

# Development of Low Clay Whiteware Bodies

A THESIS IN THE PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE  
DEGREE OF BACHELOR OF TECHNOLOGY

BY

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## **CERTIFICATE**

This is to certify that the thesis entitled, "Development of Low Clay Whiteware Bodies" submitted by **Mr. Arun Sawaiyan (Roll No. 111CR0110)** in partial fulfillment of requirements for the award of Bachelor in Technology in Ceramic Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the results presented in this thesis has not submitted to any other University or Institute for the award of any other certificate or degree.

DATE-

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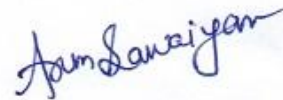
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## **ABSTRACT**

In this project, triaxial body was prepared using clay quartz and feldspar. In the traditional triaxial body the percentage of clay is generally taken within 40- 60 weight percentage. In the present work the percentage of clay has been kept 25 weight percentage and quartz amount has been fixed to 50 weight percentage. Two different types of clays have been used for this present work.. All samples are prepared by uniaxial pressing method, after pressing the samples are sintered at different temperatures at 1350<sup>0</sup>c and 1450<sup>0</sup>c with holding time of 2 hours. After sintering linear shrinkage was observed. To study the sintering behavior by dilatometry analysis has been carried out. After sintering the bulk density and apparent porosity was measured by Archimedian principle. Sintered body are grounded to powder and phase identification was analyzed by X-ray diffraction analysis. For mechanical property analysis , Brazilian Disk test has been carried out.is measured.

# **CHAPTER 1**

## **INTRODUCTION**



## **Introduction:**

Triaxial body was combination of clay, feldspar and quartz. The major compositions of raw materials taken for manufactures of white wares ceramics are: China clays, Ball clays, Potash feldspar, Quartz.

Specification of raw materials:

China clays: China clays have relatively large particles, medium plasticity and dry shrinkage, poor dry strength but white fired color. China clays of kaolin grade, but of relatively plastic type, are the preferred choice of in porcelain manufacture. Crude clays after washing should essentially consist of the mineral kaolinite ( $\text{Al}_2\text{O}_3, 2\text{SiO}_2, 2\text{H}_2\text{O}$ ) having a theoretical composition of 39.8%  $\text{Al}_2\text{O}_3$ , 46.3%  $\text{SiO}_2$  and 13.9%  $\text{H}_2\text{O}$ .

Ball clays: These are secondary clays. They impart high strength and good workability to whiteware bodies. The primary mineral phase present in clays is kaolinite. Micaceous minerals and quartz are the dominant minerals present as impurities along with minor amounts of feldspar, chlorite, titanite, hematite etc. plastic/ ball clays have fine particles, high plasticity and dry shrinkage, high green strength and contain large amounts of impurities which make body buff burning, low maturity and less translucent. According to the particles desired in body, a balance is struck by using minimum amount of ball clays, which is essentially required to develop plasticity and dry strength in body the remaining part being white burning but non plastic china clay.

Quartz:  $\text{SiO}_2$  in quartz mineral is major composition. It is widely used because it is expansive, hard, chemically stable and relatively infusible. Silica in the form of quartz is another important constituent in a whiteware composition. It does not react at low temperature and at high temperature dissolves partially to form a viscous liquid. Very finely milled quartz, accelerates the dissolution and thus improve the translucency. Special type of quartzite with their ability to invert easily are of advantages in porcelain bodies compared to quartz, quartz sand and glass sand. Another major source of silica is sand stone which consist of lightly bonded quartz grains.

Feldspar: Feldspar is an anhydrous alumina-silicate contain  $\text{K}^+$ ,  $\text{Na}^+$ , or  $\text{Ca}^+$  as a flux which aids in formation of a glassy phase. The major materials of commercial interests are potash feldspar (microcline or orthoclase),  $\text{K}_2\text{O}$ ,  $\text{Al}_2\text{O}_3$ ,  $6\text{SiO}_2$ . The feldspar acts as a flux, lower the vitrification temperature .

Effects of impurities in raw materials on the properties of whiteware products:

Free silica/ free quartz: The presence of free silica/ free quartz is responsible for reduced plasticity and shrinkage, tendency to warp, tendency to crack at the inversion temperature of quartz, reduce tensile and compressive strength, increase in porosity and reduction of the thermal shock resistance of clay body. Detrimental influence of free quartz is less pronounced when the particles are fine.

Alkalis: The presence of alkalis in clay is responsible for producing scum on the surface of the body during drying and for increased fusibility of the clay body.

Organic impurities: The presence of organics in excess of 5% in clays is harmful. These are responsible for change in color before and after firing; increased porosity, water absorption and shrinkage after firing of the body. The most serious deleterious effect is the strong reducing action on the oxides of iron and formation fused slag or black core.

Lime and Magnesia: CaO and MgO bearing compounds and alkalis react with free silica to form low melting compounds which lower the maturing temperature as well as the vitrification range of the clay body.

Iron Compounds: These are invariably present in all the clays and other raw materials. They lower the maturing temperature and also impair the color of the fired products.

