed description is given in the following Sections.

### 4.1 DETERMINATION OF INTRINSIC PHYSICAL PROPERTIES OF THE SUPPLIED CLAYS

Clay lumps, whose sample names were KanKara, Kano KRD, Goronyo MWF1, Bomo, IKebedi, Cross River, BuKuru, Bida and Kaoje clays, sampled from various parts of Nigeria, were supplied for the determination of their intrinsic physical properties and the subsequent selection of the clays to be used in the development of drypressed electrical porcelain bodies.

Each of the nine clays was processed as depicted in Figure 3, and except for BuKuru clay, whose specimens for green and fired properties determinations, and Ikebedi clay, whose specimens for fired properties determination, were damaged in the course of fabrication, the intrinsic physical properties of all the clays were determined.

Two kilograms of each of the clay lumps were crushed, in an electric grinder, to powder. It is the powders that were used for the subsequent processes of Specimen fabrication.

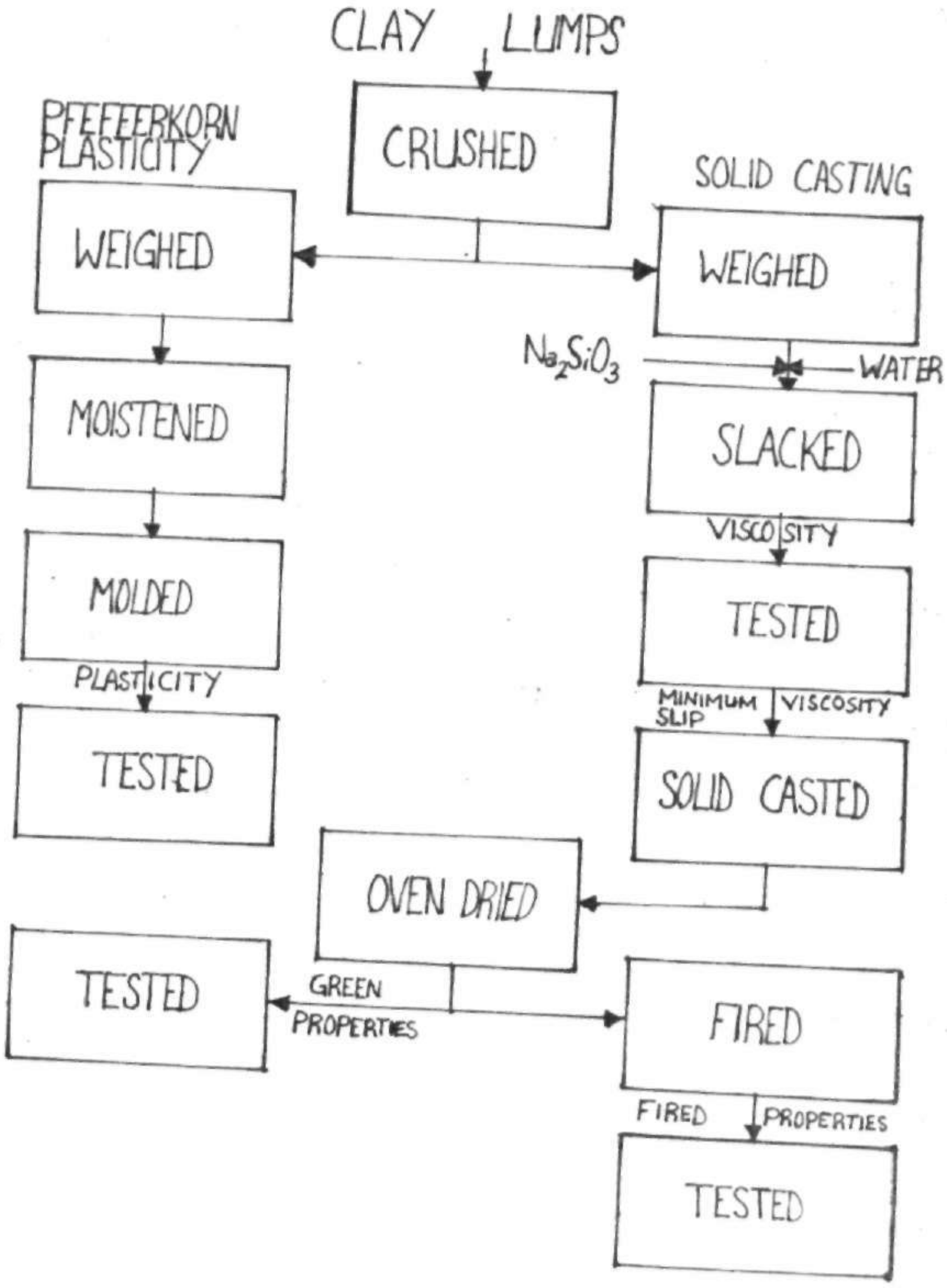


FIGURE 3: DETERMINATION OF INTRINSIC PROPERTIES OF THE SUPPLIED CLAYS

4.1.1 PFEFFERKORN PLASTICITY TEST

Test for determining the Pfefferkorn plasticity index was chosen as the basis of relative comparison, from plasticity view point.

Three hundred grams of each powdered clay were weighed and divided in to five batches of sixty grams. Known but different quantities of water were added to each batch to produce five clay samples of different moisture contents. These moistened clays were used, with the aid of cylinderical mold to fabricate five clay cylinders of different moisture contents.

The moist cylinders were deformed, one after the other, in Pfefferkorn apparatus by dropping its flat headed plunger, weighing seven hundred and eighty grams, on to the clay cylinder, from a fixed height. Extent of deformations of the cylinders were read from a graduated scale.

Plots of the five different deformations, referred to as Pfefferkorn heights were made against the respective moisture contents.

Moisture contents corresponding to a Pfefferkorn height of fifteen Milimeters were the Pfefferkorn Plasticity indices of the clays.

4.1.2. SOLID CASTING

The minimum viscosities of the clay slips were determined, and used to solid cast cylinderical test specimens. These were further processed, for the determinations of the physical properties, such as shrinkages, densities, firing weight losses, moduli of rupture, apparent porosities, and water absorptions, of the dried (green) and fired wares made from such clays.

a) Minimum Viscosity Determination

Six batches of 130g clay, all from the same clay sample, were weighed, known, but same amounts of water (solid to water ratio) were added to the batches. Deflocculant (Sodium Silicate) concentrations of 0.1 wt% to 0.6 wt% were added, one concentration to one batch, to the clay/water mixtures. Determinations of the correct solid to water ratios, for the clay samples, that gave viscosities measurable with the RVT Brook field viscometer, were done through trial and error, and with least deflocculant additions. The clay-water- deflocculant mixtures were ball milled, each for one hour, to slake the clay particles away from the, probably, associated silts. Charge to pebble ratio, weight basis, used for the milling was 1:1.

The prepared clay slips were poured one at a time, into a 75mls glass beaker, and using spindle number 3 of the Brooke field synchro-lectric viscometer, the viscosities of the slips, corresponding to different deflocculant concentrations, were determined. Other clays were treated in the same manner.

Plots of viscosities against the respective deflocculant concentrations were used to determine deflocculant concentrations corresponding to minimum voscosities of the slips.

b) Test Specimens Casting

The solid to water ratios, and deflocculant additions that gave minimum viscosities of the clay slips, in the rheological studies, were used to prepare slips for solid casting the test specimens.

Eight hundred grams of each clay were weighed, on to it the determined amounts of water and deflocculant were added. The mixture was milled, via ball mill, with a charge to pebble ratio of 1:1, weight basis, for one hour, to ensure homogeneity.

Slip, from each clay, was poured in to six plaster of paris casting molds, whose cylindericas cavities had been dusted with some fine powdery grog. The female and male parts of the casting molds were held tight together with rubber bands before the slip was poured into them. Reffilings were done as the poured slips were being dehydrated to form solid masses in the molds. Casting was completed with the solid mass filling the cylinderical cavities of the molds. Casts were left, in the molds, to dry further and shrink away from the walls of the casting molds, then removed and air dried.

Casts were then oven dried, at 110$^{\circ}$C, to constant weight.

Dimensions and weight of the dried (green) casts were determined with micro meter screw gauge, venier calipers, and weighing machine (maximum capacity 1000g).

Green properties were calculated from the determined dimensions, weights, and shear breaking loads. The procedure of determining the shear breaking loads on dried casts has been out lined in section 4.4.1.

Three dried casts, of each clay, were fired in an electric furnace, in accordance with the firing schedule given in Figure 5, to a peak temperature of 1290$^{o}$C within 23.75 hours, and left to cool to ambient temperature in a period of 8 hours.

Dimensions, dry fired weights, saturated weights, suspended weights, and fired shear breaking loads were determined, and subsequent calculations of fired properties were done.

Procedures of determining shear breaking loads, saturated and suspended weights have been outlined in section 4.4.3 and 4.4.2 respectively.

## 4.2 ELECTRICAL PORCELAIN: RAW MATERIAL SELECTION

Having done the preliminary studies on the nine supplied clays, based on binding abilities of the clays, Bomo, Bukuru, and Bida clays were chosen as possible binder clays to be added to the four preselected, from literature, possible porcelain making compositions for improvement of the green strength of the pressed specimens. Kankara clay, and Okene feldspar were chosen for the Kaolin and feldspar requirements of the classical electrical porcelain respectively, based on availability and purity. Silica sand of Kurnar Danjibga in Danbatta Local Government of Kano State was taken for the silica requirement on the basis of raw materials proximity and the relative low cost of sand compared to silica rock.

### 4.2.1 PORCELAIN TRIAL PRESSING

Trial pressings of the porcelain test specimens were done to find the pressure that could be employed for the pressing, and select one of the three preselected binding clays for use in pressing the porcelains.

Moisture content of 15% wt was chosen, from literature, for the pressing to enabale good compaction with much less pressure relative to that required with lower moisture content.

Out of the four preselected possible porcelain making compositions, the composition with the least clay content was used for the production of the porcelain trial pressing specimens, in which 10% out of the clay proportion was substituted with either Bomo, Bukuru, or Bida clay.

Two stage pressing was tried with primary to secondary pressure ratio of 1:3, and Secondary pressure of 4.5TNS, pressure was maintained for 10 seconds in each stage. Single stage pressing was tried with a peak pressure of 2TNS because of excessive loss of porcelain mixture, through the punch/die clearance, in the course of being pressed with 4.5TNS Peak pressure.

Specimens produced were compared on the basis of their green Moduli of rapture and fired colours. Based on this comparison, the binding clay for the porcelain bodies, to be developed, was chosen.

Two hundred and eighty grams of the least clay containing body (Body tagged "D") was dry milled (charge to ball ratio was 1:1, weight basis) for thirty minutes, to make the mixture homogeneous. Fourty two mililitres of water (fifteen percent weight basis) were added, hand mixed, and aged, for three hours, for moisture homogeneity, and pressed into the trial pressing porcelain test specimens.

## 4.3 DEVELOPMENT OF DRY PRESSED ELECTRICAL PORCELAIN BODIES

Processes done to develop the dry pressed porcelain bodies have been summarily depicted in Figure 4, description follows below.

Fifteen Kilograms of Kankara clay were crushed, dry sieved through 500Um mesh, and made into slurry. The slurry was thoroughly hand blunged, and wet sieved with 75Um mesh. The clay/water suspension was allowed to settle for seven days, supernatant liquid decanted, while the thickened clay paste was transferred to earthen ware containers for further dehydration. The paste was then finally filter pressed to remove more water. The resultant moist clay cake was air dried, followed by oven drying, at 110°C, to constant weight. The dried clay lumps were ground, in an electric grinder, to clay powder.

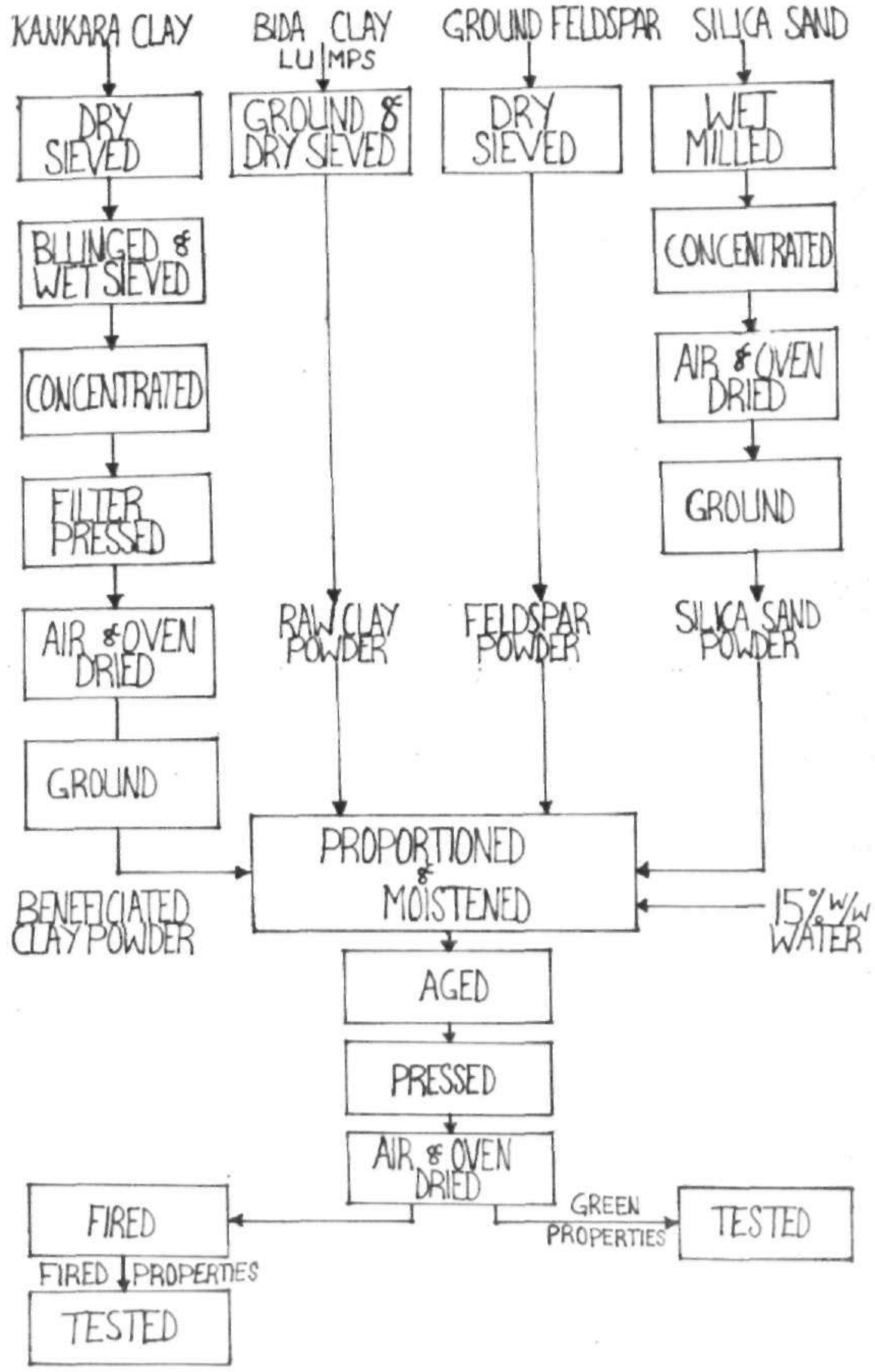


FIGURE 4: DEVELOPMENT OF DRY PRESSED ELECTRICAL PORCELAIN.

Five Kilograms of Bida clay lumps were crushed, ground, in an electric grinder, to powder, and dry sieved through 500Um mesh.

Five Kilograms of ground Okene feldspar were dry sieved through 500UM mesh.

Ten Kilograms of Danbatta silica sand were wet milled (Pebble to charge to water ratio was 1:1:1.25) in a pebble mill for eighteen hours. The suspension was allowed to settle, and the supernatant liquid decanted. The moist silica sand, obtained was air dried, followed by oven drying, at 110$^oC$, to constant weight. The dried silica sand was crushed back to powder in an electric grinder.

The composition of pebbles used for the wet milling of the silica sand as given in Table 5.

TABLE 5: PEBBLES COMPOSITION USED IN WET MILLING THE SILICA SAND

| Proportion of Pebbles Used (Weight Basis) | Average Diameter (Milimeter) |
|---|---|
| 1/3 | Greater than 80 |
| 1/3 | Less than 80 |
| 1/3 | Less than 50 |

The raw materials, Kankara and Bida clays, Okene feldspar, and Danbatta silica sand were proportioned to get four different, possible, porcelain compositions. The proportions of Bida clay were kept constant, and was always constituting 10% out of the clay requirements of the recipe. The four compositions used in this research work were as given in Table 6.

TABLE 6: BODY COMPOSITIONS USED

| Designation | Clay% wt | Feldspar% wt | Silica Sand% wt |
|---|---|---|---|
| A | 45 | 25 | 30 |
| B | 45 | 30 | 25 |
| C | 50 | 30 | 20 |
| D | 40 | 35 | 25 |

One Kilogram of each body was weighed dry ball milled (charge to ball ratio was 1:1) for two hours, to make the mix homogeneous. One hundred and fifty grams of water (15%wt) was then added, hand mixed, and aged for three hours. The moistened mixture was divided into batches of fifty grams each, then pressed into test specimens, batch after batch.

The mold (Die and Punches) was lubricated with used engine oil, positioned on the hydraulic press, with one of the punches protrunding below the die, but blocking the die cavity at the bottom. A batch of the moistened porcelain mix was poured into the die cavity, onto the upper surface of the lower punch. The upper punch was then placed in position on top of the moistened mix, and pressurised untill the hydraulic press read two metric tons. The pressure was maintained at its peak for fifteen seconds. The pressed porcelain was then ejected out of the die cavity. Eighteen specimens were pressed out of each of the four selected bodies.

The porcelains were air dried for eighteen hours, then oven dried, at 110°C, to constant weight, and cooled to ambient conditons.

Weights, and dimensions of the dried specimens were determined.

Four dried specimens of each of the four bodies were crushed with a three point loading for transverse shear breaking load determination as outlined in section 4.4.1. Also other green properties were calculated from the determined data. Sample calculations have been provided in Appendix E.

Four different peak firing temperatures, within the range of 1180 to 1300°C , were used to fire the pressed porcelains. Same firing schedule and time of total heat treatment were used all through. Oxidising atmosphere was maintained throughout the firing cycle. Three specimens from each body were fired to each of the four peak firing temperatures. The firing schedule has been shown on Figure 5.

The fired weights and dimensions were determined. Saturated and suspended weights of the specimens were determined as outlined in section 4.4.2. Transverse shear breaking loads, for fired specimens, were also determined as outlined in section 4.4.3. Fired properties were calculated from the determined data. Sample calculations have been included in Appendix E.

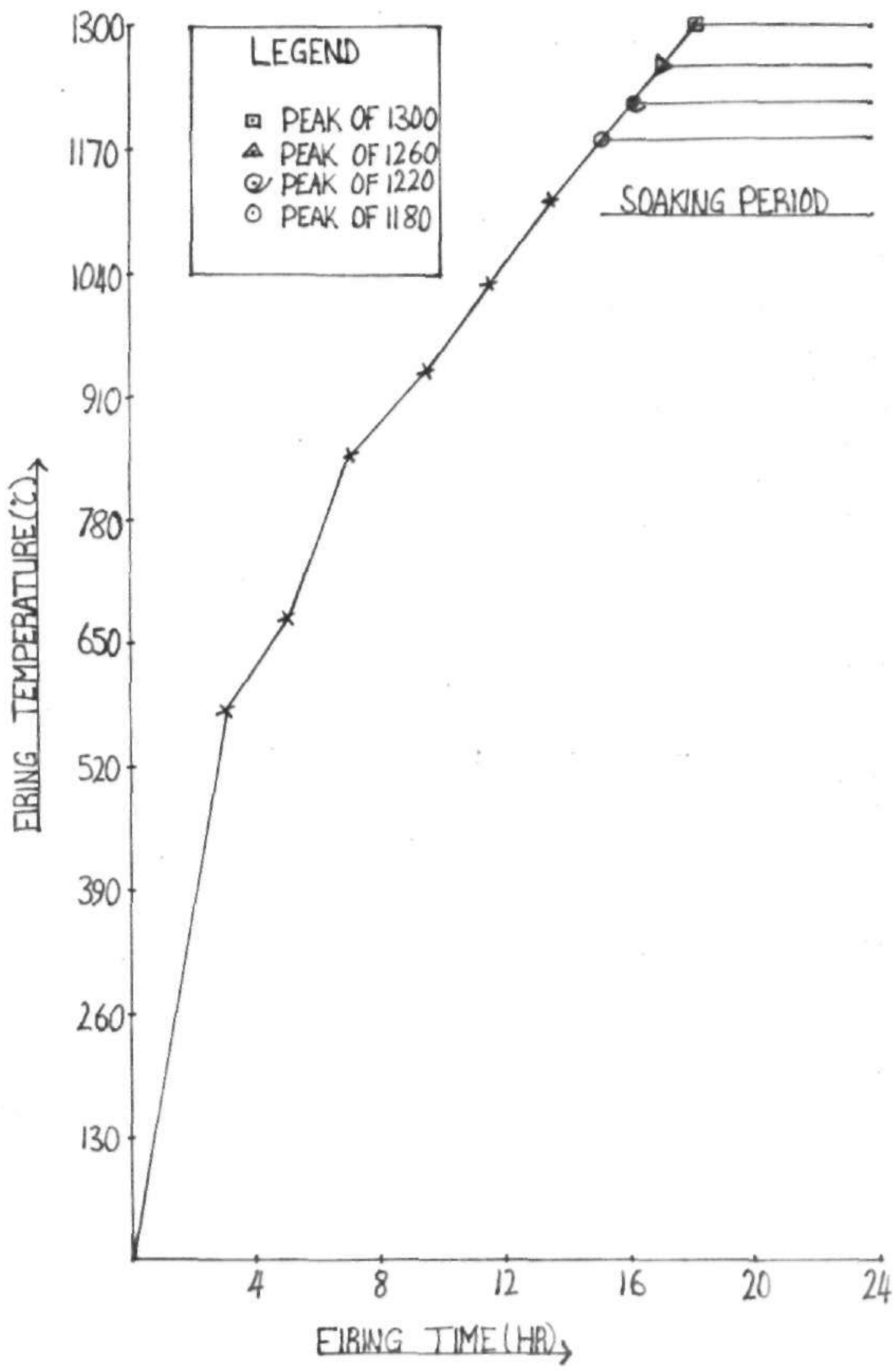


FIGURE 5: FIRING SCHEDULES USED FOR FIRING THE CLAY SOLID CASTS AND THE PRESSED ELECTRICAL PORCELAINS

## 4.4 REPEATED PROCEDURES

Procedures referred frequently in the description of the methodology of this work are outline below.