Titania: It is present in clays and other raw materials as rutile ( $\text{TiO}_2$ ) or calcium titanate ( $\text{CaTiO}_3$ ) and they act as powerful fluxes. The fired color due to iron impurities deepens in the presence of titanic.

Importance of low clay whiteware bodies:

- A capacity to shape into complex shape.
- This features demonstration together to lessen bending of whiteware created by firing
- To continue coloring oxides for whiteness at low level.
- Gives low conductivity of electricity.
- To remove the deleterious effect of clay platelet.

# **CHAPTER 2**

# **LITERATURE REVIEW**

## **2. Literature Review:**

Whiteware ceramics are final bodies which are highly dense after the firing. The body basically contain translucent, white, high dense and fine texture.

Low amount of clay whiteware bodies contain 88 % of whiteware which is non plastic pre fired material and contain 12 % of clay, which are different from conventional whiteware..

The low clay whiteware was bearing major phase which is anorthite and minor phase which is glass and mullite.

There are three main type of translucent whiteware bodies as follow:

**Bone china** is a combination of china clay, feldspar and calcined animal bone .weight percentage of china clay is 25%, feldspar is 25% and calcined animal bone is 50% [1-4]. The combination is being mixture. After mixing, it was pressed by pressing machine to get green strength of bodies. Now this green bodies are fired at  $\sim 1230^{\circ}\text{C}$  to obtain the fully densified body and then it is glazed and fired around  $1100^{\circ}\text{C}$ . Bone china is  $\sim 70\%$  bearing strength  $\sim 100\text{MPa}$  which is very high.

**Fine translucent china** is a mixture of feldspar, quartz and kaolin. Weight percentage of feldspar is (25 to 40) %, quartz is (30 to 50) % and kaolin is (30 to 40) % [5]. To obtain the translucency, the amount of feldspar and quartz must be high and carry lesser clay. The combination are to be mixed. After mixing, it was pressed by pressing machine to get green strength of bodies .Fine translucent china is soft porcelain, this green bodies are fired around  $1235^{\circ}\text{C}$  and glost fired at  $1160^{\circ}\text{C}$ . bone china contain highest amount of toughness & strength as compared to fine translucent china having a glassy phase is less and it can be scratched glaze. Bone china is also contain scratched glaze.

**Hard porcelain** basically consists of kaolin, quartz and feldspar. Weight percentage of quartz is 25%, feldspar is 25% and kaolin is 50%[6-9]. The combination is being mixed. After mixing, it was pressed by pressing machine to get green strength of bodies Now this green bodies are biscuit

fired at about 1000°C, then it applied to glaze and glaze fired at around 1400°C. Hard porcelain is having low value of strength, hence it has high value of glassy phase (~70%) [10].

## **2.1 Development and design of whiteware ceramics, following aim should be considered :**

- 1) Whiteware must be fired at high temperature to get the densification.
- 2) In the whiteware, the glaze are used. This glaze need to be having high value of silica, so that it can minimize the scratch.
- 3) After glazing the whiteware, the bodies must be fired at high temperature of 1350°C .and the coefficient of thermal expansion must be low.
- 4) Low thermal expansion coefficient in whiteware are the result of good glazing fitting and having high resistance to thermal shock.
- 5) Low amount of glassy phase, during glaze firing whiteware will gives the low pyroclastic deformation and inhibits the crack.
- 6) For high translucency, the phase of fired bodies, whose refractive indices nearly equal to glass phase. And residual porosity obtained less.

## **2.2 Fabrication technique of typical whiteware body :**

### **Reaction during firing of whiteware body:**

Upto 200°C ⇒ physical adsorbed water will be going out.

450°C - 550°C ⇒ clay losses its chemical bound water or clay decompose will take place. And then Meta kaolin formation take place.

450°C - 550°C



At 573°C ⇒ alpha-quartz is converted to its high temperature form, Beta- quartz accompanied by about 2% volume expansion.

573°C



At 970°C ⇒ Silica is liberated and spinel ( $2\text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$ ) like phase formation take place from meta-kaolin.

970°C



At 985°C ⇒ first liquid phase formation in the system, which the first eutectic temperature in the system between the reaction of potash feldspar and silica. Feldspar first react with silica eliminated from kaolinite during thermal decomposition where silica will be released from clay.

With increase in temperature, more and more liquid phase will be forming. At the firing temperature of 1300°C, almost all feldspar will be in the liquid state. This liquid will progressively dissolve  $\text{SiO}_2$  from quartz,  $\text{Al}_2\text{O}_3$  and silica from clay decomposed product, as well as liquid will precipitate out mullite.

Some mullite will also formed from spinel type decomposed product clay.

At 1100°C ⇒ mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) formation take place.

At 1300°C - 1400°C ⇒ most of feldspar is liquid. During this (25 – 30) vol % liquid is present, which is very high to conventional liquid phase sintering. Hence it is termed as vitrification.

## **2.3 It can be concluded that :**

- It is studied that toughness and flexural strength of material depends upon function of porosity. Porosity increase, the fracture toughness and strength is getting reduced.
- Increase in fracture toughness is associated to increasing the surface area with decreasing size of flow.
- And increase in flexural strength is associated to increasing in fracture toughness with decreasing porosity.



# Chapter 3

## Objective of the project

- 1) The aim of the project is to study the development of low clay whiteware bodies
- 2) Study of variation in bulk density of four composition with increase in temperature.
- 3) Study of variation in apparent porosity of four composition with increase in temperature.
- 4) Study of variation in linear shrinkage of four composition with increase in temperature.
- 5) Study of variation tensile strength of four composition with increase in temperature.
- 6) Study the dilatometry analysis and phase analysis by X-Ray Diffraction.

# **Chapter 4**

## **EXPERIMENTAL**

## **4. Experimental:**

### **4.1. Batch preparation:**

For the development of low clay whiteware bodies, we took the four different composition.

First composition was china clay (25 wt. %)-feldspar (25wt%)-quartz (50wt %), second composition was ball and china clay (25 wt. %)-feldspar (25 wt.%)-quartz (50 wt. %), third composition was ball and china clay (50 wt. %)-feldspar (25wt%)-quartz (25wt %) and fourth composition was china clay (50 wt. %)-feldspar (25 wt. %)-quartz (25 wt. %)

### **4.2. Batch calculation:**

Raw materials required,

Quartz

China clay

Ball clay

Feldspar

Total four batch composition have taken.

Table 1 (batch weight 200 gm.)

Raw materials	Weight %	Weight (gm. )
feldspar	25	50
quartz	50	100
China clay	25	50

Table 2 (batch weight 200 gm.)

Raw materials	Weight %	Weight (gm.)
feldspar	25	50
quartz	50	100
China clay & Ball clay	25	50

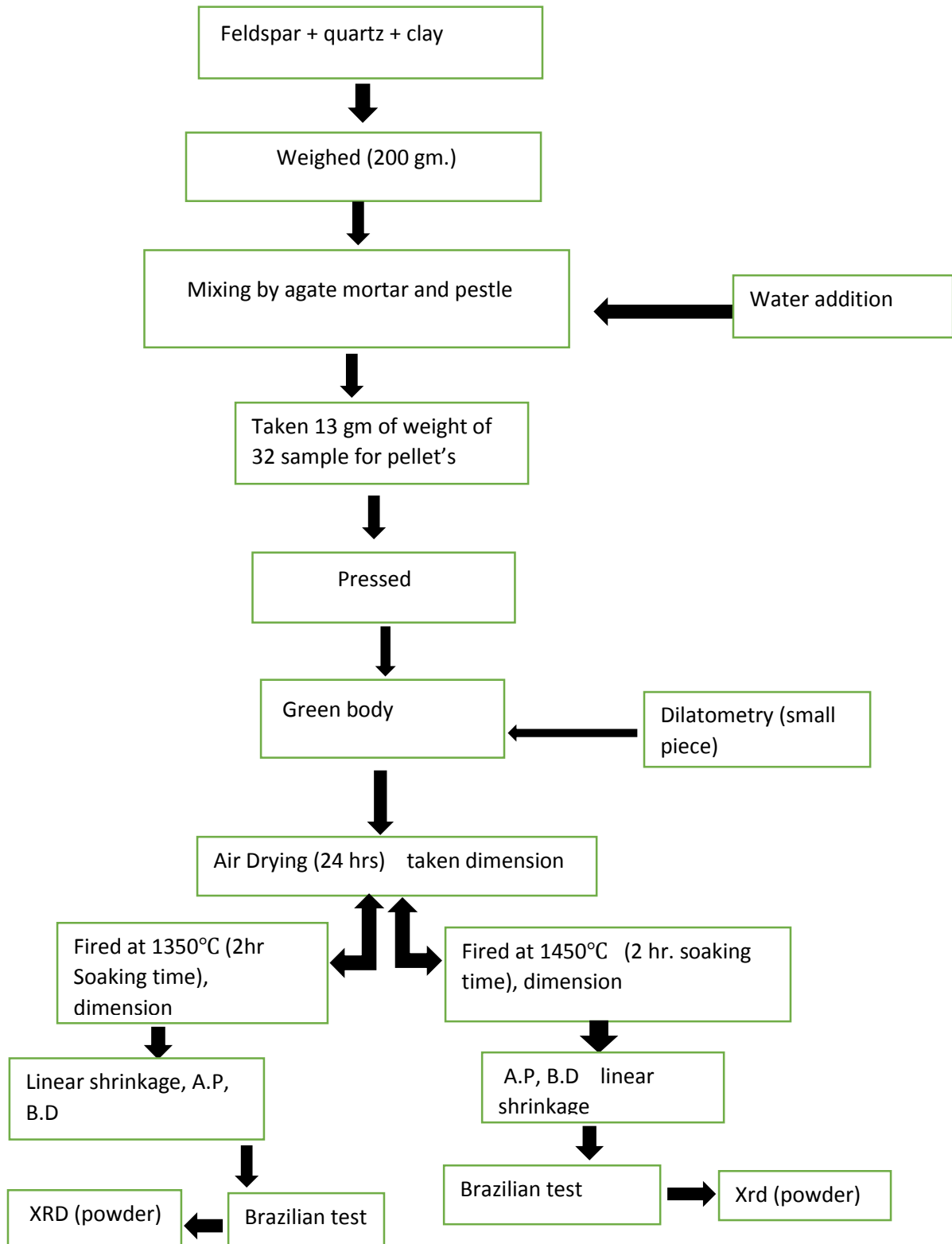
Table 3 (batch weight 200 gm.)