### 4.4.1 GREEN MODULUS OF RUPTURE

Three of the dried soild casts of each clay, and four of the dried pressed porcelains, from each of the bodies used, were subjected to shear breaking load using three point loading.

A specimen (rectangular or Cylinderical) was placed on two knife edged supports, 76.80mm apart. A rod was firmly tied to the mid-bottom of a plastic bucket, and placed onto the specimen, midway between the supports. Water was then poured into the bucket gradually, using a small diameter hose, and water flow rate of 75l/hr, till the specimen fractured. The bucket together with its contents and accessories were weighed and taken as maximum shear breaking load. The green modulus of rupture was calculated from this load, distance between supports (Span), and some dimensions of the specimen which depends on its geometry.

4.4.2 SATURATED AND SUSPENDED WEIGHTS

Preweighed fired specimens were boiled for two hours, and left in the water for eighteen hours. Specimens were then cleaned with a piece of saturated, with water, cloth and weighed. These weights were the saturated weights.

The Specimen were suspended in water, as shown in Figure 6, and the apparent weights found were the suspended weights.

Apparent porosity and water absorption were calculated from these data.

4.4.3 FIRED MODULUS OF RUPTURE

Three point loading was used to determined the maximum shear breaking load resistance of the fired specimens.

Fired specimens (rectangular and cylinderical) were placed centrally on two support, separated 82.00mm apart. A small rod was fixed to the point of pressure application on the Denison Srength Testing Machine, Model T.42.C.2. Pressure was applied to the centre of the specimens untill fracture occured. Dimensions at the point of rupture were determined with vernier calipers. Modulus of rupture was calculated from the acquired data.

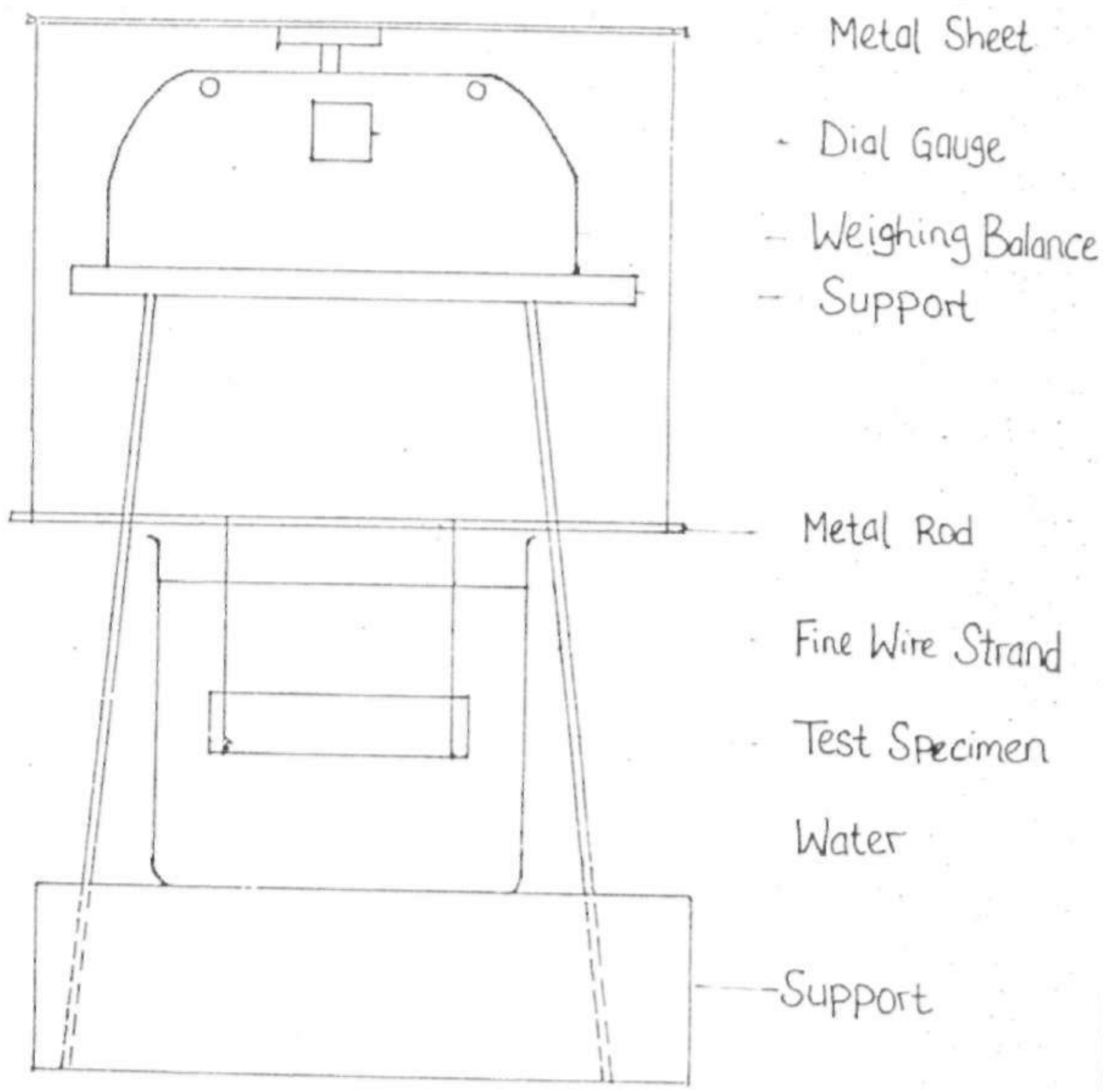


FIGURE 6: SUSPENDED WEIGHTS DETERMINATION

# CHAPTER FIVE

## 5. RESULTS

The results of this research are presented in Tables 7 - 16 and Figures 7 - 27. Detailed results have been included in Appendices.

### 5.1 INTRINSIC PHYSICAL PROPERTIES OF THE STUDIED CLAYS

Results from the plasticity tests, rheological studies, and tests on the green and fired solid casts of the clays are presented in Table 7 - 10 and Figures 7 - 17.

TABLE 7: PFEFFERKORN PLASTICITIES OF THE CLAYS

| Clay Name | Designation | Pfefferkorn Platicity |
|---|---|---|
| Kaaoje | KJ | 26.50 |
| Bukuru | BK | 27.40 |
| Bomo | BM | 31.00 |
| Goronyo MWF1 | F | 33.50 |
| Bida | BD | 39.50 |
| Ikebedi | IK | 43.50 |
| Cross River | CR | 45.00 |
| White KanKara | KK | 45.10 |
| Kano KRD | R | 46.50 |

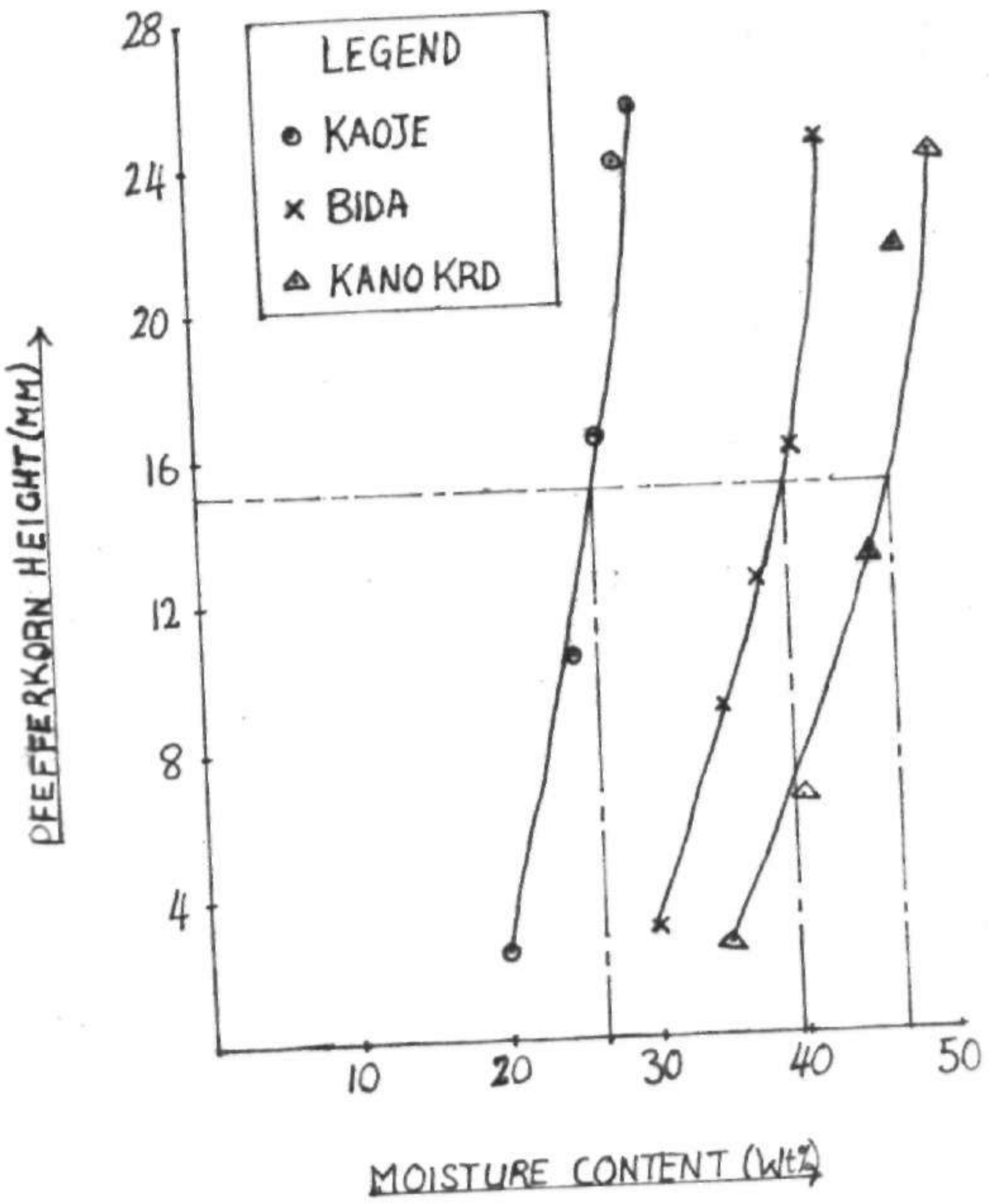


FIGURE 7: PLASTICITY TEST ON KAOJE, BIDA, AND KANO KRD CLAYS

TABLE 8: MINIMUM VISCOSITIES OF THE CLAY SLIPS AND THE DEFLOCCULANT AND WATER ADDITIONS

| S/N. | Clay Name | Water Content % wt | Deflocculant Concentration % wt | Viscosity Cp |
|---|---|---|---|---|
| 1. | Cross River | 70 | 0.465 | 8.00 |
| 2. | Kaoje | 40 | 0.235 | 32.00 |
| 3. | Bukuru | 45 | 0.400 | 55.00 |
| 4. | Kano KRD | 45 | 0.380 | 56.00 |
| 5. | Goronyo MWF1 | 50 | 0.355 | 68.00 |
| 6. | White Kankara | 45 | 0.265 | 88.00 |
| 7. | Ikebedi | 70 | 0.545 | 144.00 |
| 8. | Bida | 65 | 0.300 | 189.00 |
| 9. | Bomo | 45 | 0.325 | 192.00 |

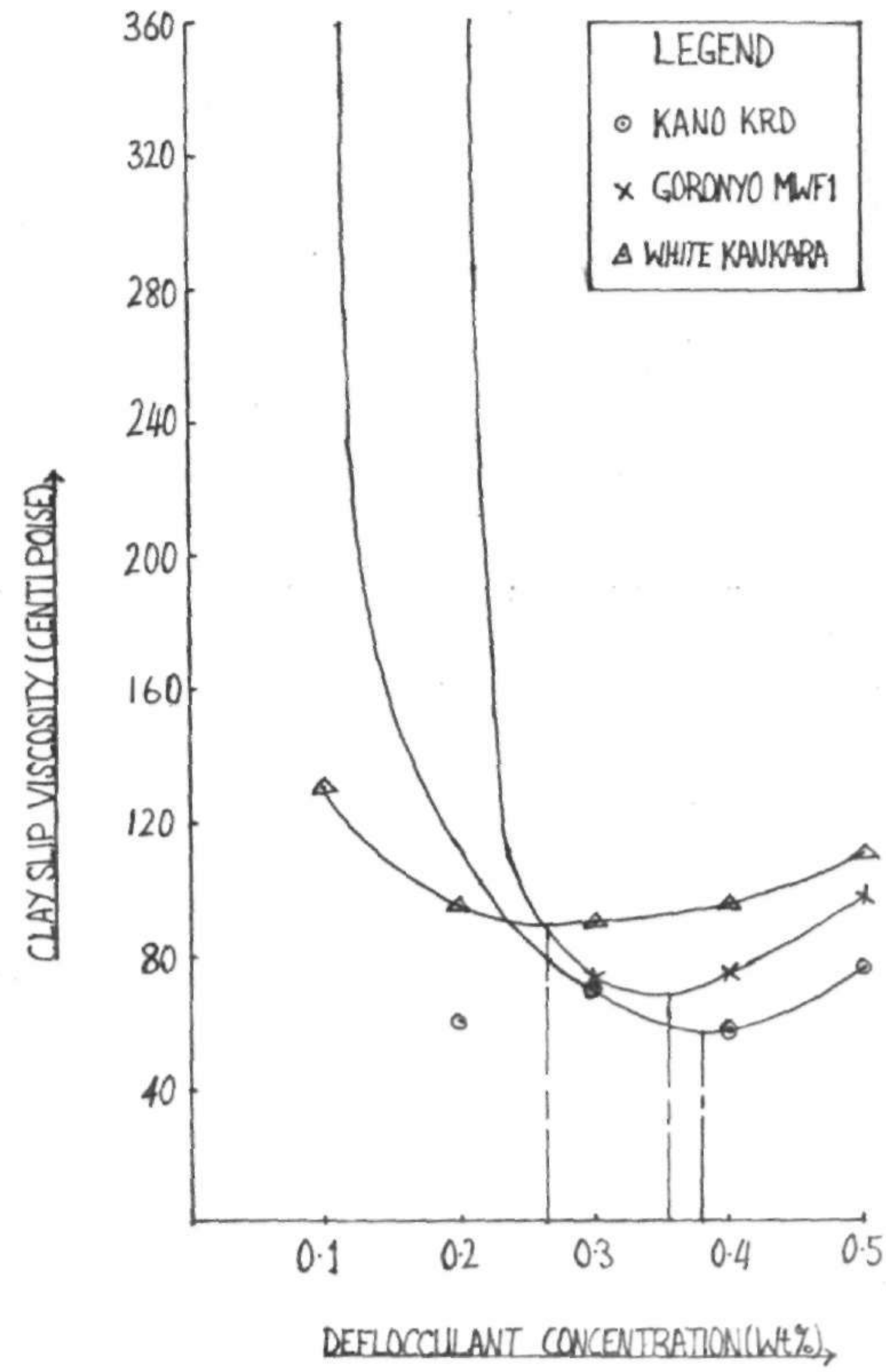


FIGURE 8: KANO KRD, GORONYO MWF1, AND WHITE KANKARA CLAY SLIPS MINIMUM VISCOSITY DETERMINATION

TABLE 9: SUMMARISED GREEN PROPERTIES OF THE CLAYS'S SOLID CASTS

| Clay Name | Linear Drying ShrinKage (LDS) % | Green Density (GD)Kg/cm | Green Modulus of Rupture (GMOR) N/Sq.cm |
|---|---|---|---|
| Kaoje | 0.56 | 1648.74 | 68 |
| Kano KRD | 1.84 | 1449.46 | 76 |
| White KanKara | 2.65 | 1413.34 | 117 |
| Ikebedi | 6.54 | 1511.77 | 158 |
| Cross River | 8.50 | 1664.55 | 190 |
| Bida | 8.93 | 1807.57 | 233 |
| Bomo | 9.41 | 1894.19 | 537 |
| Goronyo MWF1 | 11.14 | 1943.35 | 706 |
| Bukuru | NA | NA | NA |