Raw materials	Weight %	Weight (gm.)
feldspar	25	50
quartz	25	50
China clay & Ball clay	50	100

Table 4 (batch weight 200 gm.)

Raw materials	Weight %	Weight (gm.)
feldspar	25	50
quartz	25	50
China clay	50	100

### 4.3 Flow chart :



#### 4.4 procedure:

- Ist we have separately taken the weight of china clay, ball clay, feldspar and quartz by weighing machine according to batch composition.
- The raw materials contain some agglomerates. Hence separately done the sieving process. In order to get fine size of particles.
- Now the materials are fully fine size of particles, hence all materials are mixed in agate mortar and pestle, added small amount of water and properly.
- All the materials are mixed properly for good densification of circular pellets.
- Mixing should be carried out till it should not contain any agglomerates.
- After mixing, we have weight 13 gm. of each sample from 200 gm. batch composition for preparation of pellet's.
- Prepared 32 sample of circular pellet's with load of 6 ton and dwell time 90 second.
- For pressing purpose, the mold will required. The name of mold is cylindrical mold die for preparation of pellet's.
- For cleaning purpose, acetone will be required.
- For lubrication purpose, steric acid will be used.
- After cleaning the die, 13 gm. of mixture was put into the cylindrical mold and kept inside the pressing machine. And it was pressed.
- After preparation of sample, it was kept in dry oven for air drying for 24 hours, where all the moisture will be removed.
- Then the dimension of green body sample was noted.
- After the drying process, 4 sample of pellets has taken from each composition for firing at temperature of 1350°C .
- The firing was done at 500°C for 2 hours soaking time. And then it was next fired at **1350°C for 2 hours soaking time**, in both the cases the heating rate was 2°C / minute.
- Total 8 samples of pellet's has taken from two composition ( ball clay & china clay= 25 %) and ( china clay = 25%) for firing at temperature of 1450°C.
- The firing was done at 500°C for 2 hours soaking time. And then it was next fired at **1450°C for 2 hours soaking time**, in both the cases the heating rate was 2°C / minute.
- Then the dimension of fired body was noted.

- From there the value of **linear shrinkage** was calculated. By the formula of

$$\text{Linear shrinkage} = (\text{Dia}_{\text{dry}} - \text{Dia}_{\text{fired}}) / \text{Dia}_{\text{dry}} * 100$$

Where,  $\text{Dia}_{\text{dry}}$  = diameter of dried body,  $\text{Dia}_{\text{fired}}$  = diameter of fired body

- The testing method for determination of **apparent porosity (A.P) and bulk density (B.D)** of circular pellet's and tiles. The density of pellets was done by Archimedes principle.
- Firstly we have measured the **dry weight (D)** of fired body samples.
- Then placed the sample in water and boil for **45 minutes**. During the boiling period, keep them entirely covered with water, and allow no contact with the heated bottom of the container.
- After the boiling period, cool the test samples to room temperature while still completely covered with water. After boiling keep the samples immersed in water for a minimum of half hours until the sample reach to room temperature.
- For measurement of **suspended weight (i)**, Determine the weight, S, of each test samples after boiling and while suspended in water in grams.
- The weight has done by suspending the samples in a halter of copper wire hung from one arm of the balance. The balance shall be previously counter-balanced with the wire in place and immersed in water to the same depth as is used when the pellets are in placed.
- For measurement of **Saturated Weight (S)**, after determining the suspended weight, blot each samples lightly with a wet cotton cloth to remove all drops of water from the surface and determine the saturated weight (S) in grams by weighing in air.
- From dry weight , suspended weight and saturated weight , the value of apparent porosity and bulk density was measured, by the formula as given below

$$\text{A.P} = (\text{S} - \text{D}) / (\text{S} - \text{i}) * 100 \quad ; \quad \text{B.D} = \text{D} / (\text{S} - \text{i})$$

Where, S= saturated weight    D= dry weight    i = suspended weight



- **Brazilian Test** is a test for obtaining the tensile strength. A disc circular pellet sample is carried by two strip loads at the disc periphery. The load has given to samples until sample will failure. The loading rate 20 to 50 kN/min. At the failure, the tensile strength of samples has been measured.
- **Dilatometry analysis** is the test to identify the sintering stages of whiteware ceramics. And to measure the shrinkage of whiteware bodies. The heating profile consisted of initial heating from **room temperature (RT) to 1250 °C at 10°C/min in presence of oxygen atmosphere.**
- After observing the Brazilian test, the finer powder particles from the pellets were taken. These fine powders were grounded using mortar and pestle. Then they were analyzed for constituent phases by **X-ray Powder Diffraction** method.
- The position ( $^{0}2\theta$ ) range in which the analysis was done, **5<sup>0</sup>-60<sup>0</sup>** and the rate was **15<sup>0</sup>/min.** The intensity vs position data was obtained for 6 samples. The data was analyzed using **X'Pert HighScore** and phases were identified by tallying them with **JCPDs** file data.