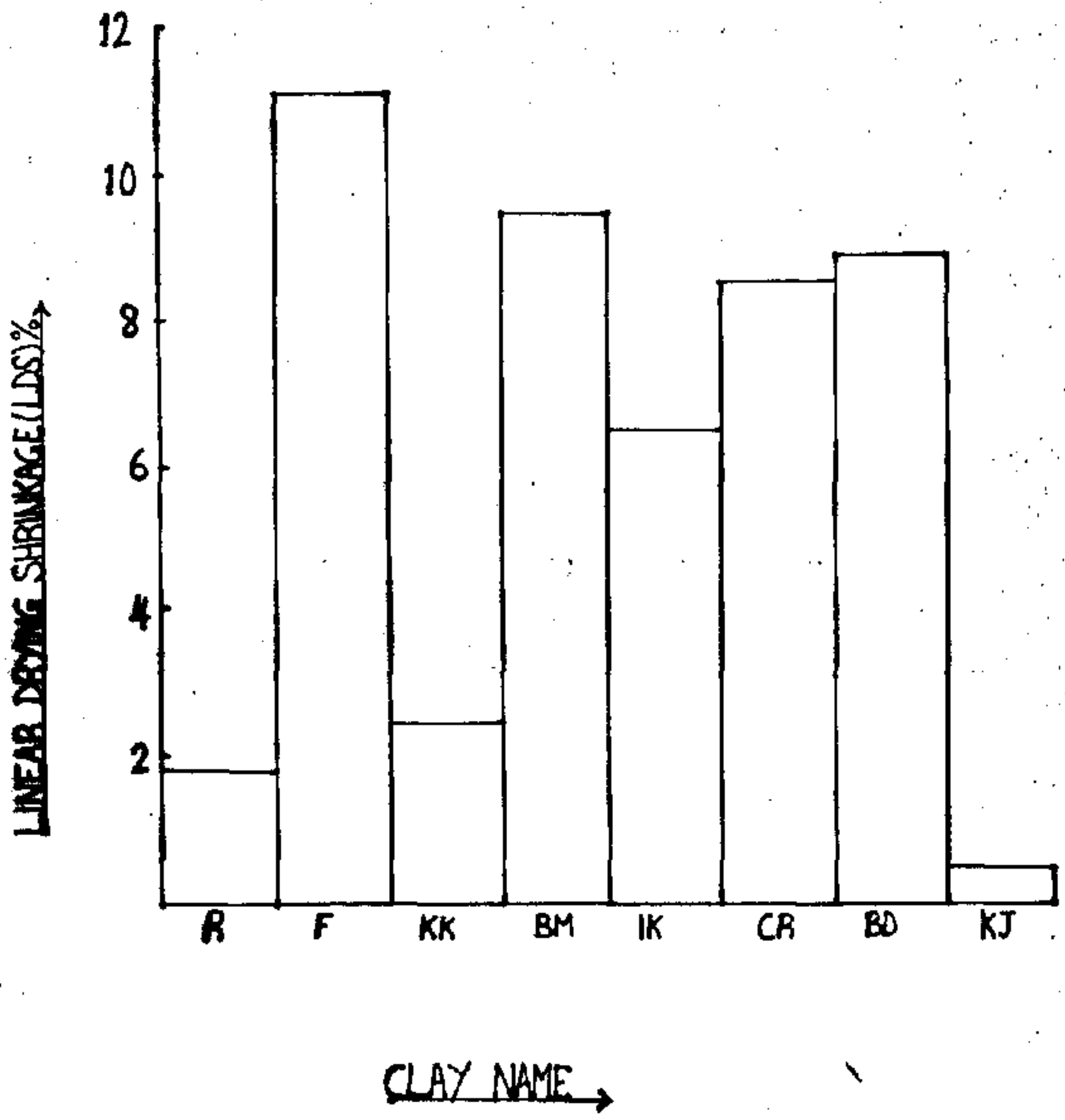


FIGURE 9: AVERAGED LDS OF THE VARIOUS CLAYS

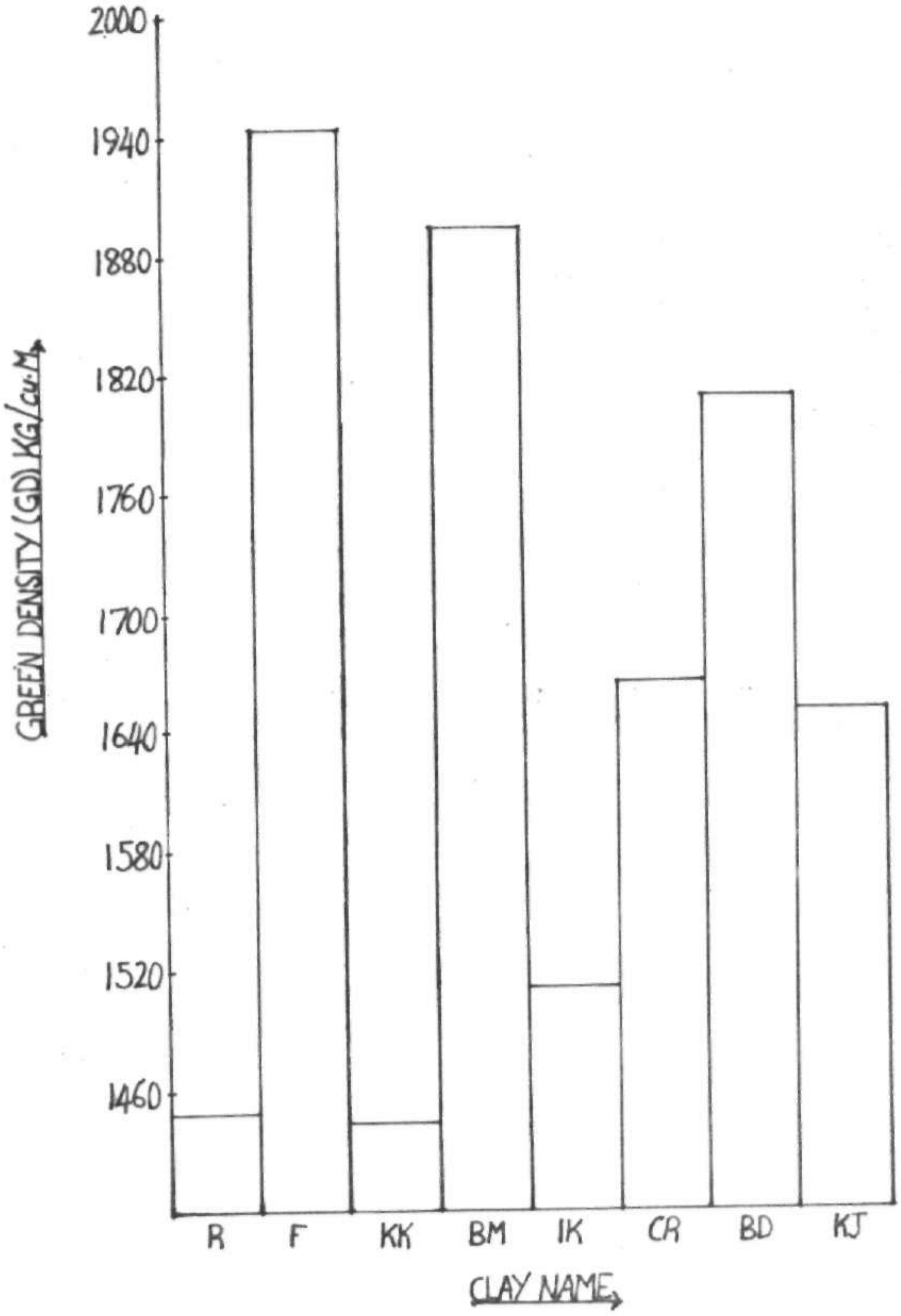


FIGURE 10: AVERAGED GD OF THE VARIOUS CLAYS

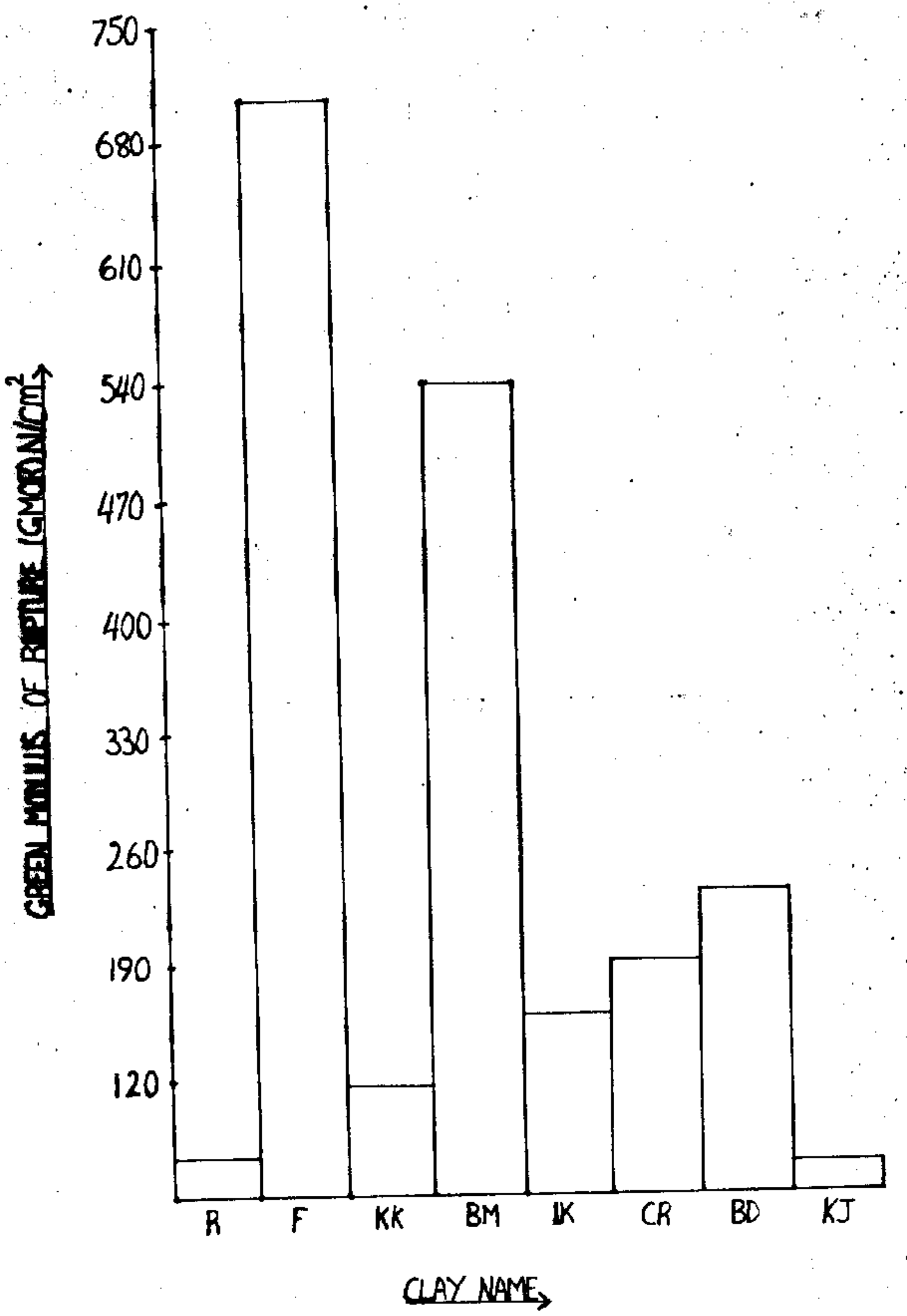


FIGURE 11: AVERAGED GMOR OF THE VARIOUS CLAYS

TABLE 10: SUMMARISED FIRED PROPERTIES OF THE CLAYS'S SOLID CASTS

| Clay Name | Linear Firing Shrink-age(LFS) wt% | Firing Weight Loss FWL wt% | Fired Density FD(Kg/Kg/m$^3$ | Fired Modulus of Rupt-ure(FMOR) KN/cm$^2$ | Apparent Porosity (Ap)(wt%) | Water Absor-ption WA(wt%) | Fired Colour (FC) |
|---|---|---|---|---|---|---|---|
| Kaoje | 4.04 | 8.45 | 1726.95 | 3.02 | 29.36 | 16.72 | White |
| Kano KRD | 10.22 | 13.12 | 1800.73 | 3.62 | 26.66 | 14.38 | Pure-white |
| White Kankara | 11.76 | 12.89 | 1883.00 | 4.73 | 22.49 | 11.82 | Dull White |
| Ikebedi | NA | NA | NA | NA | NA | NA | Cream |
| Cross river | 9.98 | 9.42 | 2273.74 | 1.74 | 12.03 | 5.61 | Dark Cream |
| Bida | 7.55 | 9.21 | 2049.08 | 3.53 | 14.76 | 7.49 | Light Brow |
| Bomo | 4.76 | 5.17 | 2167.84 | 2.48 | 0.00 | 0.00 | Dark Brown |
| Goronyo MWF1 | 2.63 | 6.35 | 2109.27 | 4.01 | 10.66 | 5.19 | Yellowish Br |
| Bukuru | NA | NA | NA | NA | NA | NA | Brown |