**CHAPTER 5**

**RESULTS &**

**DISCUSSIONS**

**5.1 Bulk Density (BD) (in g.cm<sup>-3</sup>):** the density of pellets was obtained by Archimedes principle. Where we measured the dry weight, suspended weight and soaked weight.

Table NO: (5) bulk density vs temperature

Serial No	Temperature	C(25 wt%)	BC(25wt%)	BC(50wt%)	C(50wt%)
1	1350 <sup>o</sup> c	2.11	2.15	3.31	2.16
2	1450 <sup>o</sup> c	1.72	1.71	1.7	1.71

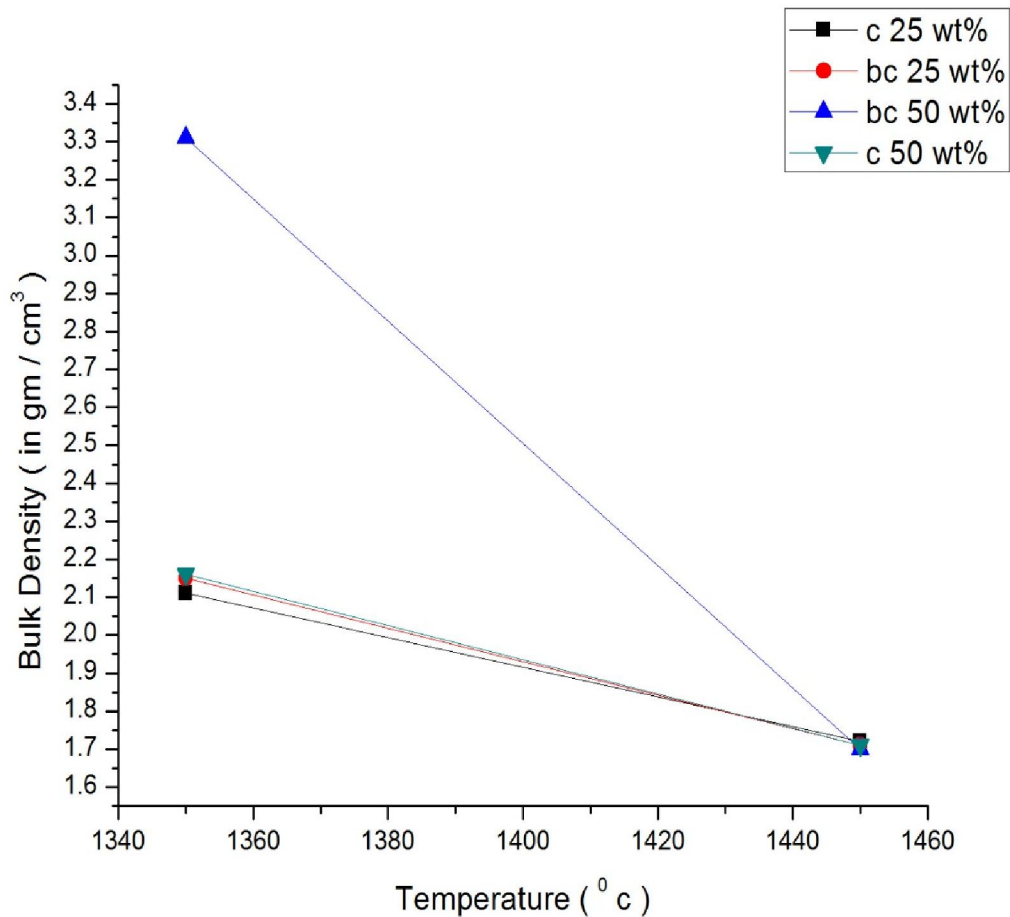


Figure No.1: Bulk density vs. temperature graph of four composition: china clay (25wt%)-feldspar (25 wt. %)-quartz (50wt %) and ball clay& china clay (25wt %)-feldspar (25 wt%)-quartz(50wt%) And china clay (50wt%)-feldspar (25wt%)-quartz (25wt %) And ball & china clay (50 wt.%)-feldspar (25wt%)-quartz (25wt %)

Figure no 1 shows the variation of bulk density of pellets with the increase of temperature.

At 1350<sup>0</sup>c , bulk density of first composition i.e china clay (25 wt%)-feldspar (25 wt%)-quartz(50 wt %) has lowest density (2.11 gm/cm<sup>3</sup>) . And bulk density of third composition i.e Ball & china clay (50 wt %)- quartz(25wt%)- feldspar( 25 wt%) has found highest density (3.31 gm/cm<sup>3</sup>).