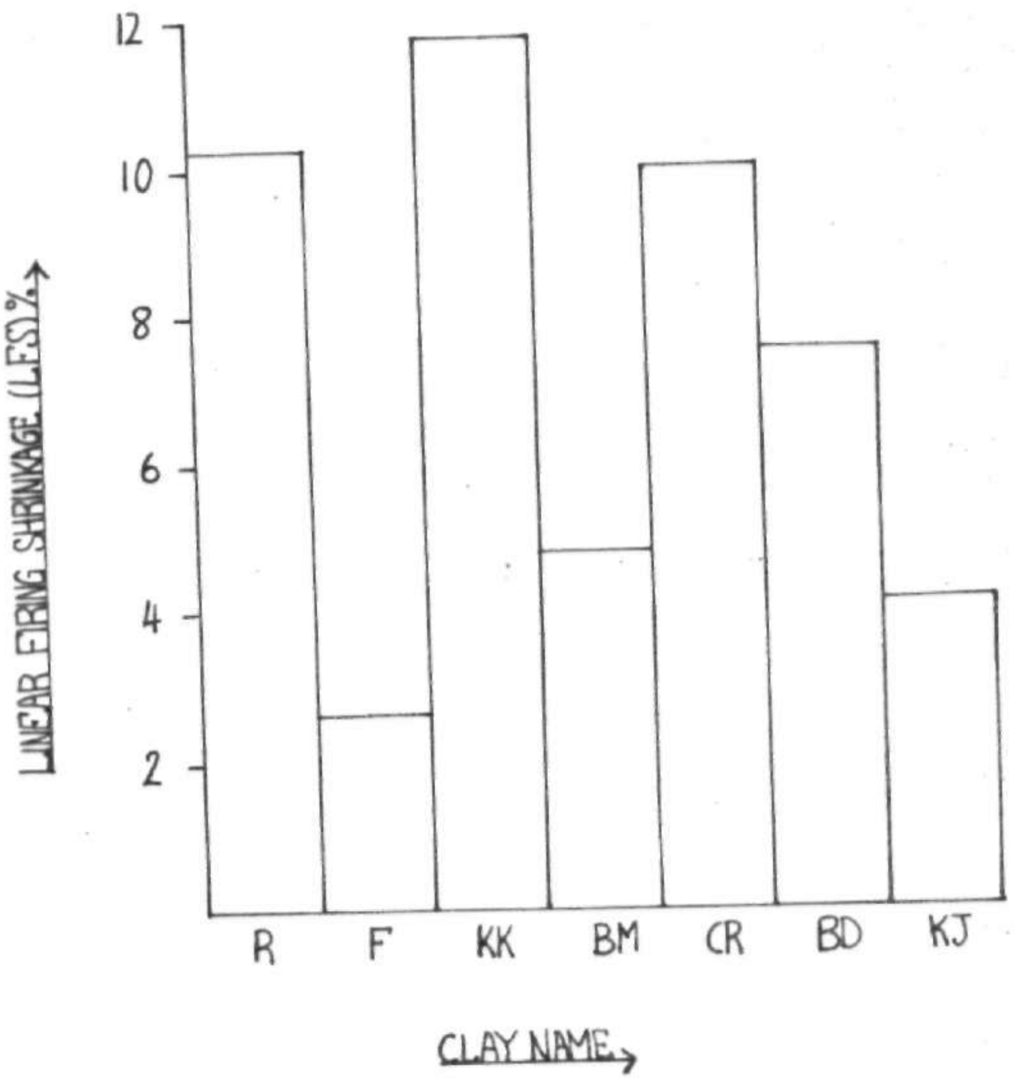


FIGURE 12: AVERAGED LFS OF THE VARIOUS CLAYS

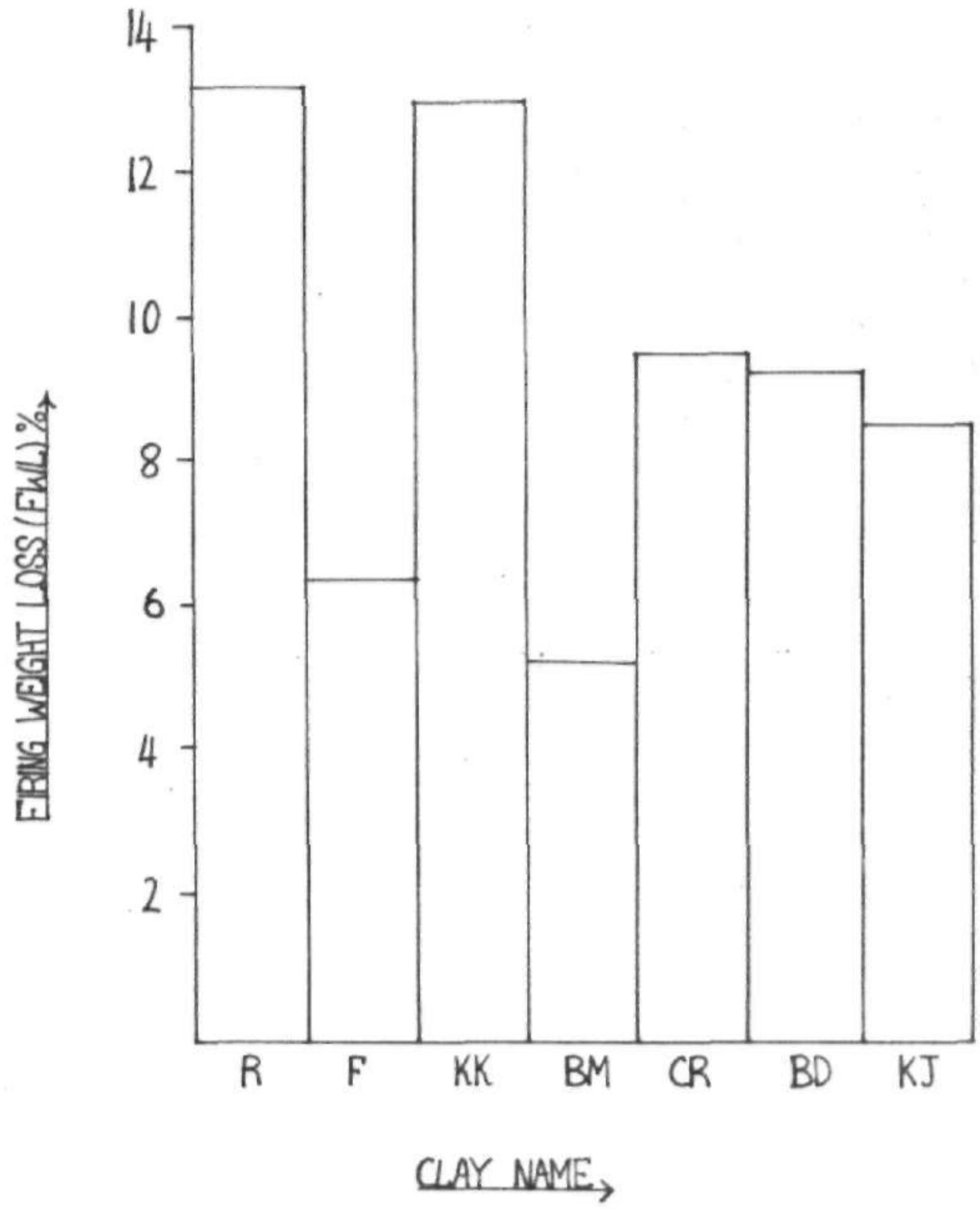


FIGURE 13: AVERAGED FWL OF THE VARIOUS CLAYS

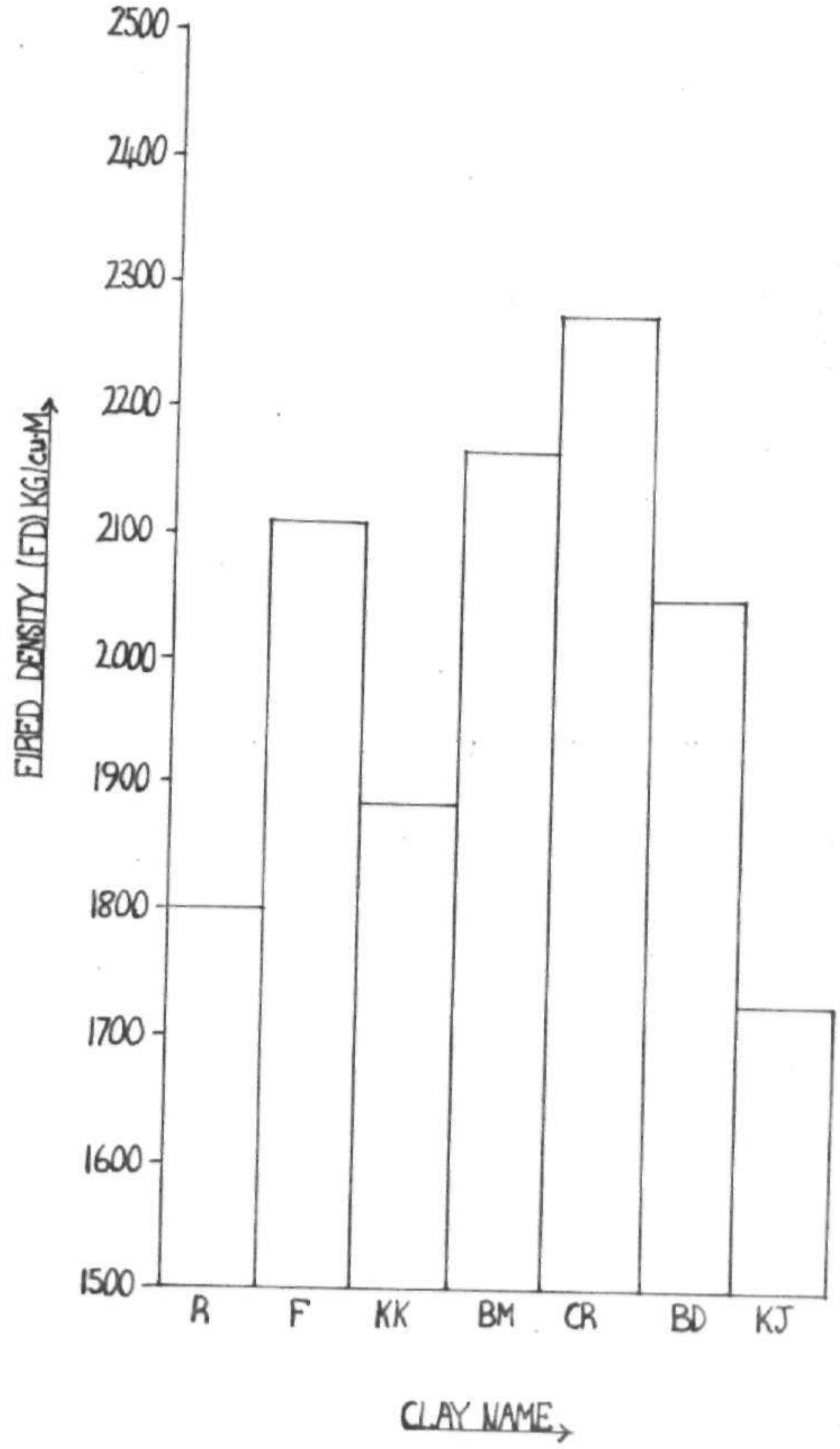


FIGURE 14: AVERAGED FD OF THE VARIOUS CLAYS

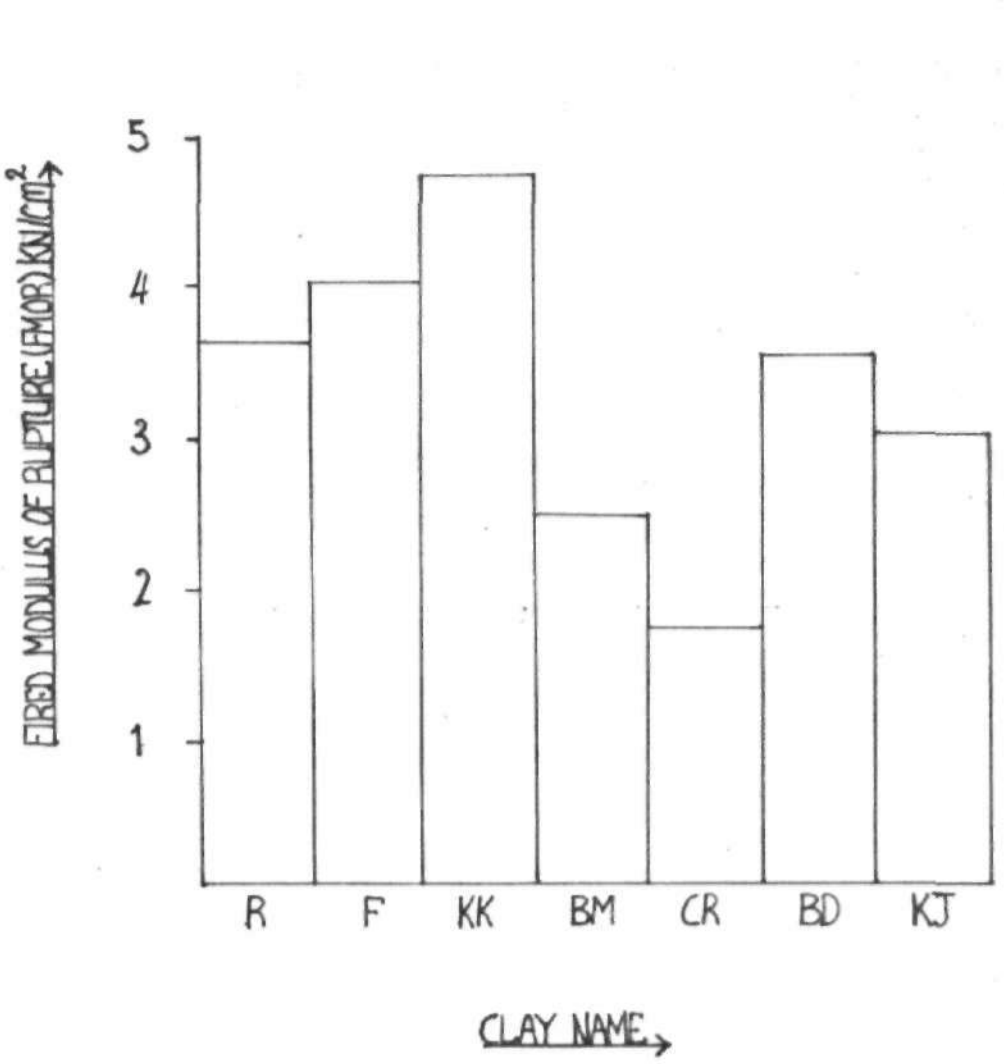


FIGURE15 : AVERAGED FMOR OF THE VARIOUS CLAYS

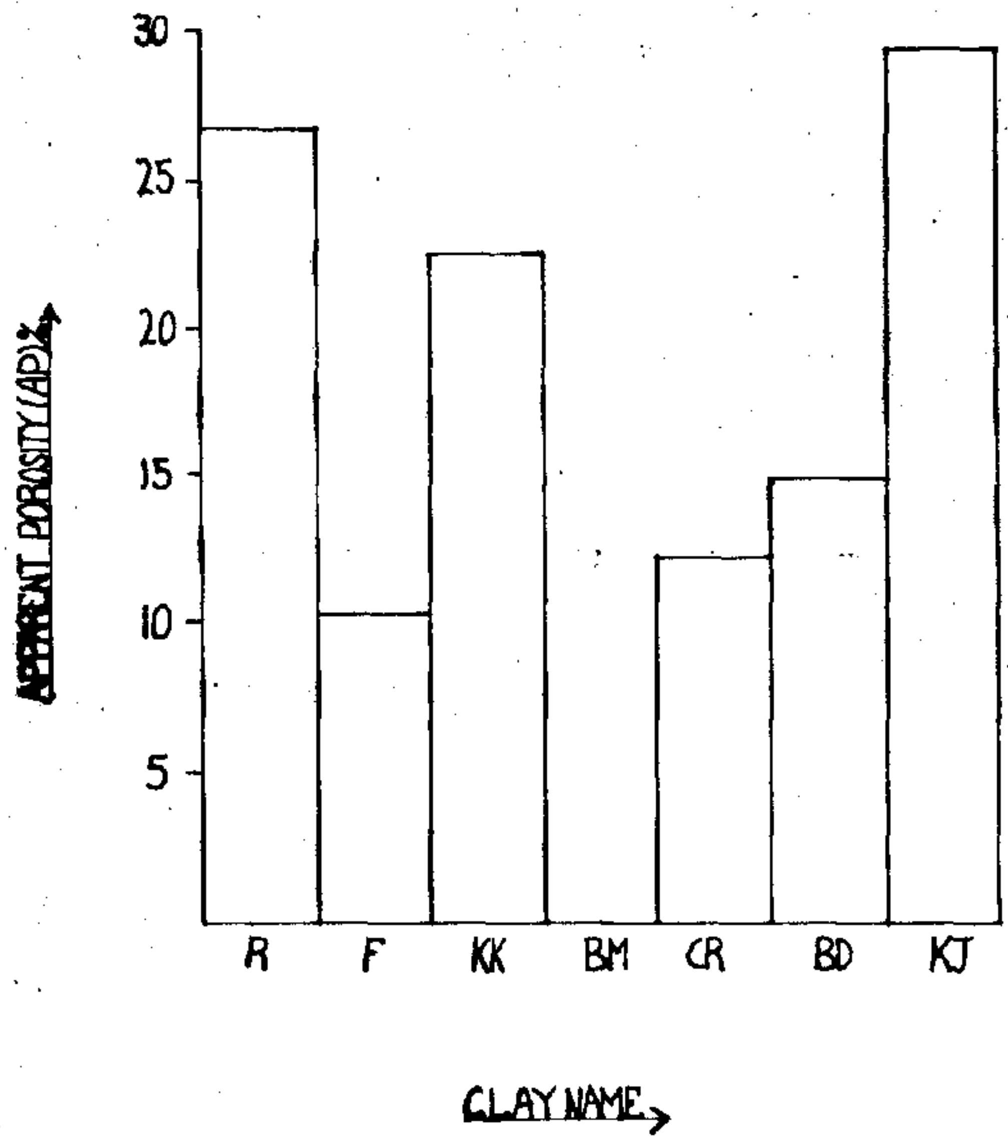


FIGURE 16: AVERAGED AP OF THE VARIOUS CLAYS

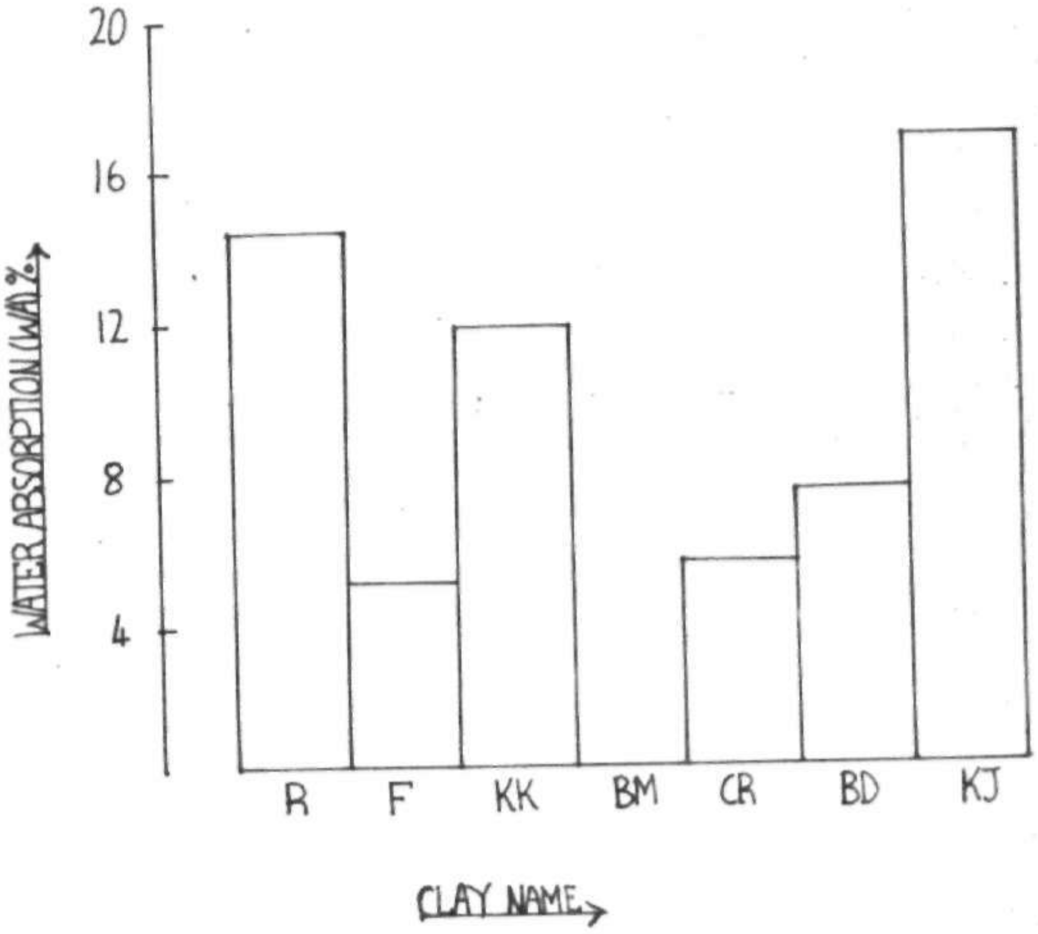


FIGURE17: AVERAGED WA OF THE VARIOUS CLAYS

## 5.2 GREEN PHYSICAL PROPERTIES OF THE TRIAL PRESSED PORCELAINS

Results of the test on the physical properties, namely, linear drying shrinkages, green densities, and green moduli of rupture, of the trial porcelains, produced using either Bomo, Bukuru, or Bida clay as the binding clay, and a pressure of either 2.0t (102.07kg per sq.cm) or 4.5t (229.67kg per sq.cm) are presented in Table 11 and Figures 18 and 19.

TABLE 11: SUMMARISED GREEN PROPERTIES OF THE TRIAL PRESSED PORCELAINS

| Binding Clay | Design-tion | Pressing Pressure/ Stage (t) | Linear Drying Shrinkage (LDS)% | Green Density (GD) Kg/cu.m | Green Modulus of Rupture (GMOR)N/Sq.cm |
|---|---|---|---|---|---|
| Bomo | PHBMD | 4.5/1.5:4.5 | 0.00 | 1879.62 | 88 |
| Bukuru | PHBKD | 4.5/1.5:4.5 | 0.00 | 1861.27 | 96 |
| Bida | PHBDD | 4.5/1.5:4.5 | 0.00 | 1890.08 | 112 |
| Bida | PLBDD | 2.0/Single Stage | 0.00 | 1807.88 | 64 |

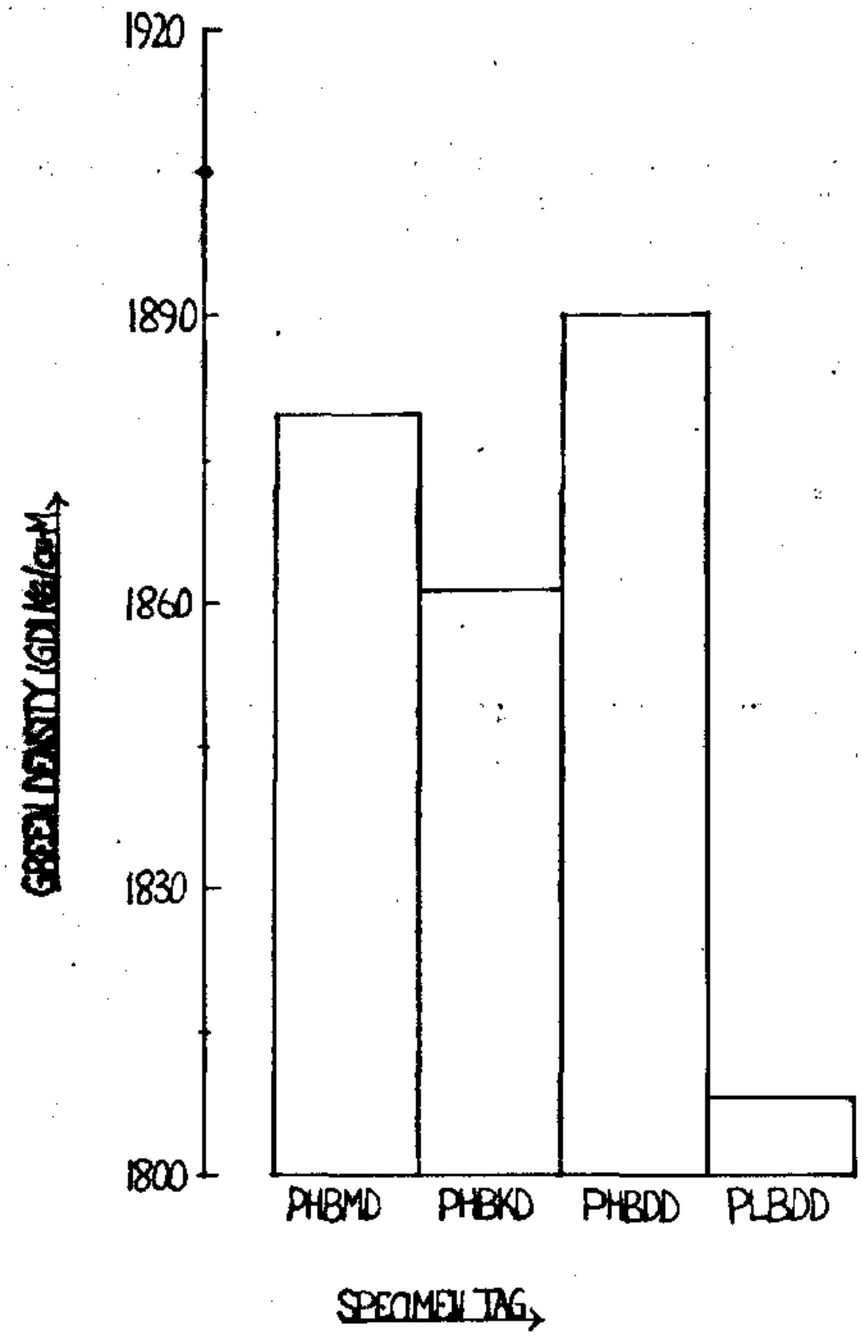


FIGURE18: AVERAGED GD OF THE TRIAL PRESSED PORCELAINS

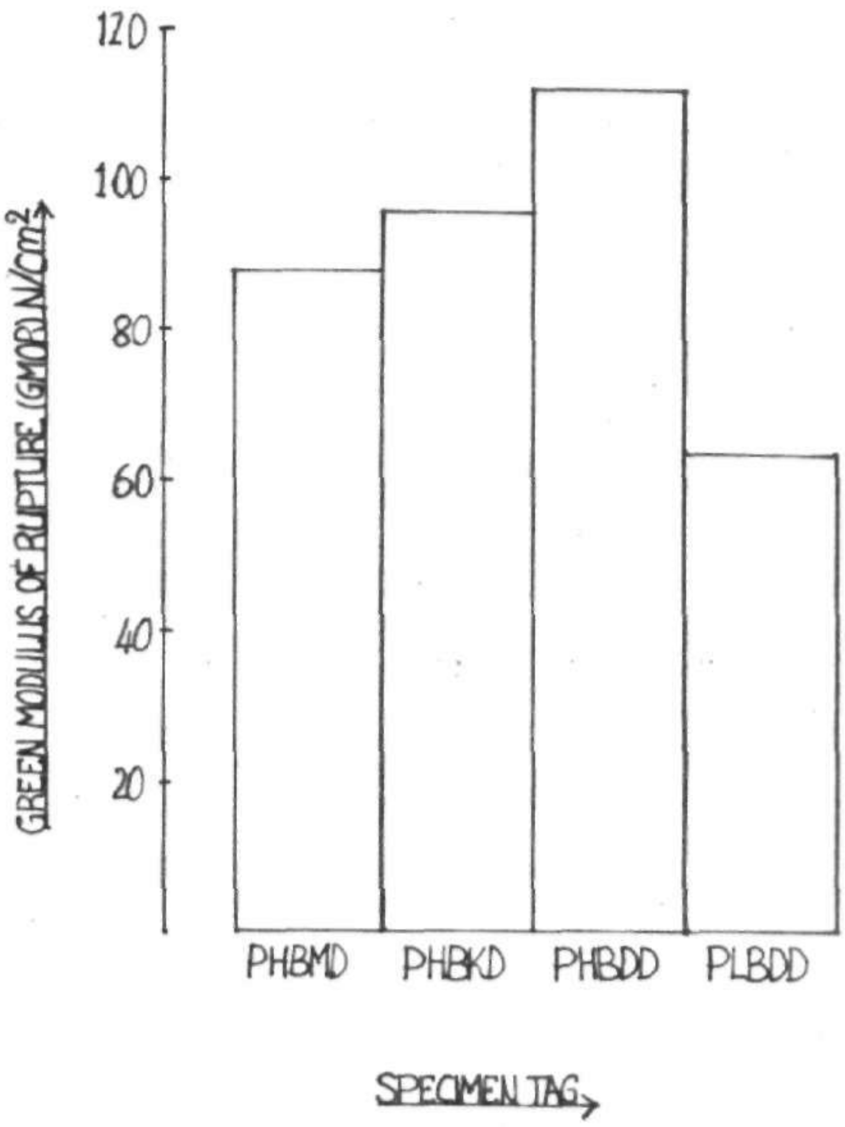


FIGURE 19: AVERAGED GMOR OF THE TRIAL PRESSED PORCELAINS

## 5.3 GREEN AND FIRED PROPERTIES OF THE PRESSED PORCELAINS

Results of the tests Carried out, for green and fired physical properties, on the presed porcelains are presented in table 12-16 and illustrated in Figures 20-27.

TABLE 12: SUMMARISED GREEN PROPERTIES OF THE PRESSED PORCELAINS

| COMPOSITION | | | | | Liner Drying Shrink-age. (LDS)% | Green Density (GD) $Kg/m^3$ | Green Modulus of Rupture GMOR N/Sq.cm |
|---|---|---|---|---|---|---|---|
| Body | %KK Clay | %BD Clay | %S.Sand | %Feld-Spar | | | |
| A | 35 | 10 | 30 | 25 | 0.00 | 1756.22 | 62 |
| B | 35 | 10 | 25 | 30 | 0.00 | 1813.38 | 59 |
| C | 40 | 10 | 20 | 30 | 0.00 | 1821.27 | 65 |
| D | 30 | 10 | 25 | 35 | 0.00 | 1797.72 | 51 |

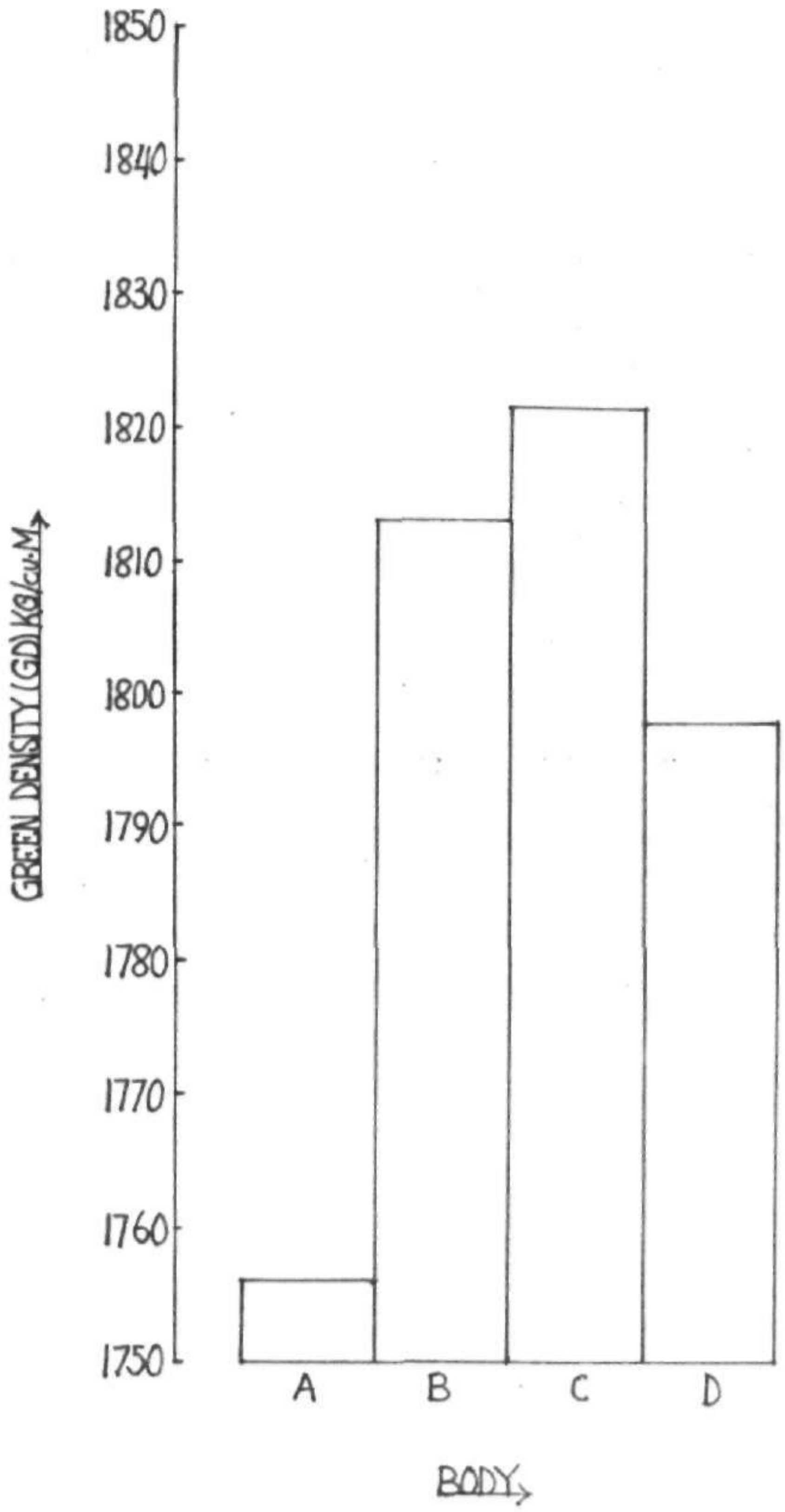


FIGURE20: AVERAGED GD OF THE PRESSED PORCELAINS

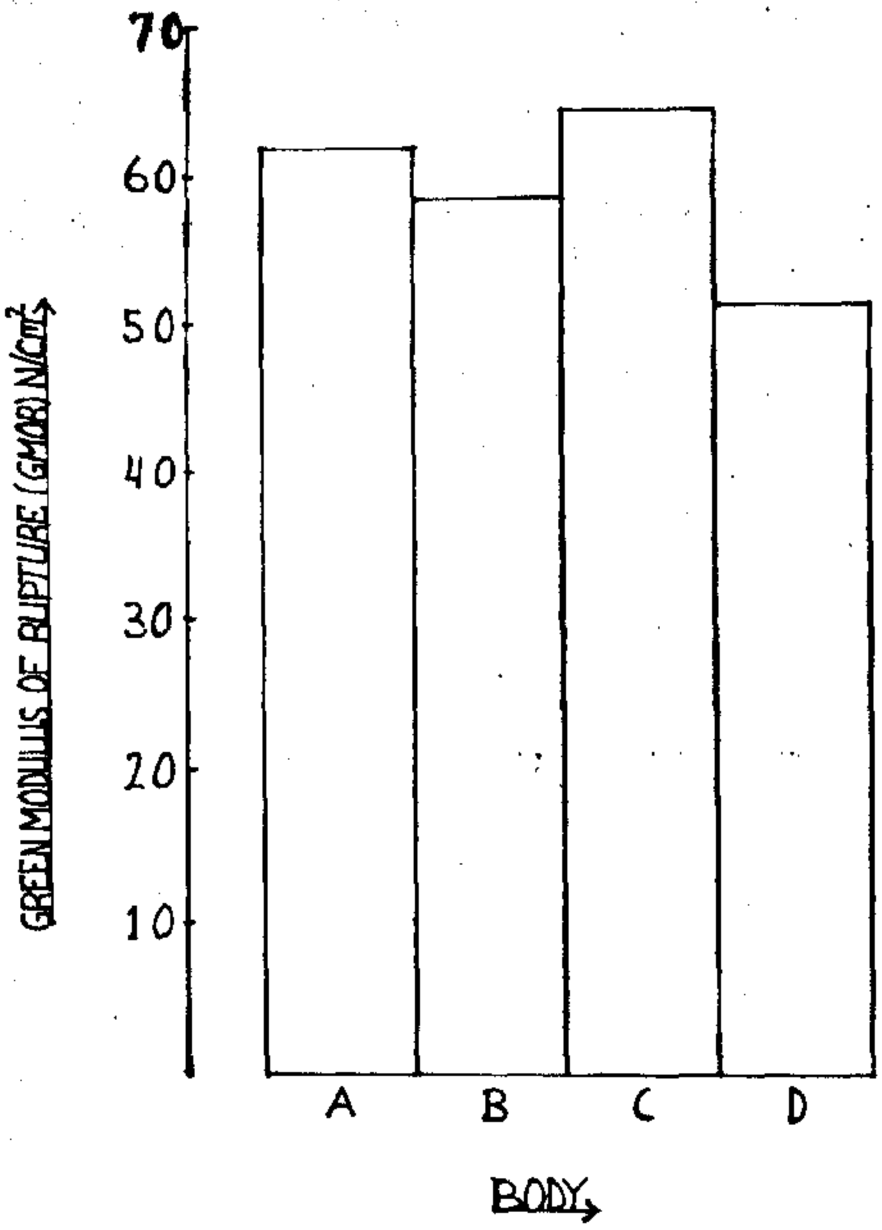


FIGURE 21: AVERAGED GMOR OF THE PRESSED PORCELAINS

TABLE 13: SUMMARISED FIRED PROPERTIES OF THE PRESSED PORCELAINS FIRED TO 1180°C

| Body | Linear Firing Shrink-age(LFS) wt% | Firing Weight Loss (FWL) wt% | Fired Density (FD) Kg/m$^3$ | Fired Modulus of Rupt-ure(FMOR) KN/cm$^2$ | Apparent Porosity (Ap) wt% | Water Absor-ption (WA) wt% |
|---|---|---|---|---|---|---|
| A | 6.63 | 5.87 | 2055.15 | 2.38 | 12.74 | 6.15 |
| B | 7.15 | 5.89 | 2145.56 | 2.60 | 11.20 | 5.29 |
| C | 7.25 | 6.56 | 2152.91 | 2.59 | 6.79 | 3.11 |
| D | 7.39 | 5.28 | 2157.06 | 3.06 | 4.90 | 2.25 |

TABLE 14: SUMMARISED FIRED PROPERTIES OF THE PRESSED PORCELAINS FIRED TO 1220°C

| Body | Linear Firing Shrink-age(LFS) wt% | Firing Weight Loss (FWL) wt% | Fired Density (FD) Kg/m$^3$ | Fired Modulus of Rupt-ure(FMOR) KN/Sq.cm | Apparent Porosity (Ap)wt% | Water Absor-ption (WA) wt% |
|---|---|---|---|---|---|---|
| A | 6.81 | 6.12 | 2031.44 | 2.3 | 12.66 | 6.15 |
| B | 7.17 | 6.20 | 2132.83 | 2.30 | 9.45 | 4.23 |
| C | 7.05 | 6.68 | 2144.90 | 3.31 | 9.14 | 4.30 |
| D | 7.52 | 5.30 | 2143.07 | 3.44 | 4.94 | 2.28 |

TABLE 15: SUMMARISED FIRED PROPERTIES OF THE PRESSED PORCELAINS FIRED TO 1260°C

| Body | Linear Firing Shrink-age(LFS) wt% | Firing Weight Loss (FWL) wt% | Fired Density FD(Kg/ Kg/$m^3$ | Fired Modulus of Rupt-ure(FMOR) KN/Sq.cm | Apparent Porosity (Ap)wt% | Water Absor-ption (WA)wt% |
|---|---|---|---|---|---|---|
| A | 7.94 | 6.16 | 2164.11 | 2.92 | 6.92 | 3.21 |
| B | 8.29 | 6.14 | 2238.42 | 3.46 | 4.91 | 2.23 |
| C | 8.25 | 6.77 | 2185.28 | 2.96 | 2.48 | 1.12 |
| D | 8.31 | 5.41 | 2201.23 | 3.35 | 0.08 | 0.04 |

TABLE 16: SUMMARISED FIRED PROPERTIES OF THE PRESSED PORCELAINS FIRED TO 1300°C

| Body | Linear Firing Shrink-age(LFS) wt% | Firing Weight Loss (FWL) wt% | Fired Density FD(Kg/ Kg/$m^3$ | Fired Modulus of Rupt-ure(FMOR) KN/Sq.cm | Apparent Porosity (Ap)wt% | Water Absor-ption (WA)wt% |
|---|---|---|---|---|---|---|
| A | 8.13 | 6.26 | 2125.83 | 35.6 | 0.98 | 0.45 |
| B | 8.11 | 6.38 | 2171.94 | 32.9 | 0.13 | 0.06 |
| C | 8.64 | 6.82 | 2231.19 | 34.3 | 0.00 | 0.00 |
| D | 8.41 | 5.57 | 2033.44 | 36.4 | 0.00 | 0.00 |