It was showed that from first composition to second composition to third composition , density increased from 2.11 gm/cm<sup>3</sup> to 2.15 gm/cm<sup>3</sup> to 3.31 gm/cm<sup>3</sup> . and then decrease to 2.16 gm/cm<sup>3</sup> of fourth composition i.e china clay (50 wt%)-quartz(25wt%)-feldspar(25wt%).hence from first composition to third composition gives good packing during casting turn to increase the bulk density.

When fired at 1450<sup>0</sup>c of different composition of triaxial bodies. It was observed that there is gradually decrease in the bulk density from first composition to second composition to third composition, density decrease from 1.72 gm/cm<sup>3</sup> to 1.71gm/cm<sup>3</sup> to 1.70 gm/cm<sup>3</sup> and then slightly increment of density (1.71 gm/cm<sup>3</sup>). This is due to the formation of blister and bloating, when fired above the vitrified range, close porosity leads to increase. Hence the bulk density got reduced.

## 5.2 Apparent porosity (A.P) (in %) :

Table No: (6) Apparent porosity vs. temperature

Serial No	Temperature	C (25 wt %)	BC(25wt %)	BC(50wt%)	C(50wt%)
1	1350 <sup>0</sup> c	16.61	14.138	26.01	24.03
2	1450 <sup>0</sup> c	16.98	20.76	28.13	25.28

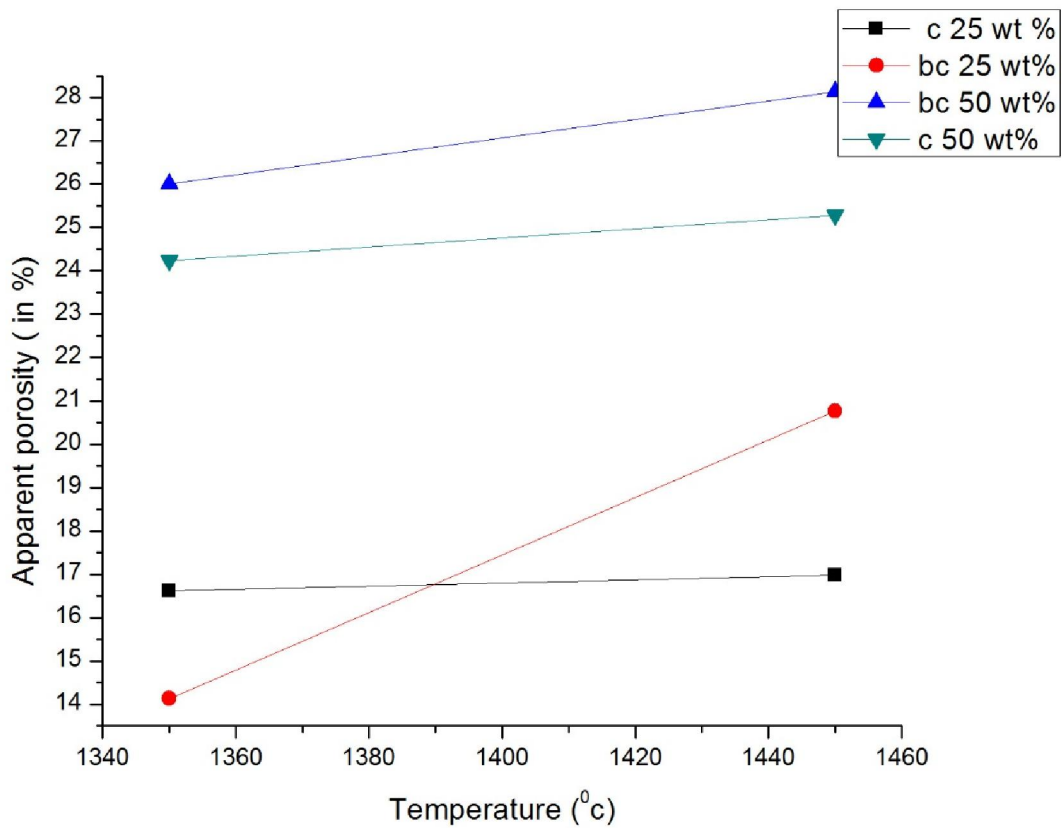


Figure No.2: Apparent porosity vs temperature graph of four composition: china clay (25wt%)-feldspar (25 wt%)-quartz (50wt %) and ball clay&china clay (25wt %)-feldspar (25 wt%)-

quartz(50wt%) And china clay (50wt%)-feldspar (25wt%)-quartz (25wt %) And ball & china clay (50 wt%)-feldspar (25wt%)-quartz (25wt %).

Figure no (2) indicates the variation of apparent porosity of pellets with the increase of temperature.

Four composition of samples are fired at different temperature 1350<sup>0</sup>c and 1450<sup>0</sup>c. And apparent porosity was observed.

At 1350<sup>0</sup>c , it was found that from first composition i.e china clay (25wt%)-feldspar(25wt%)-quartz(50 wt %) to 2<sup>nd</sup> composition i.e ball&china clay (25wt%)-feldspar (25wt%)-quartz (50wt%)

Apparent porosity decrease from 16.61 % to 14.38% .