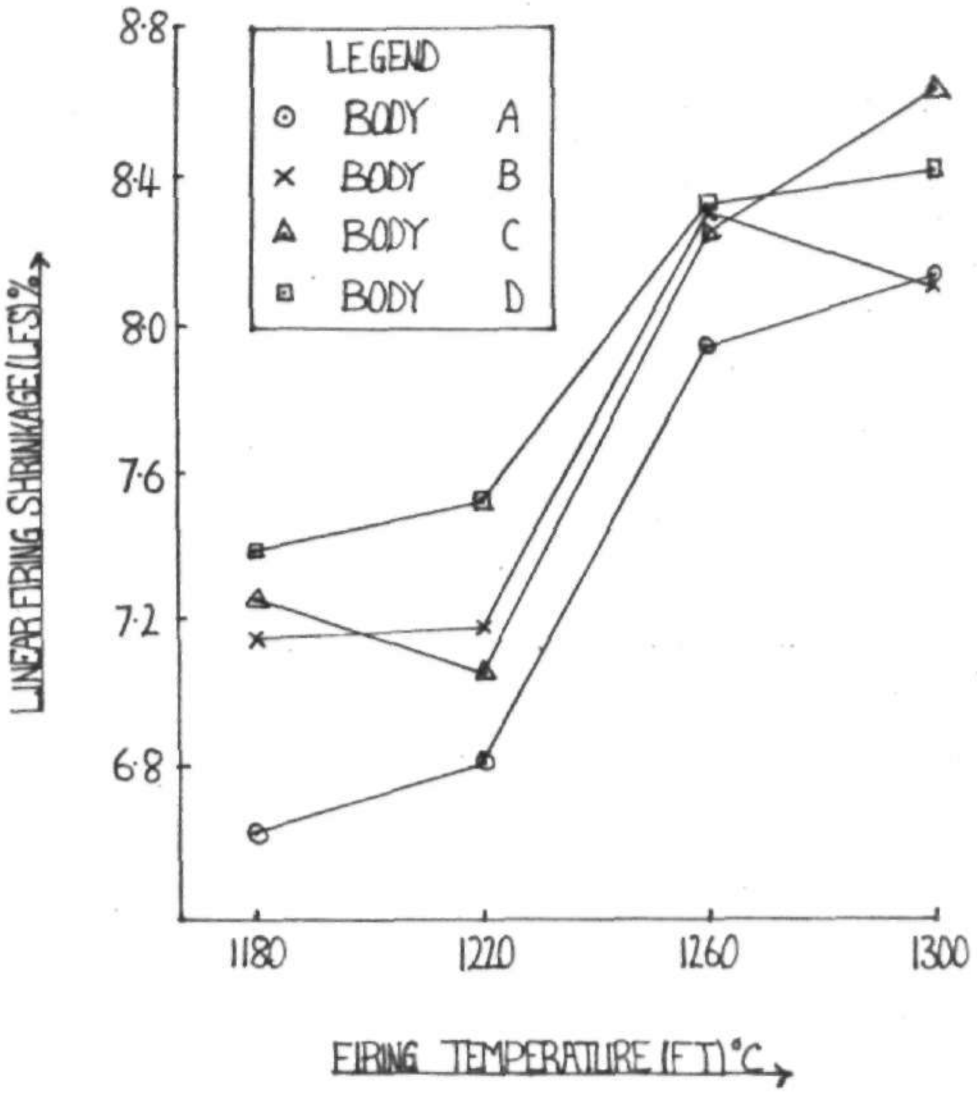


FIGURE 22: AVERAGED LFS OF THE PRESSED PORCELAINS

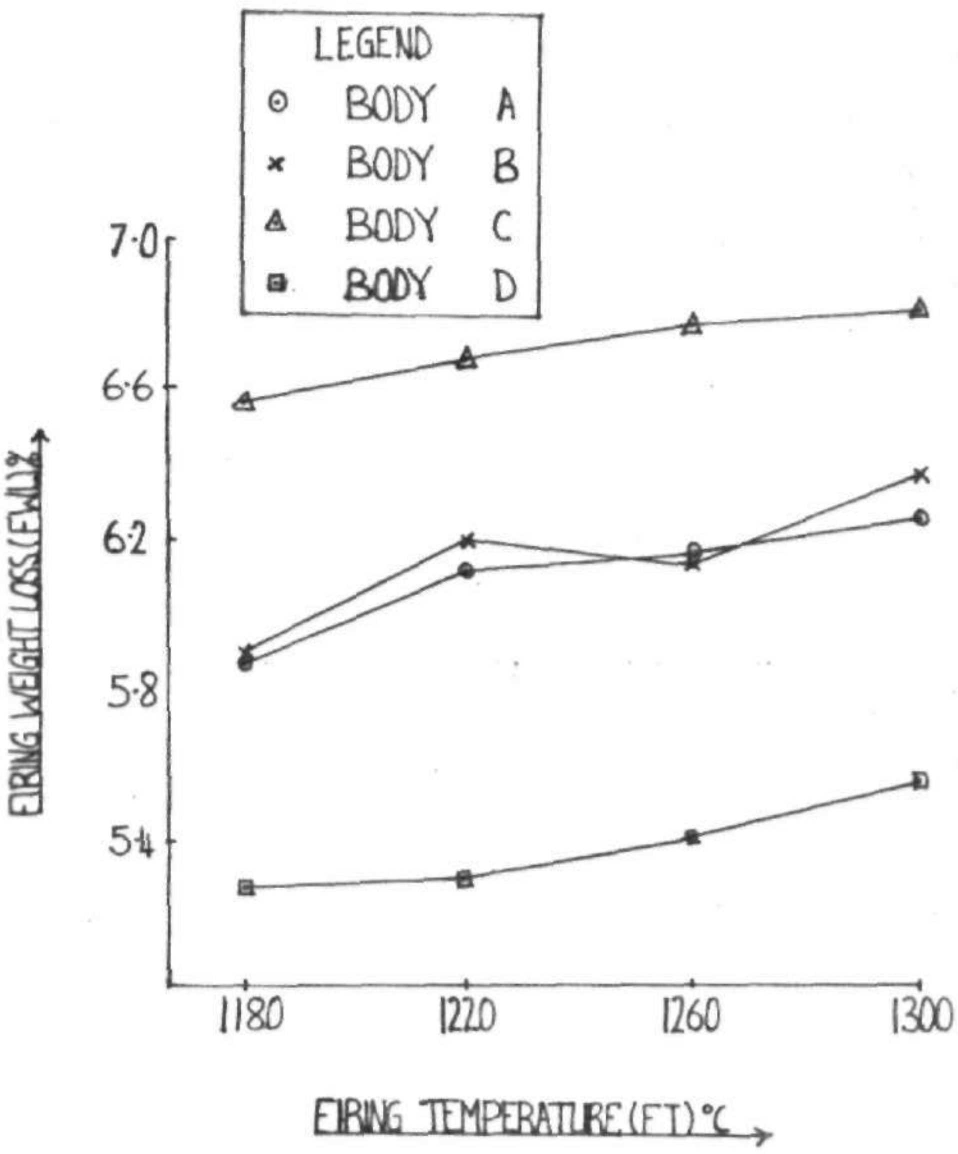


FIGURE 23: AVERAGED FWL OF THE PRESSED PORCELAINS

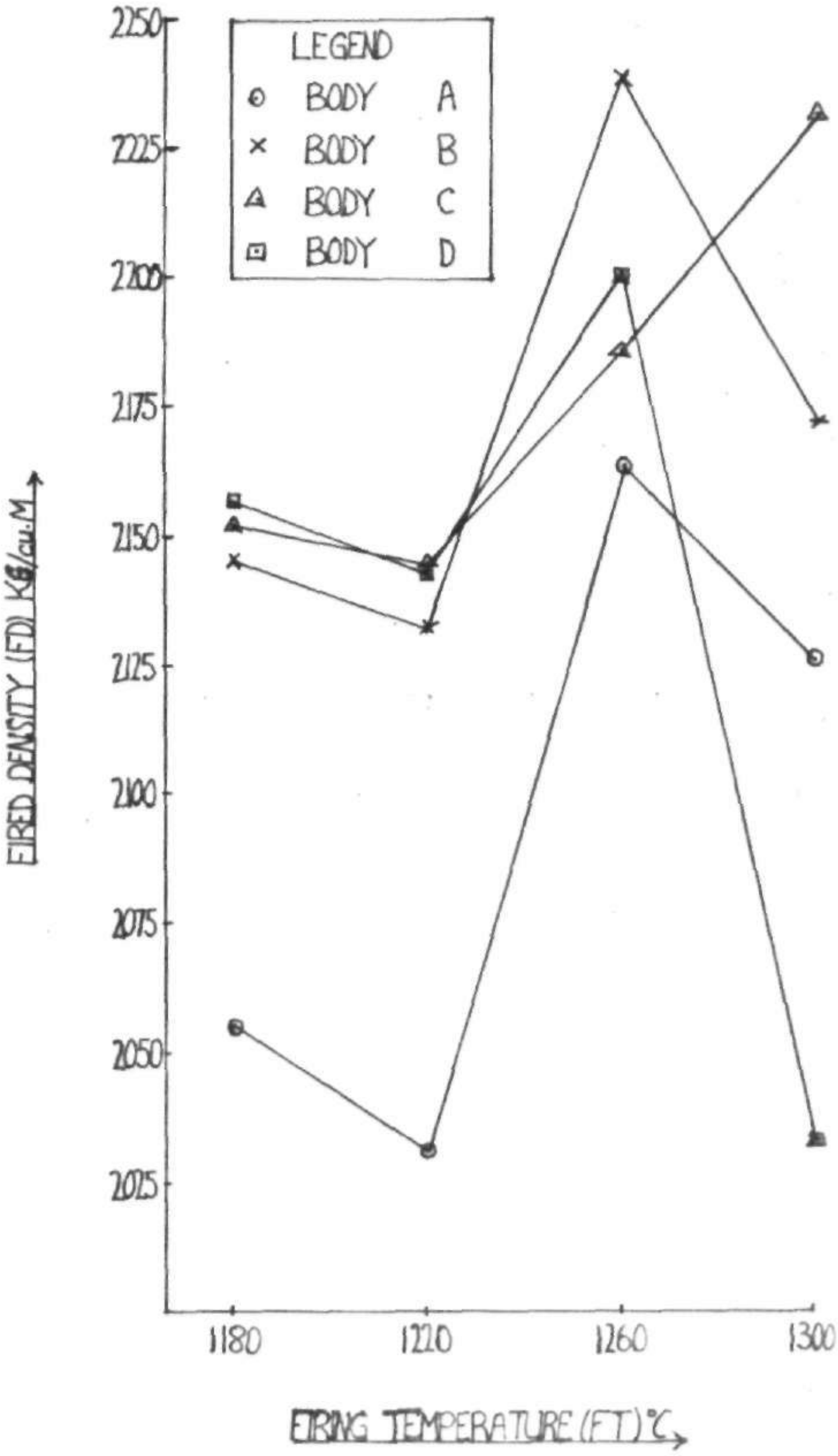


FIGURE 24: AVERAGED FD OF THE PRESSED PORCELAINS

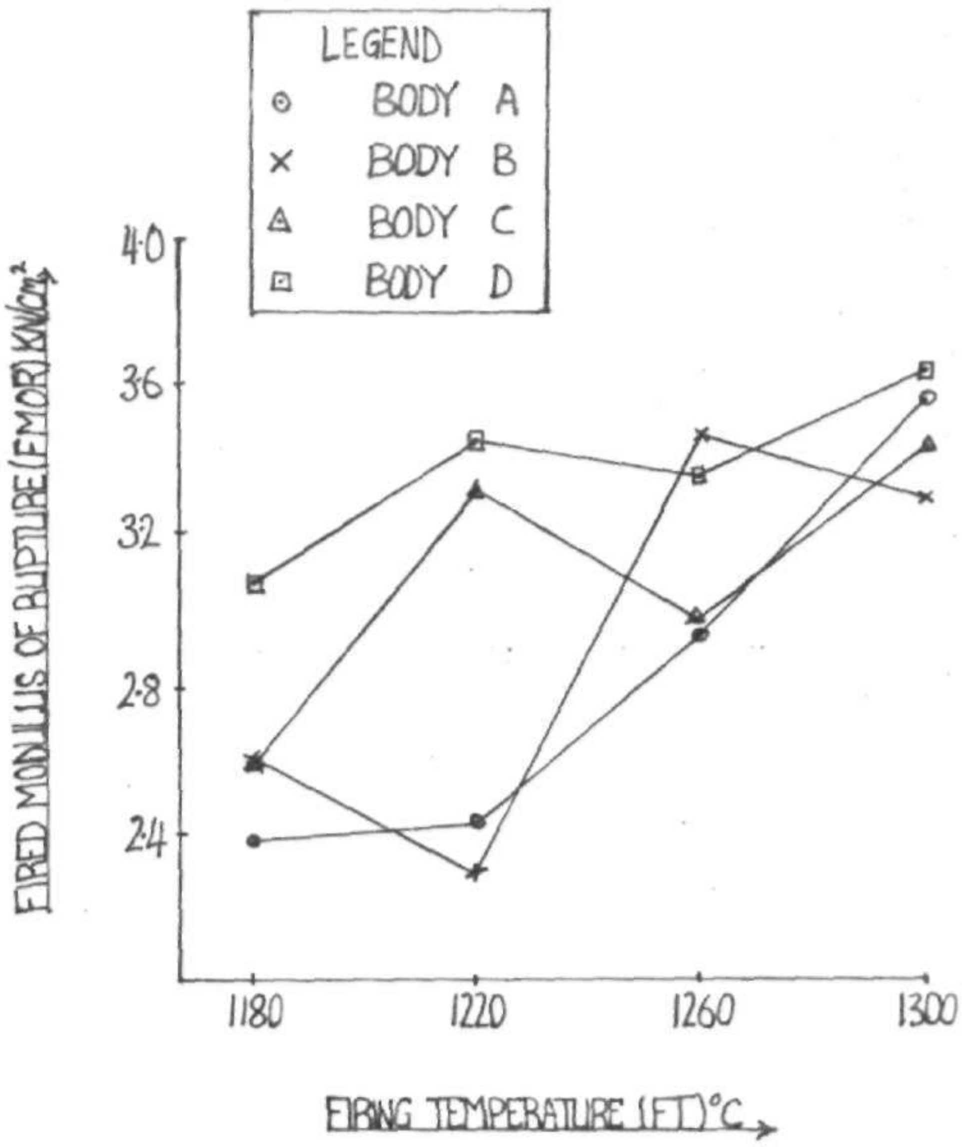


FIGURE 25: AVERAGED FMOR OF THE PRESSED PORCELAINS

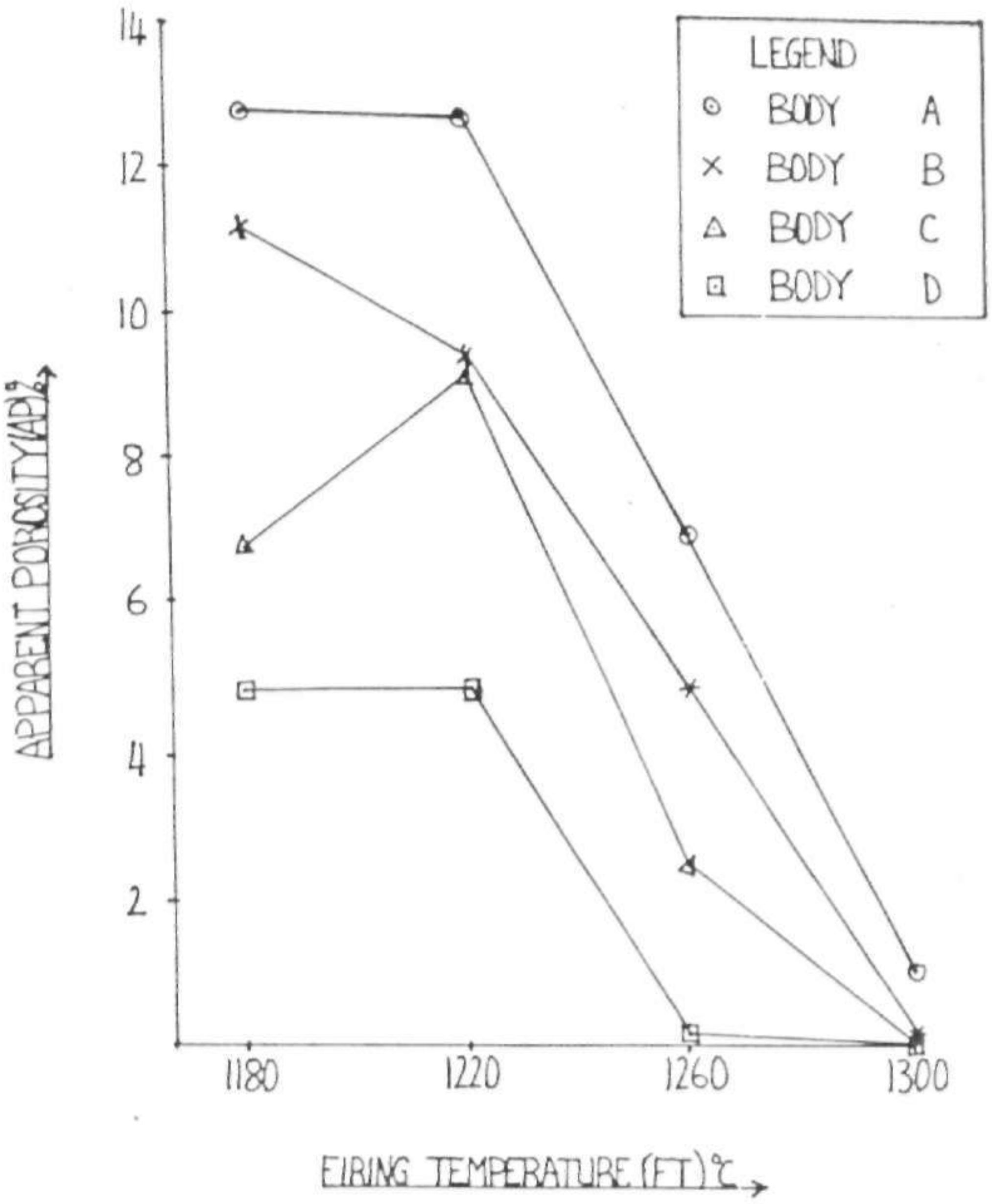


FIGURE 26: AVERAGED AP OF THE PRESSED PORCELAINS

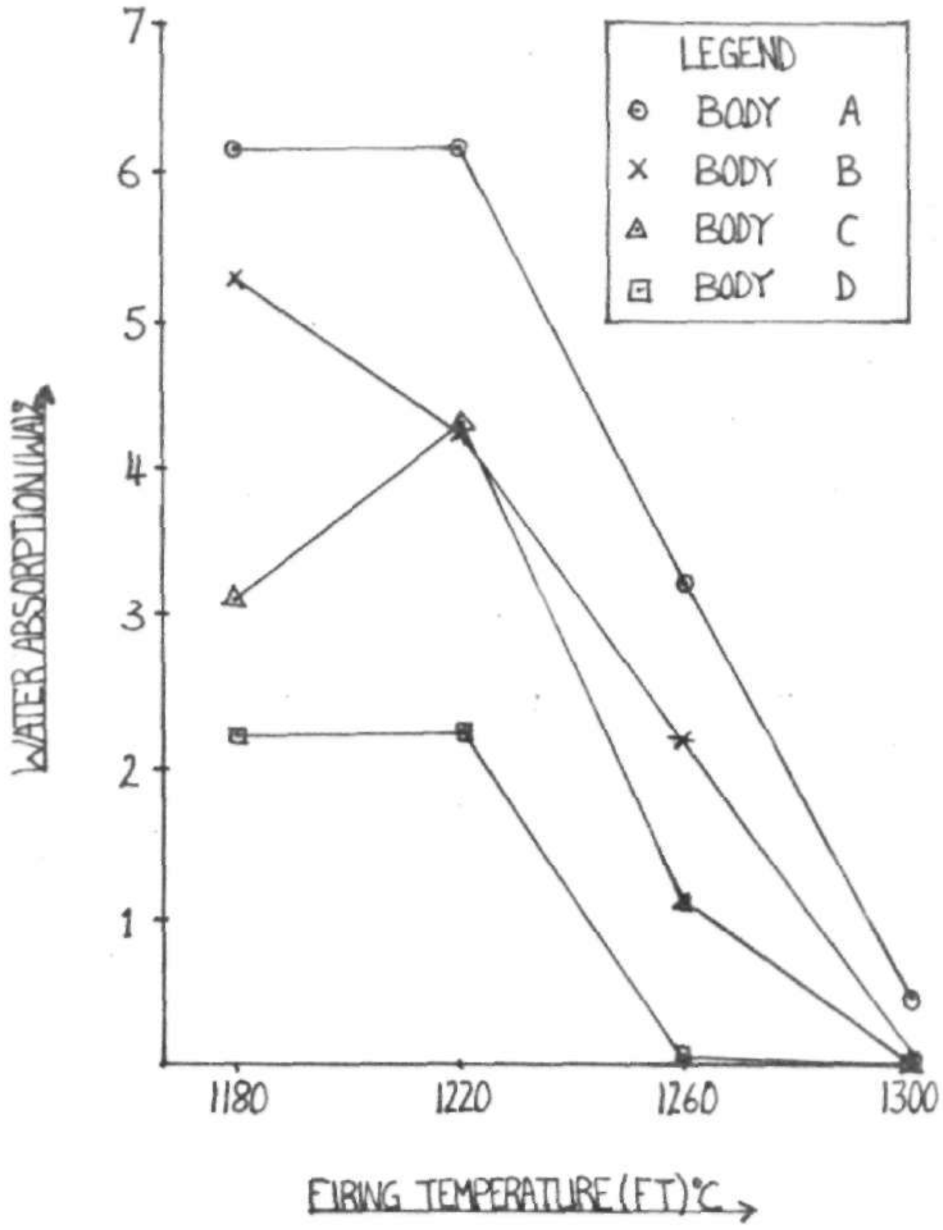


FIGURE 27: AVERAGED WA OF THE PRESSED PORCELAINS

# CHAPTER SIX

## 6. DISCUSSION OF RESULTS

Discussions are presented in two parts mainly, the first relates to the intrinsic physical properties of the clays, and the other part centres on the properties of the dry pressed electrical porcelains.

### 6.1 INTRINSIC PHYSICAL PROPERTIES OF THE CLAYS

The plasticities of the clays, and the dried physical properties, such as, linear drying shrinkages, dried densities, green moduli of rupture, and the fired physical properties, such as, linear firing shrinkages, firing weight losses, fired densities, fired moduli of rupture, apparent porosities and water absorption of the casts were studied and are discussed.

#### 6.1.1. Pfefferkorn Plasticity Indices

The Pfefferkorn plasticity indices of the nine clays showed a range of 26.50 - 46.50, with Kaoje being the most plastic and Kano KRD being the least plastic. The clays showed the following pattern of decreasing Plasticity: Kaoje> Bukuru> Bomo> Goronyo MWF1> Bida> Ikebedi> Cross river> Kankara> Kano KRD.

6.1.2 <u>Minimum Viscosities of the Clay slips</u>

These were determined for the purpose of determining the most suitable deflocculant concentration that would give a better solid casts of the clays.

The minimum viscosities of the clay slips were within 8 - 192Cp. The following order of increasing minimum viscosity of the slips was observed. Cross river<Kaoje <Bukuru<KanoKRD<Goronyo MWF1< Kankara<Ikebedi<Bida<Bomo.

However, castability was best with the slips of Kano KRD, Kankara, and Kaoje.

6.1.3 <u>Green Physical Properties of the Clays Solid Casts</u>

The linear drying shrinkages, dried densities and green moduli of rupture have been presented in Table 8 and depicted in form of histograms, for quick comparisons, in Figure 9-11.

Kano KRD, Kankara and Kaoje Clays have very low drying Shrinkages relative to the rest. This probably points to these clays as Kaolin.

Goronyo MWF1, Bomo, and Bida Clays have the highest linear drying Shrinkages, and also high dried Strengths. These clays might be stoneware clays. They showed high dry strengths. Cross river and Ikebedi clays had behaviours in-between those of Kaolins and Stone ware clays. Thus, from the fired colours and plastic properties, it does seems that no ball clay is among the samples investigated, though Ikebedi came close but was not plastic enough.

6.1.4 Fired Properties of the Clays's Solid Casts

Linear firing shrinkages, firing weight losses, fired densities, fired moduli of rupture, apparent porosities and water absorptions of the casts were presented in the form of histograms in Figures 12-17 and Table 9.

Solid casts of Ikebedi clay shatterred to pieces on firing, as such, its fired properties could not be defermined.

Kano KRD, KanKara and Cross river Clays showed high linear firing shrinkages which could suggest high flux contents in these clays leading to high degree of vitrification.

Kaoje Clay casts did not, however, show high linear firing shrinkage probably due to higher degree of purity leading to lesser vitrification. Goronyo MWF1, Bomo and Bida showed very low linear firing shrinkage probably due to higher content of fine particles which could be the possible reason for their high linear drying shrinkages, bulk green densities and moduli of rupture. However, relative to Goronyo MWF1 and Bomo, Bida clay had higher firing shrinkage possibly due to higher content of carboneous matter, as suggested by its unfired and fired colours, which were black and light brown respectively, and its higher firing weight loss.

Kano KRD and KanKara clays showed the highest firing weight losses and white fired colours.

Bomo clay exhibited low firing weight loss, dark brown fired colour, high fired density, low linear firing shrinkage, no apparent porosity, and no water absorption, together with low fired modulus of rupture. These indicates the possible present of colour firing non volatile matter, high content of fluxes which made the fired cast more vitrified and hence more brittle and low content of volatile matter.

Cross river clay showed appreciable firing weight loss and linear firing shrinkage, the highest fired density, lowest fired modulus of rupture, some apparent porosity and water absorption, together with a dark creamy fired colour. This points to the possibility of the clay containing appreciable volatile matters together with high proportion of fluxes, which densified the cast strongly. However, lowest modulus of rupture might be due to the brittleness of the glass formed and the existance of body flaws, as suggested by the existence of apparent porosity inspite of the highest fired density and an appreciable linear firing shrinkage.

However, these behaviours could have been influenced by the differences in the particle sizes, and their relative proportions, that made the respective clays. Beneficiation could have helped in eliminating or reducing some of these differences arising from sourcing. Beneficiation was not employed because the interest of the work was in intrinsic properties.

## 6.2 Behaviours of the Dry Pressed Electrical Porcelains

Green and fired properties, such as Shrinkages, densities, moduli of rupture, weight losses, apparent porosities and water absorptions of the developed porcelains are discussed in this section.

### 6.2.1 Green Properties

The green properties to be discussed are presented in Table 11 and 12, and Figures 18 - 21.

Green properties showed a higher bulk density and modulus of rupture at a higher pressing pressure than lower one. This was observed by comparing bodies made with the same composition, including the binding clay (Bida clay), but using pressing pressures of 10.01 MPa (102.07 Kg/Sq.Cm) and 22.53M Pa (229.67Kg/sq cm). However, employing a pressing pressure of 22.53MPa, but different binding clays namely, Bomo, Bukuru and Bida, showed higher bulk density and green modulus of rupture with Bida Clay. Bukuru clay gave a higher modulus of rupture relative to Bomo, but lower bulk density.

Substituting clay for either silica or feldspar gave higher bulk density and green modulus of rupture. However, substituting Silica for feldspar gave higher modulus of rupture but lower bulk density. Linear drying shrinkage was, virtually, non existent.

6.2.2 Fired Properties

The fired properties are presented in Tables 13-16 and Figures 22-27.

6.2.2.1 Effects of Firing Temperature

Linear firing shrinkage, generally, increases with increasing firing temperature but the increase was sharper in the range of 1220 - 1260°C. This was probably because most of the feldspar in the bodies melted within this range. Lower rate of increase of the linear firing shrinkage in firing temperatures lower than 1220°C could probably mean lower vitrification rate, and as a result more porous ware. Lower rate of increase in linear firing shrinkage in temperatures higher than 1260°C could mean vitrification, practically, has been completed at about 1260°C, and bodies fired much above this temperature could be over vitrified.

Firing weight loss was, generally, increasing with increase in firing temperature. Rate of increase was minimum within 1220 - 1260°C range. This is probably a stable vitrification zone. Ware fired below 1220°C could be very porous while those fired above 1260°C could contain more closed voids than those fired within the vicinity of 1260°C, and as a resuslt, might be less in bulk density than those fired at 1260°C.

Fired bulk density was found to be decreasing within 1180-1220°C firing temperature range, and increasing within 1220-1260°C. At firing temperatures greater than 1260°C, bulk density decreases with increasing firing temperature. However, body C showed an increasing trend in bulk density even at firing temperature greater than 1260°C.

Bodies 'B' and 'D' fired at 1260°C had bulk densities within the range of 2200 - 2400kg/cu m, which is required in the specifications for low tension porcelains. Body 'C' needed higher temperature, 1300°C, firing to get its bulk density within that range. However, body 'A', even at 1300°C, could not satisfy this requirement.

Fired modulus of rupture, generally increases with increase in firing temperature. However, differences, as regards the manner of increase existed with body composition. Body 'B' had maximum fired modulus of rupture at a firing temperature of 1260°C and minimum at 1220°C. While bodies 'A', 'C' and 'D' had their maximum at 1300°C with bodies 'C' and 'D' exhibiting a sag in the rate of increase at 1260°C. Specification requirements regarding fired modulus of rupture is 2.1 - 4.1 KN/sq.cm (21-41MPa). All the bodies used had their fired moduli of rupture within the range at all the firing temperatures used.

Apparent porosity, generally, decreased with increase in firing temperature. Within 1180 - 1220°C, bodies of composition 'A' and 'D' had their apparent porosities, almost, unaffected by firing temperature. While within 1220-1300°C their porosities, decreased sharply with body 'D', almost, attaining zero porosity at a firing temperature of 1260°C. However, body 'B' had its apparent porosity decreasing with increasing temperature, within 1180 - 1220°C, while body 'C' had its porosity increasing with increase in temperature over the same range. Within 1220-1300°C, bodies 'B' and 'C' also had their porosities decreasing with increase in firing temperature. All bodies virtually, had their apparent porosities reduced to zero at 1300°C.

Water absorption followed the same pattern as the apparent porosity. Going by the Stadards quoted in Table 27 of Appendix -F, only body 'D' satisfy the requirement of water absorption limits at a firing temperature within 1180-1220°C. At a temperature of 1260°C, bodies, 'B', 'C' and 'D' proved satisfactory. Though at 1300°C, all the bodies satisfy this requirement, body 'A' still is not satisfactory because it could not satisfy the bulk density requirement.

6.2.2.2 <u>Compositional Effects</u>

Effect of substituting feldspar for silica, clay being constant, was studied by comparing the properties of body'A' and body'B' fired to the same temperature. This substitution gave rise to higher linear firing shrinkage, except for samples fired at 1300°C, which showed no appreciable difference. Firing weight loss was essentially unchanged. Bulk fired density increased. At 1180°C and 1260°C, more feldspar resulted in higher fired modulus of rupture, while at 1220 and 1300°C MOR was lowered by this substitution. Apparent porosity and water absorption were lowered by having more feldspar and less silica.

Substituting feldspare for clay, silica being constant, resulted in higher linear firing shrinkage, lower firing weight loss, lower apparent porosity and water absorption. At 1180 - 1220°C, bulk density was enhanced. While at temperatures greater than 1220°C, this substitution lowered the bulk density. At 1180-1220°C bulk density increased, probably because of higher degree of vitrification due to more feldspar, while at higher temperatures, vitrification points of the bodies being compared are, essentially, the same and the effect of higher densities of clays relative to feldspar came in to play. This observations were drawn by comparison of body 'B' and body 'D's' properties.

The effect of replacing silica sand with clay, feldspar being constant, was studied by comparing bodies 'B' and 'C'. At 1180 - 1260°C, linear firing shrinkage was not significantly affected, and the insignificant change did not follow any definite direction. However, at 1300°C linear firing shrinkage increased by 0.5% as a result of this change possibly due to the presence of more fluxes in the clay than in the silica. Firing weight loss, generally, increased. Bulk fired density increased at 1180-1220 and 1300°C, while at 1260°C, this change lowered the bulk density. MOR was, almost, unaffected at 1180°C, whereas at 1220 and 1300°C, more clay increased it. At 1260°C, the change lowered MOR. Apparent porosity and water absorption were lowered all through.

# CHAPTER SEVEN

## 7. CONCLUSION

Based on the findings of this research, the following conclusion are drawn.

1. Kano KRD, KanKara, Cross river and Ikebedi clays are, most probably, Kaolins.

2. Bomo, Goronyo MWF1, and Bida clays are stone ware clays, probably.

3. Kaoje and BuKuru clays had behaviours in-between Kaolins and stone ware clays.

4. Plasticity indices showed the following order of decrease: Kaoje> BuKuru> Bomo> Goronyo MWF1> Bida> Ikebedi> Cross river> KanKara> Kano KRD.

5. Rheological studies showed the following order of increasing minimum viscosity: Cross river<Kaoje<BuKuru <Kano KRD< Goronyo MWF1< KanKara< Ikebedi<Bida<Bomo.

6. Bida and KanKara clays were adequate, in terms of plastic and binding abilities, for production of dry pressed electrical porcelains.

7. Body 'C' exhibited the most favourable green properties.

8. Pressing Pressure of 10MPa proved to be adequate enough, for dry pressing electrical porcelains, under the conditions used.

9. Firing temperature of 1260$^{o}$C proved to be the best firing temperature used.

10. Bodies 'B', 'C' and 'D' were very adequate for producing dry pressed electrical porcelains using Kankara and Bida clays, Okene feldspar and Danbatta Silica sand.

11. Body 'A' was not good enough due to high degree of water absorption in all the cases investigated, and its inability to satisfy the bulk fired density of low tension porcelains as specified in appendix F.

12. No good electrical porcelain was found using a firing temperature of 1180 or 1220$^{o}$C.

# CHAPTER EIGHT

## 8. RECOMMENDATION

Based on the experience with this research, the following recommendations are made.

1. Ikebedi, Cross river and Kaoje clays require a good beneficiation prior to being used.

2. The developed low-tension porcelain could be commercialised. Bodies 'B', 'C' and 'D' are recommended provided peak firing temperature is up 1280°C, and heat treatment time is just as enough as to satisfy the requirements of density, MOR, porosity and water absorption.