And again from third composition i.e ball clay & china clay (50 wt %) to fourth composition i.e china clay (50 wt%)-feldspar (25wt%)-quartz(25wt%)

Apparent porosity decrease from 26.01% to 24.23%. This can be explained by during this firing of temperature ,large number of pores are being eliminated. And due to better packing during casting numbers of pores became less. And leads to reduced the apparent porosity.

At 1450<sup>0</sup>c , apparent porosity increase from first composition to 2<sup>nd</sup> to third composition .

From 16.98% to 20.76% to 28.13%. And then decrease to fourth composition (25.28%). It was obtained that firing above vitrified range. Gases are being entrapped and result in the formation of blister and bloating. Closed porosity are being increase. This is due to the lower amount of liquid phase formed.

### 5.3 Linear Shrinkage (in %):

Table No: (7) linear shrinkage vs temperature

Serial No	Temperature	C(25 wt%)	BC(25wt%)	BC(50wt%)	C(50wt%)
1	1350 <sup>0</sup> c	3.58	3.89	6.95	9.89
2	1450 <sup>0</sup> c	0.34	0.41	0.44	0.49

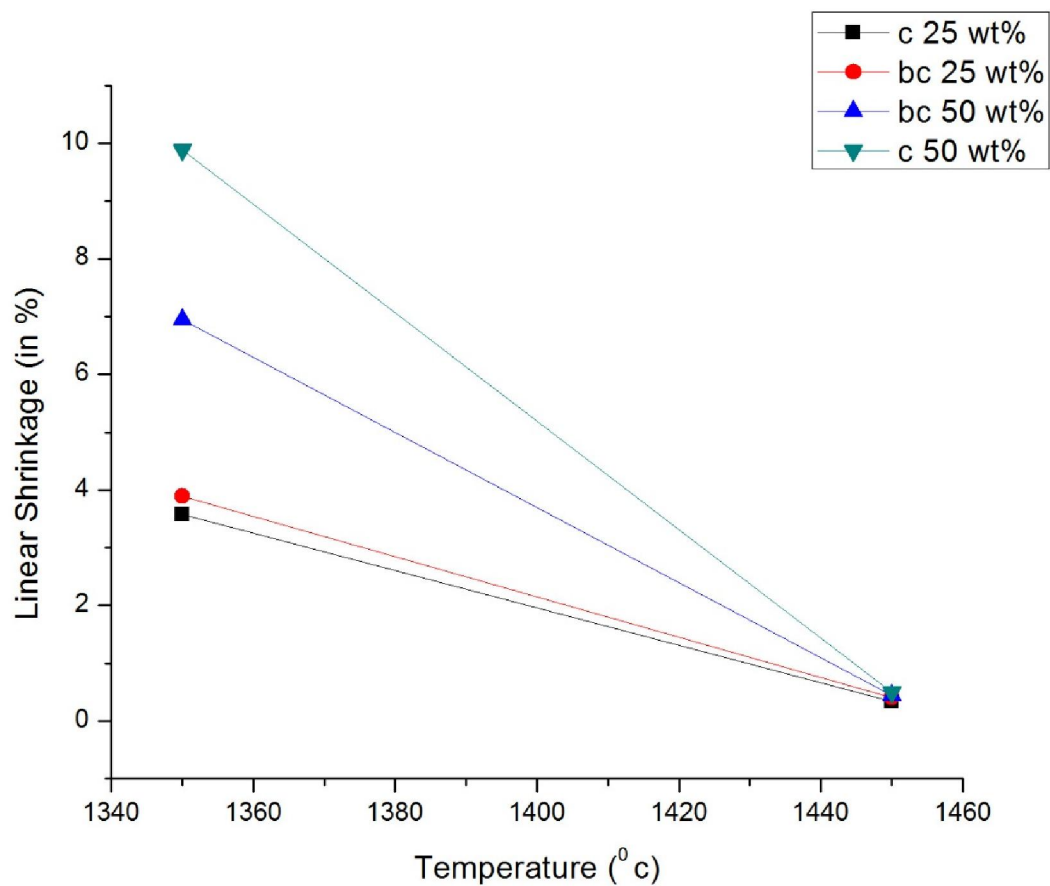


Figure No.3 : Linear shrinkage vs. temperature graph of four composition: china clay (25wt%)-feldspar(25 wt%)-quartz(50wt%) and ball clay& china clay (25wt %)-feldspar (25 wt%)-quartz

(50 wt%) And china clay (50wt%)-feldspar (25wt%)-quartz (25wt %) And ball & china clay (50 wt%)-feldspar (25wt%)-quartz (25wt %).

Figure no (3) indicates the variation of linear shrinkage of pellets with the increase of temperature.

Four composition of samples are fired at different temperature 1350<sup>0</sup>c and 1450<sup>0</sup>c. And linear shrinkage was observed.