3. Dry pressing pressure should not be less than 10MPa and should be higher if moisture content is less than 14% wt.

# CHAPTER NINE

## 9. APPENDICES

### APPENDIX-A

TABLE A1: PFEFFERKORN PLASTICITY TEST RESULTS

| Clay Name | Moisture Content wt% | Pfefferkorn Height mm |
|---|---|---|
| Kaoje | 20.00 | 2.5 |
| | 25.00 | 10.5 |
| | 27.00 | 16.5 |
| | 29.00 | 24.0 |
| | 30.00 | 25.5 |
| Bida | 30.00 | 3.0 |
| | 35.00 | 9.0 |
| | 37.50 | 12.5 |
| | 40.00 | 16.0 |
| | 42.50 | 24.5 |
| Kano KRD | 35.00 | 2.5 |
| | 40.00 | 6.5 |
| | 45.00 | 13.0 |
| | 47.50 | 21.5 |
| | 50.00 | 24.0 |
| Cross river | 30.00 | 2.0 |
| | 35.00 | 5.5 |
| | 40.00 | 9.0 |
| | 45.00 | 15.5 |
| | 47.50 | 24.0 |

| Clay Name | Moisture Content wt% | Pfefferkorn Height mm |
|---|---|---|
| Goronyo MWF1 | 30.00 | 6.5 |
| | 31.08 | 11.5 |
| | 32.50 | 13.0 |
| | 34.00 | 17.0 |
| | 35.00 | 16.0 |
| White Kankara | 35.00 | 2.5 |
| | 40.00 | 5.0 |
| | 42.50 | 11.0 |
| | 45.00 | 21.0 |
| | 50.00 | 28.0 |
| Bukuru | 22.00 | 7.5 |
| | 25.00 | 11.5 |
| | 27.50 | 15.5 |
| | 28.50 | 20.5 |
| | 30.00 | 27.0 |
| Bomo | 30.00 | 9.5 |
| | 31.00 | 18.5 |
| | 32.00 | 22.5 |
| | 33.00 | 22.5 |
| | 35.00 | 26.0 |
| Ikebedi | 30.00 | 2.5 |
| | 35.00 | 6.0 |
| | 40.25 | 10.5 |
| | 45.00 | 18.5 |
| | 47.50 | 26.5 |

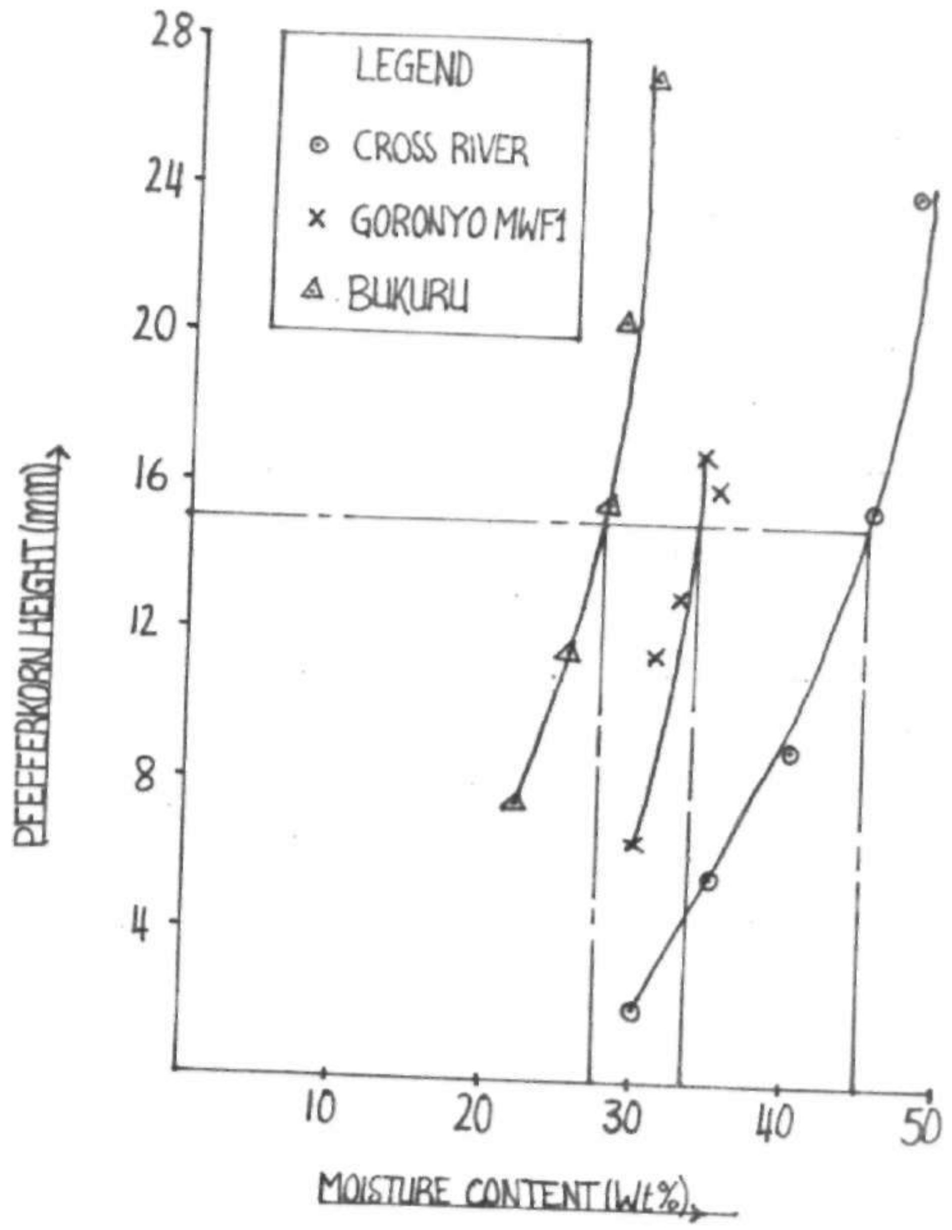


FIGURE A1: PLASTICITY TEST ON CROSS RIVER, GORONYO MWF1 AND BUKURU CLAYS

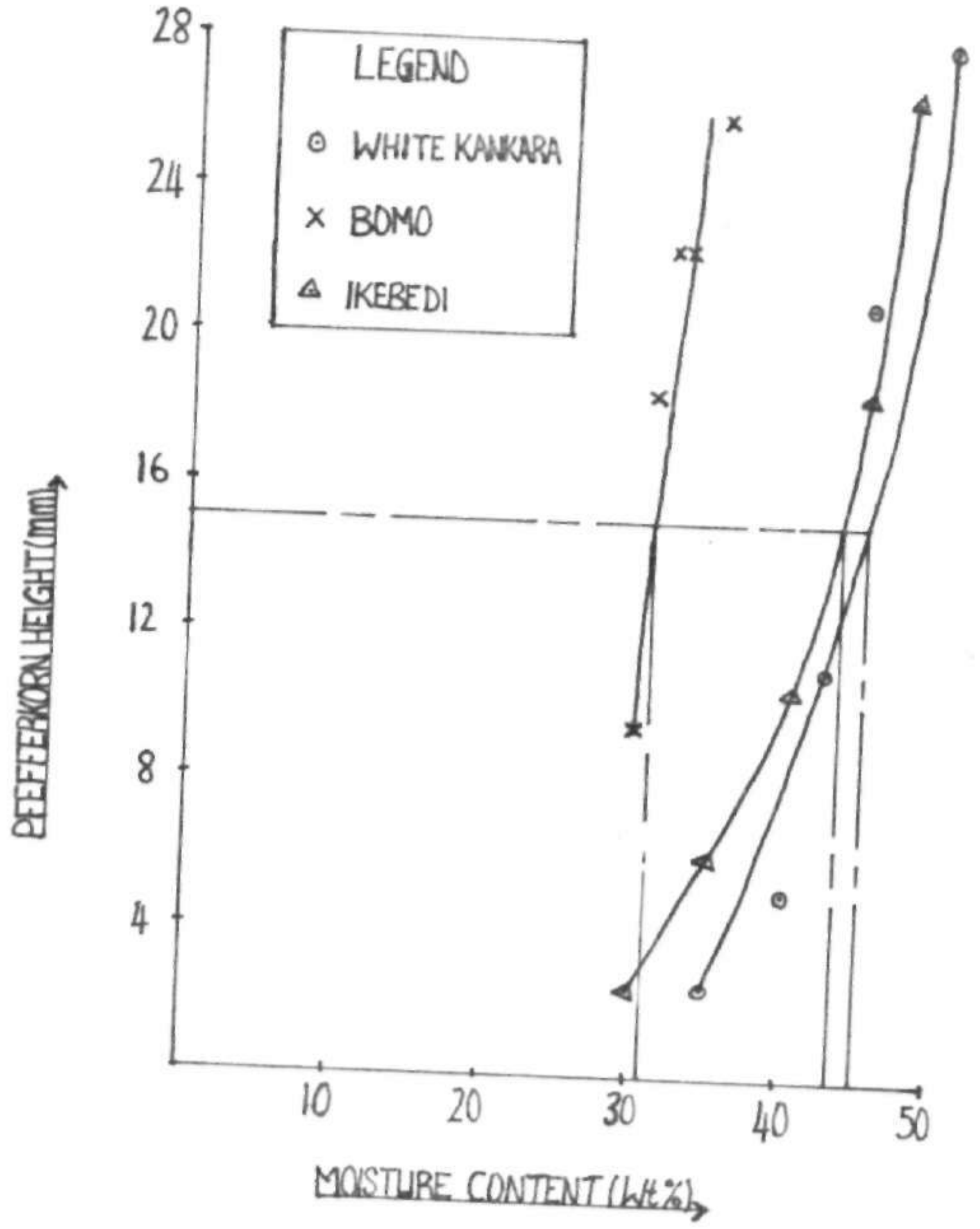


FIGURE A2: PLASTICITY TEST ON WHITE KANKARA, BOMO, AND IKEBEDI CLAYS

TABLE A2: CLAY SLIPS DEFLOCCULATION RESULTS

| Clay Type | Water Content wt% | Deflocculant Concentration wt% | Viscosity Cp |
|---|---|---|---|
| Kano KRD | 45 | 0.1 | 520.00 |
| | | 0.2 | 60.00 |
| | | 0.3 | 70.00 |
| | | 0.4 | 58.00 |
| | | 0.5 | 76.00 |
| Goronyo MWF1 | 50 | 0.1 | Too High |
| | | 0.2 | 570.00 |
| | | 0.3 | 72.00 |
| | | 0.4 | 75.00 |
| | | 0.5 | 97.00 |
| White Kankara | 45 | 0.1 | 130.00 |
| | | 0.2 | 95.00 |
| | | 0.3 | 90.00 |
| | | 0.4 | 95.00 |
| | | 0.5 | 110.00 |
| Bomo | 45 | 0.1 | 300.00 |
| | | 0.2 | 238.00 |
| | | 0.3 | 167.50 |
| | | 0.4 | 230.00 |
| | | 0.5 | 256.00 |

| Clay Type | Water Content wt% | Deflocculant Concentration wt% | Viscosity Cp |
|---|---|---|---|
| Ikebedi | 70 | 0.30 | 355.00 |
| | | 0.40 | 130.00 |
| | | 0.45 | 40.00 |
| | | 0.50 | 20.00 |
| | | 0.60 | 25.00 |
| Cross River | 70 | 0.1 | 534.00 |
| | | 0.2 | 356.00 |
| | | 0.3 | 200.00 |
| | | 0.4 | 20.00 |
| | | 0.5 | 12.50 |
| Bukuru | 45 | 0.1 | 100.00 |
| | | 0.2 | 60.00 |
| | | 0.3 | 56.00 |
| | | 0.4 | 55.00 |
| | | 0.5 | 57.50 |
| Bida | 65 | 0.1 | 227.50 |
| | | 0.2 | 205.00 |
| | | 0.3 | 189.00 |
| | | 0.4 | 218.00 |
| | | 0.5 | 350.00 |
| Kaoje | 40 | 0.1 | 155.00 |
| | | 0.2 | 36.00 |
| | | 0.3 | 38.00 |
| | | 0.4 | 38.00 |
| | | 0.5 | 48.00 |

TABLE A3: GREEN PROPERTIES OF THE CLAY'S SOLID CASTS

| Specimen Tag | Linear Drying Shrinkage (LDS) % | Green or Dried Density (GD) $Kg/m^3$ | Green Modulus of Rapture (GMOR) $KN/cm^2 x 10^{-3}$ |
|---|---|---|---|
| R1 | 1.96 | 1446.81 | - |
| R3 | 1.80 | 1493.55 | 78.00 |
| R4 | 1.91 | 1463.33 | - |
| R5 | 1.91 | 1452.61 | 68.17 |
| R6 | 1.61 | 1391.00 | 80.54 |
| MEAN | 1.84 | 1449.46 | 75.57 |
| F1 | 11.36 | 1779.88 | - |
| F2 | 11.74 | 1859.79 | 623.25 |
| F3 | 11.13 | 2062.72 | - |
| F4 | 11.61 | 2067.02 | - |
| F5 | 9.70 | 1966.41 | 807.25 |
| F6 | 11.32 | 1924.26 | 687.47 |
| MEAN | 11.14 | 1943.35 | 705.99 |

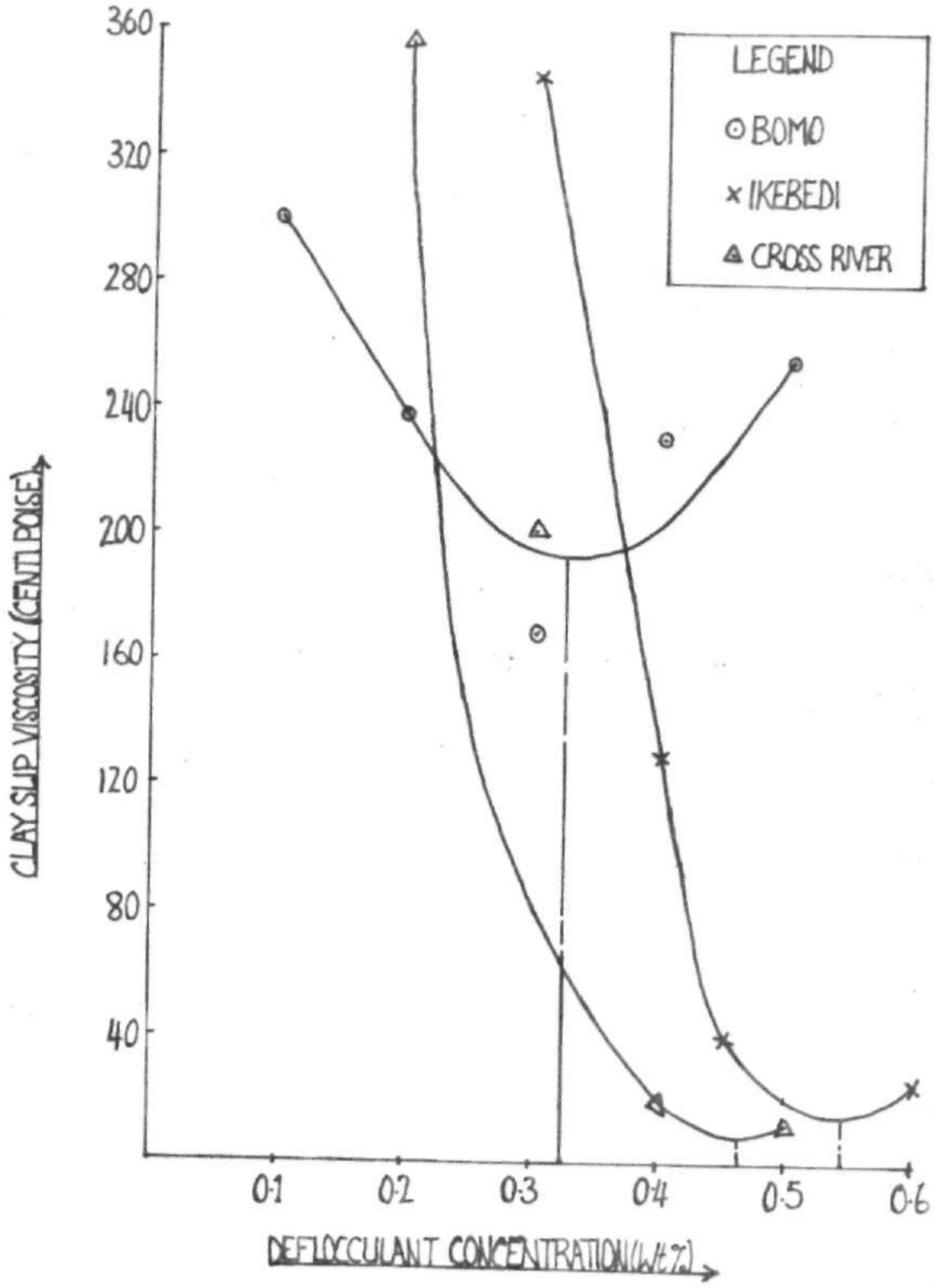


FIGURE A3: BOMO, IKEBEDI, AND CROSS RIVER CLAY SLIPS MINIMUM VISCOSITY DETERMINATION FOR SOLID CASTING

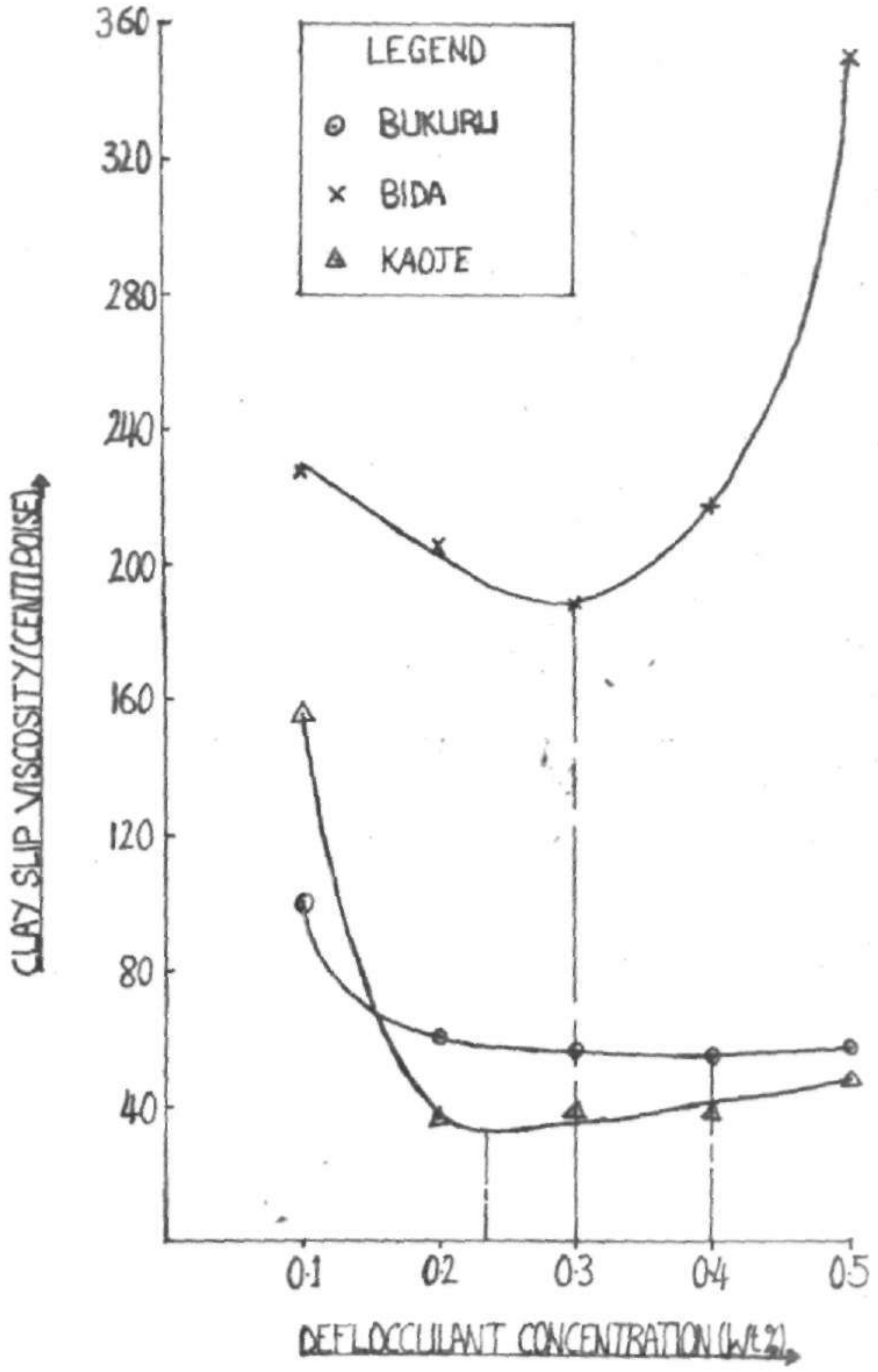


FIGURE A11: BUKURU, BIDA, AND KAOJE CLAY SLIPS MINIMUM VISCOSITY DETERMINATION FOR SOLID CASTING

| Specimen Tag | Linear Drying ShrinKage (LDS) % | Green or Dried Density (GD) $Kg/m^3$ | Green Modulus of Rapture (GMOR) $KN/cm^2 x 10^{-3}$ |
|---|---|---|---|
| KK1 | 3.22 | 1429.72 | 110.70 |
| KK2 | 2.96 | 1397.28 | 115.14 |
| KK3 | 2.48 | 1449.36 | 124.46 |
| KK4 | 1.78 | 1379.99 | - |
| KK5 | 2.83 | 1435.80 | - |
| KK6 | 2.65 | 1387.66 | - |
| MEAN | 2.65 | 1413.34 | 116.77 |
| BM1 | 9.09 | 1831.93 | 482.71 |
| BM2 | 9.78 | 1931.26 | 560.78 |
| BM3 | 9.26 | 1956.87 | - |
| BM4 | 10.04 | 1940.00 | - |
| BM5 | 8.74 | 1923.02 | - |
| BM6 | 9.57 | 1783.08 | 568.19 |
| MEAN | 9.41 | 1894.19 | 537.23 |
| IK1 | 6.96 | 1526.66 | 158.04 |
| IK4 | 5.65 | 1549.10 | - |
| IK5 | 7.00 | 1459.54 | - |
| MEAN | 6.54 | 1511.77 | 158.04 |

| Specimen Tag | Linear Drying Shrinkage (LDS) % | Green or Dried Density (GD) $Kg/m^3$ | Green Modulus of Rapture (GMOR) $KN/cm^2 x 10^{-3}$ |
|---|---|---|---|
| CR1 | 7.83 | 1611.26 | - |
| CR2 | 8.78 | 1735.82 | - |
| CR3 | 8.78 | 1604.07 | - |
| CR4 | 8.45 | 1746.64 | 227.19 |
| CR5 | 8.78 | 1659.23 | 157.82 |
| CR6 | 8.37 | 1630.26 | 186.34 |
| MEAN | 8.50 | 1664.55 | 190.45 |
| BD1 | 9.04 | 1814.67 | - |
| BD2 | 8.74 | 1828.73 | 215.49 |
| BD3 | 9.13 | 1734.48 | - |
| BD4 | 7.86 | 1868.72 | - |
| BD5 | 9.00 | 1806.65 | 250.28 |
| BD6 | 9.83 | 1792.19 | - |
| MEAN | 8.93 | 1807.57 | 232.89 |
| KJ1 | 0.60 | 1647.94 | - |
| KJ2 | 0.99 | 1642.18 | - |
| KJ3 | 0.09 | 1653.05 | 66.61 |
| KJ6 | 0.56 | 1651.78 | 70.04 |
| MEAN | 0.56 | 1648.74 | 68.33 |

| Specimen Tag | Linear Drying Shrinkage (LDS) % | Green or Dried Density (GD) Kg/m$^3$ | Green Modulus of Rapture (GMOR) KN/cm$^2$x10$^{-3}$ |
|---|---|---|---|
| BK1 | D | D | D |
| BK2 | D | D | D |
| BK3 | D | D | D |
| BK4 | D | D | D |
| BK5 | D | D | D |
| BK6 | D | D | D |
| MEAN | - | - | - |

TABLE A4: FIRED PROPERTIES OF THE CLAYS'S SOLID CASTS.

| Specimen TAG | Linear Firing Shirin-Kage(LFS) wt% | Firing Weight loss (FWL) wt% | Fired Density (FD) $Kg/m^3$ | Fired Modulus of Rap-ture(FMOR) $KN/cm^2$ | Apparent Porosity (AP) wt% | Water Absorption (WA) %wt |
|---|---|---|---|---|---|---|
| R1 | 10.07 | 13.16 | 1796.14 | 3.31 | 26.17 | 14.18 |
| R4 | 10.37 | 13.07 | 1805.31 | 3.93 | 26.66 | 14.57 |
| MEAN | 10.22 | 13.12 | 1800.73 | 3.62 | 26.42 | 14.38 |
| F1 | 2.83 | 6.33 | 2029.11 | 3.95 | 10.11 | 4.90 |
| F3 | 2.45 | 6.35 | 2172.62 | 4.76 | 10.78 | 5.23 |
| F4 | 2.61 | 6.37 | 2126.08 | 3.31 | 11.08 | 5.43 |
| MEAN | 2.63 | 6.35 | 2109.27 | 4.01 | 10.66 | 5.19 |
| KK4 | 12.00 | 12.88 | 1889.69 | 4.78 | 22.87 | 12.01 |
| KK5 | 11.77 | 12.86 | 1861.91 | 4.25 | 21.83 | 11.45 |
| KK6 | 11.52 | 12.92 | 1897.39 | 5.17 | 22.76 | 12.00 |
| MEAN | 11.76 | 12.89 | 1883.00 | 4.73 | 22.49 | 11.82 |
| BM3 | 4.98 | 5.58 | 2204.18 | 2.80 | 0.00 | 0.00 |
| BM4 | 4.30 | 4.83 | 2147.47 | 2.60 | 0.00 | 0.00 |
| BM5 | 5.00 | 5.09 | 2151.87 | 2.04 | 0.00 | 0.00 |
| MEAN | 4.76 | 5.17 | 2167.84 | 2.48 | 0.00 | 0.00 |

| Specimen TAG | Linear Firing Shirin-Kage(LFS) wt% | Firing Weight loss (FWL) wt% | Fired Density (FD) $Kg/m^3$ | Fired Modulus of Rap-ture(FMOR) $KN/cm^2$ | Apparent Porosity (AP) wt% | Water Absorption (WA) wt% |
|---|---|---|---|---|---|---|
| IK4 | D | D | D | D | D | D |
| IK5 | D | D | D | D | D | D |
| MEAN | - | - | - | - | - | - |
| CR1 | 9.43 | 9.45 | 2303.14 | 1.88 | 12.38 | 5.78 |
| CR2 | 10.01 | 9.39 | 2342.39 | 1.78 | 12.04 | 5.62 |
| CR3 | 10.49 | 9.42 | 2175.70 | 1.57 | 11.67 | 5.43 |
| MEAN | 9.98 | 9.42 | 2273.74 | 1.74 | 12.03 | 5.61 |
| BD1 | 7.41 | 9.22 | 2141.90 | 4.69 | 14.81 | 7.50 |
| BD3 | 7.61 | 9.23 | 2079.45 | 3.66 | 14.33 | 7.24 |
| BD6 | 7.62 | 9.17 | 1925.90 | 2.23 | 15.14 | 7.73 |
| MEAN | 7.55 | 9.21 | 2049.08 | 3.53 | 14.76 | 7.49 |
| KJ1 | 3.90 | 8.48 | 1757.60 | 3.38 | 28.67 | 16.14 |
| KJ2 | 4.18 | 8.42 | 1696.29 | 2.66 | 30.05 | 17.29 |
| MEAN | 4.04 | 8.45 | 1726.95 | 3.02 | 29.36 | 16.72 |

APPENDIX -B

TABLE B1: GREEN PROPERTIES OF TRIAL PRESSED WARES

| Specimen TAG | Pressing Pressure (TNS)/App-lication | Water Content wt% | Linear Drying Shrinkage (LDS) wt% | Green or Dried Density (GD) $Kg/m^3$ | Green Modulus of Rupture (GMDR) $KN/cm^2x10^{-3}$ |
|---|---|---|---|---|---|
| PHBMD1 | 4.5/1.5:4.5 | 15.00 | 0.00 | 1876.57 | 99.98 |
| PHBMD2 | 4.5/1.5:4.5 | 15.00 | 0.00 | 1899.22 | 75.67 |
| PHBMD3 | 4.5/1.5:4.5 | 15.00 | 0.00 | 1863.07 | - |
| MEAN | 4.5/1.5:4.5 | 15.00 | 0.00 | 1879.62 | 87.83 |
| PHBMD1 | 4.5/1.5:4.5 | 15.00 | 0.00 | 1843.77 | 71.21 |
| PHBMD2 | 4.5/1.5:4.5 | 15.00 | 0.00 | 1875.23 | 120.53 |
| PHBMD3 | 4.5/1.5:4.5 | 15.00 | 0.00 | 1864.82 | - |
| MEAN | 4.5/1.5:4.5 | 15.00 | 0.00 | 1861.27 | 95.87 |
| PHBMD1 | 4.5/1.5:4.5 | 15.00 | 0.00 | 1887.00 | 126.50 |
| PHBMD2 | 4.5/1.5:4.5 | 15.00 | 0.00 | 1877.90 | 97.10 |
| PHBMD3 | 4.5/1.5:4.5 | 15.00 | 0.00 | 1905.35 | - |
| MEAN | 4.5/1.5:4.5 | 15.00 | 0.00 | 1890.08 | 111.80 |
| PLBD1 | 2.0/SINGLE | 15.00 | 0.00 | 1806.01 | - |
| PLBD2 | 2.0/SINGLE | 15.00 | 0.00 | 1811.84 | 64.44 |
| PLBD3 | 2.0/SINGLE | 15.00 | 0.00 | 1805.79 | 62.80 |
| MEAN | 2.0/SINGLE | 15.00 | 0.00 | 1807.88 | 63.62 |

## APPENDIX - C

TABLE C1: GREEN PROPERTIES OF THE PRESSED PORCELAINS

| Specimen TAG | Linear Drying Shrinkage (LDS) % | Green Density (GD) $Kg/m^3$ | Green Modulus of Rupture (GMOR) $KN/cm^2 x 10^{-3}$ |
|---|---|---|---|
| A1 | 0.00 | 1770.35 | 56.77 |
| A2 | 0.00 | 1731.94 | 60.93 |
| A3 | 0.00 | 1740.54 | 66.95 |
| A4 | 0.00 | 1782.05 | 62.90 |
| MEAN | 0.00 | 1756.22 | 61.89 |
| B1 | 0.00 | 1811.53 | 57.10 |
| B2 | 0.00 | 1814.09 | 57.25 |
| B3 | 0.00 | 1824.41 | 60.09 |
| B4 | 0.00 | 1803.49 | 59.68 |
| MEAN | 0.00 | 1813.38 | 58.53 |
| C1 | 0.00 | 1818.53 | 62.66 |
| C2 | 0.00 | 1822.63 | 66.01 |
| C3 | 0.00 | 1816.95 | 66.76 |
| C4 | 0.00 | 1826.96 | 62.64 |
| MEAN | 0.00 | 1821.27 | 64.52 |
| D1 | 0.00 | 1815.20 | 54.84 |
| D2 | 0.00 | 1784.18 | 52.36 |
| D3 | 0.00 | 1808.93 | 47.04 |
| D4 | 0.00 | 1782.58 | 51.63 |
| MEAN | 0.00 | 1797.72 | 51.47 |

APPENDIX - D

TABLE D1: FIRED PROPERTIES OF PORCELAINS OF COMPOSITION "A"

| Firing Temper-ature FT(°C) | Specimen TAG | Linear Firing Shirin-kage (LFS) wt% | Firing Weight Loss (FWL) wt% | Fired Density (FD) $Kg/m^3$ | Fired Modulus Rapture (FMOR) $KN/cm^2$ | Apparent Porosity (Ap) wt% | Water Absorption (WA) wt% |
|---|---|---|---|---|---|---|---|
| | A1T1 | 7.96 | 6.05 | 2143.07 | 3.68 | 0.43 | 0.20 |
| | A2T1 | 8.16 | 6.03 | 2113.59 | 3.39 | 1.08 | 0.50 |
| 1300 | A3T1 | 8.26 | 6.70 | 2120.84 | 3.61 | 1.44 | 0.66 |
| | MEAN | 8.13 | 6.26 | 2125.83 | 3.56 | 0.98 | 0.45 |
| | A1T2 | 8.11 | 6.14 | 2223.54 | 3.04 | 5.80 | 2.67 |
| 1260 | A2T2 | 7.82 | 6.16 | 2122.57 | 2.87 | 7.07 | 3.28 |
| | A3T2 | 7.88 | 6.19 | 2146.22 | 2.85 | 7.88 | 3.67 |
| | MEAN | 7.94 | 6.16 | 2164.11 | 2.92 | 6.92 | 3.21 |
| | A1T3 | 6.89 | 6.07 | 2011.90 | 2.20 | 13.63 | 6.68 |
| 1220 | A2T3 | 6.94 | 6.13 | 2050.73 | 2.45 | 11.59 | 5.57 |
| | A3T3 | 6.61 | 6.16 | 2030.80 | 2.64 | 12.75 | 6.20 |
| | MEAN | 6.81 | 6.12 | 2031.44 | 2.43 | 12.66 | 6.15 |
| | A1T4 | 6.51 | 5.83 | 2052.22 | 2.19 | 13.35 | 6.46 |
| 1180 | A2T4 | 6.80 | 5.90 | 2070.71 | 2.70 | 12.09 | 5.82 |
| | A3T4 | 6.57 | 5.88 | 2042.51 | 2.25 | 12.78 | 6.18 |
| | MEAN | 6.63 | 5.87 | 2055.15 | 2.38 | 12.74 | 6.15 |

TABLE D2: FIRED PROPERTIES OF PORCELAINS OF COMPOSITION "B"

| Firing Temper-ature FT($^oC$) | Specimen TAG | Linear Firing Shirin-Kage (LFS) wt% | Firing Weight Loss (FWL) wt% | Fired Density (FD) $Kg/m^3$ | Fired Modulus Rapture (FMOR) $KN/cm^2$) | Apparent Porosity (Ap) wt% | Water Absorption (WA) wt% |
|---|---|---|---|---|---|---|---|
| | B1T1 | 8.08 | 6.20 | 2107.21 | 3.40 | 0.05 | 0.02 |
| 1300 | B2T1 | 8.17 | 6.30 | 2209.42 | 3.44 | 0.33 | 0.15 |
| | B3T1 | 8.08 | 6.65 | 2199.20 | 3.02 | 0.00 | 0.00 |
| | MEAN | 8.11 | 6.38 | 2171.94 | 3.29 | 0.13 | 0.06 |
| | B1T2 | 8.22 | 6.15 | 2235.40 | 3.45 | 4.85 | 2.21 |
| 1260 | B2T2 | 8.28 | 6.09 | 2230.84 | 3.39 | 4.81 | 2.16 |
| | B3T2 | 8.38 | 6.17 | 2249.01 | 3.55 | 5.08 | 2.32 |
| | MEAN | 8.29 | 6.14 | 2238.42 | 3.46 | 4.91 | 2.23 |
| | B1T3 | 6.95 | 6.31 | 2138.42 | 2.34 | 10.88 | 5.18 |
| 1220 | B2T3 | 7.20 | 6.16 | 2129.48 | 2.70 | 10.23 | 4.81 |
| | B3T3 | 7.35 | 6.14 | 2130.70 | 1.86 | 7.25 | 3.33 |
| | MEAN | 7.17 | 6.20 | 2132.83 | 2.30 | 9.45 | 4.23 |
| | B1T4 | 7.15 | 5.89 | 2128.32 | 2.10 | 10.90 | 5.13 |
| 1180 | B2T4 | 7.10 | 5.87 | 2145.57 | 3.04 | 11.44 | 5.41 |
| | B3T4 | 7.20 | 5.91 | 2162.80 | 2.67 | 11.25 | 5.33 |
| | MEAN | 7.15 | 5.89 | 2145.56 | 2.60 | 11.20 | 5.29 |

TABLE D3: FIRED PROPERTIES OF PORCELAINS OF COMPOSITION "C"

| Firing Temper-ature FT(°C) | Specimen TAG | Linear Firing Shirin-kage (LFS) wt% | Firing Weight Loss (FWL) wt% | Fired Density (FD)( Kg/m³ | Fired Modulus Rapture (FMOR) KN/cm² | Apparent Porosity (Ap) wt% | Water Absorption (WA) wt% |
|---|---|---|---|---|---|---|---|
| | C1T1 | 8.42 | 6.75 | 2238.92 | 3.17 | 0.00 | 0.00 |
| 1300 | C2T1 | 8.52 | 6.84 | 2239.75 | 3.51 | 0.00 | 0.00 |
| | C3T1 | 8.97 | 6.87 | 2214.89 | 3.62 | 0.00 | 0.00 |
| | MEAN | 8.64 | 6.82 | 2231.19 | 3.43 | 0.00 | 0.00 |
| | C1T2 | 8.65 | 6.75 | 2264.66 | 2.26 | 2.27 | 1.02 |
| 1260 | C2T2 | 8.17 | 6.80 | 2143.68 | 2.89 | 2.87 | 1.30 |
| | C3T2 | 7.93 | 6.75 | 2147.51 | 3.74 | 2.31 | 1.04 |
| | MEAN | 8.25 | 6.77 | 2185.28 | 2.96 | 2.48 | 1.12 |
| | C1T3 | 7.30 | 6.71 | 2170.04 | 3.40 | 8.11 | 3.80 |
| 1220 | C2T3 | 6.65 | 6.67 | 2086.05 | 2.96 | 8.79 | 4.14 |
| | C3T3 | 7.20 | 6.67 | 2178.62 | 3.56 | 10.52 | 4.96 |
| | MEAN | 7.05 | 6.68 | 2144.90 | 3.31 | 9.14 | 4.30 |
| | C1T4 | 7.29 | 6.55 | 2118.25 | 2.85 | 7.05 | 3.22 |
| 1180 | C2T4 | 7.15 | 6.56 | 2172.42 | 2.80 | 6.91 | 3.13 |
| | C3T4 | 7.30 | 6.57 | 2168.05 | 2.13 | 6.40 | 2.97 |
| | MEAN | 7.25 | 6.56 | 2152.91 | 2.59 | 6.79 | 3.11 |

TABLE D4: FIRED PROPERTIES OF PORCELAINS OF COMPOSITION "D"

| Firing Temperature FT(°C) | Specimen TAG | Linear Firing Shirinkage (LFS) wt% | Firing Weight Loss (FWL) wt% | Fired Density (FD) Kg/m$^3$ | Fired Modulus Rapture (FMOR) KN/cm$^2$ | Apparent Porosity (Ap) wt% | Water Absorption (WA) wt% |
|---|---|---|---|---|---|---|---|
| | D1T1 | 9.78 | 5.55 | 2082.79 | 3.72 | 0.00 | 0.00 |
| 1300 | D2T1 | 8.88 | 5.60 | 2049.81 | 3.65 | 0.00 | 0.00 |
| | D3T1 | 6.57 | 5.57 | 1967.71 | 3.56 | 0.00 | 0.00 |
| | MEAN | 8.41 | 5.57 | 2033.44 | 3.64 | 0.00 | 0.00 |
| | D1T2 | 7.88 | 5.45 | 2176.70 | 2.95 | 0.10 | 0.06 |
| 1260 | D2T2 | 8.56 | 5.40 | 2216.26 | 3.70 | 0.10 | 0.04 |
| | D3T2 | 8.48 | 5.37 | 2210.72 | 3.39 | 0.05 | 0.02 |
| | MEAN | 8.31 | 5.41 | 2201.23 | 3.35 | 0.08 | 0.04 |
| | D1T3 | 7.48 | 5.39 | 2060.73 | 3.16 | 4.74 | 2.18 |
| 1220 | D2T3 | 7.54 | 5.35 | 2202.26 | 3.94 | 4.88 | 2.25 |
| | D3T3 | 7.54 | 5.16 | 2166.22 | 3.22 | 5.19 | 2.40 |
| | MEAN | 7.52 | 5.30 | 2143.07 | 3.44 | 4.94 | 2.28 |
| | D1T4 | 7.44 | 5.26 | 2163.93 | 2.89 | 4.87 | 2.24 |
| 1180 | D2T4 | 7.35 | 5.29 | 2150.36 | 2.97 | 4.70 | 2.15 |
| | D3T4 | 7.39 | 5.30 | 2156.89 | 3.31 | 5.13 | 2.36 |
| | MEAN | 7.39 | 5.28 | 2157.06 | 3.06 | 4.90 | 2.25 |

## APPENDIX - E

## CALCULATIONS

a) Recipe:

Basis 1kg of Recipe

i) Composition A had 45% clay, 25 feldspar, and 30% silica sand

Amounts of Respective Constituents:

| | Clay | Feldspar | Sand |
|---|---|---|---|
| Kankara | Bida | Okene | Dambatta |
| 35/100 x 1000g | 10/100 x 1000g | 25/100 x 1000g | 30/100 x 1000g |
| 350g | 100g | 250g | 300g |

Moisture content was 15% allthrough

:: Amount of water required for 1kg of material =

15/100 x 1000 = 150g

Similarly for:

ii) Composition B had 45% clay, 30% feldspar, and 25% silica sand

| Kankara Clay | Bida Clay | Feldspar | Sand |
|---|---|---|---|
| 350g | 100g | 300g | 250g |

Water = 150g

iii) Composition C had 50% Clay, 30% feldspar, and 20% Silica sand.

| Kankara Clay | Bida Clay | Feldspar | Sand |
|---|---|---|---|
| 400g | 100g | 300g | 200g |

Water = 150g.

iv) Composition D had 40% Clay, 35% feldspar, and 25% Silica sand

| Kankara Clay | Bida Clay | Feldspar | Sand |
|---|---|---|---|
| 300g | 100g | 350g | 250g |

Water = 150g

b) <u>Viscosity of Slip</u>

Viscosity calculations were done in the following Manner;

Viscosity = Viscometer's Dial Reading multiplied by F Cp

Where F is a conversion factor which is a function of viscometer model, Spindle Number, and rotational speed at which the dial reading was noted. Spindle Number 3, and Viscometer model RVT were used through out this work.

c) <u>Green and Fired Properties</u>

i) LDS = $(L_i - L_d) \times 100/L_i$ %

ii) LFS = $(L_d - L_f) \times 100/L_d$ %

iii) GD = $W_d \times 7/22 \times 4/(d' \times d') \times 1/L_d$ valid for cylinderical samples.

iv) GD = $W_d/(L_d \times Bd \times H_d)$ valid for rectangular samples

v) FD = $W^f \times 1000/(W_{sat} - W_s)$

vi) FWL = $(W_d - W_F) \times 100/W_d$

vii) AP = $(W_{sat} - W_F) \times 100/(W_{sat} - W_s)$ %

Viii) WA = $(W_{sat} - W_F) \times 100/W_F$ %

ix) GMOR = $8 \times 7 \times F' \times S"/(22 \times dd \times dd \times dd)$ valid for cylinderical samples.

x) GMOR = $3 \times F' \times S"$ $(2 \times Bd \times Hd \times Hd)$ valid for Rectangular samples

xi) FMOR = $8 \times 7 \times F' \times S"$ $(22 \times dF \times dF \times dF)$ valid for cylinderical samples

xii) FMOR = $3 \times F' \times S"$ $/(2 \times BF \times HF \times HF)$ Valid for Rectangular Samples.

## APPENDIX -F

TABLE F1: STANDARDS FOR LOW - TENSION PORCELAIN INSULATORS AS QUOTED BY THURNAUR(1954)and Usman (1985)

| Properties | | Range of Values |
|---|---|---|
| Fired Bulk Density | ($x1000 Kg/m^3$) | 1.9-2.48 2.2-2.4[1] |
| Water Absorption | (%) | 0.2-2.6 0.5-2.0 |
| Coef. of Linear Expansion: | | |
| Value/°C x $10^{-6}$ | | 2.5-5.5 |
| Thermal Conductivity | (C.g.s) | 0.002 - 0.0039 |
| Tensile Strength | (Kg/sq.Cm) | 105 - 422 |
| Compressive Strength | (Kg.sq.Cm) | 2109 - 4570 |
| Modulus of Rapture | (Mpa) | 20.69 - 58.64 24-41[1] |
| Thermal shock | (°C) | 370-840 |
| Dielectric Constant | (V/mm) | 3540-7870 |
| Resistivity | (Ohm/ccm) | 1.8 - 18 |

N.B Values with a superscript 1 were Ranges of values given by Thurnaur (1954) . Other values were given by Usman (1985).

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