In both temperature , linear shrinkage increase from first composition i.e china clay (25wt%)-feldspar (25wt%)- quartz(50wt%) to fourth composition i.e china clay (50 wt%)-feldspar(25wt%)-quartz(25wt%) . but at 1350<sup>0</sup>c there is large change in the linear shrinkage from 2<sup>nd</sup> composition i.e ball clay& china clay (25wt %)-feldspar (25 wt%)-quartz(50wt%) to fourth composition i.e china clay (50 wt%)-feldspar(25wt%)-quartz(25wt%) : from 3.89% to 6.95% to 9.89%. During this temperature, pores are come closer to each other near the vitrification range. Liquid formation take place. no of pores are filled by the liquid. And leads to enhancement of linear shrinkage.

At 1450<sup>0</sup>c , linear shrinkage are gradually increased from from first composition i.e china clay (25wt%)- feldspar (25wt%)- quartz(50wt%) to fourth composition i.e china clay (50 wt%)-feldspar(25wt%)-quartz(25wt%) . increase from 0.34% to 0.41% to 0.44% to 0.49%.It was found that firing above vitrified range . amount of liquid phase has become less. Hence due nonwettability of phase leads to reduced the linear shrinkage.



**5.4 Tensile strength value ( in Newton) : Brazilian Test** is a test for obtaining the tensile strength . A disc circular pellet sample is carried by two strip loads at the disc periphery. The load has given to samples until sample will failure. . At the failure, the tensile strength of samples has been measured.

Table No: (8) tensile strength vs temperature

Serial No	Temperature	C(25 wt%)	BC(25wt%)	BC(50wt%)	C(50wt%)
1	1350 <sup>o</sup> c	4233.5 N	2500.5 N	6096 N	8421 N
2	1450 <sup>o</sup> c	2158.5 N	1872.5 N	4560 N	6182 N

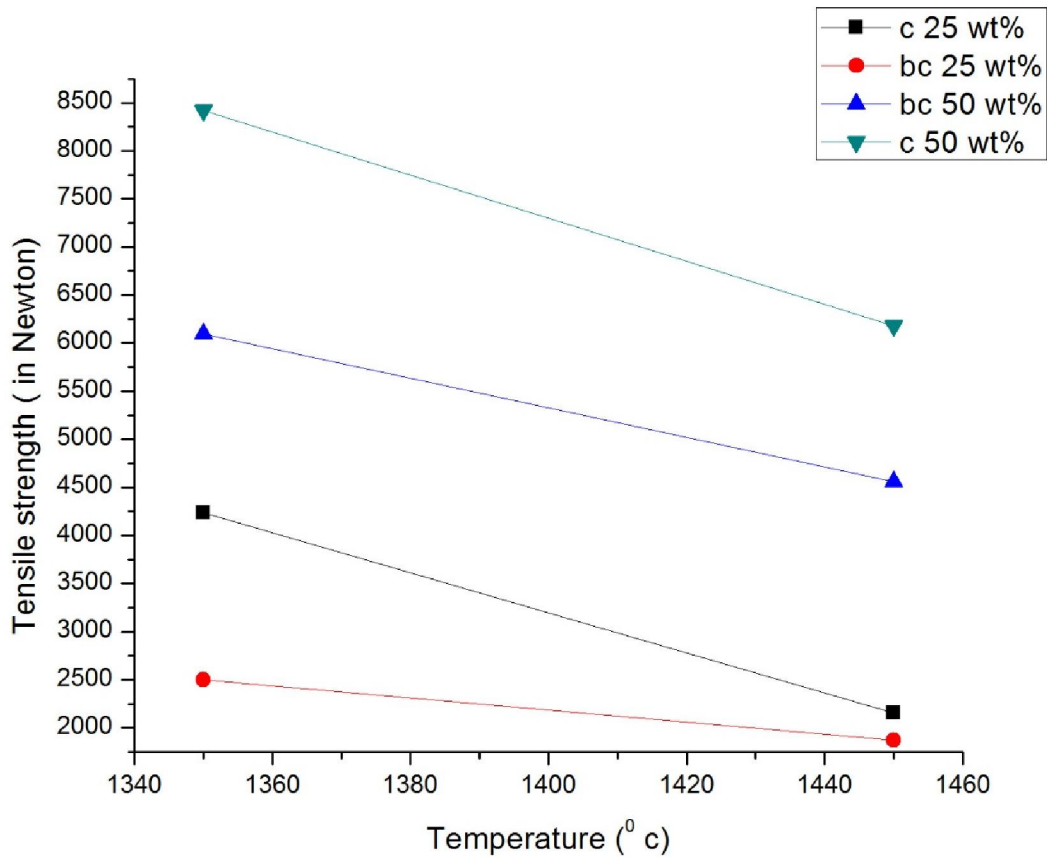


Figure No.4: tensile strength vs. temperature graph of four composition: china clay (25wt%)-feldspar (25 wt%)-quartz (50wt %) and ball clay& china clay (25wt %)-feldspar (25 wt%)-quartz (50wt %) And china clay (50wt%)-feldspar (25wt%)-quartz (25wt %) And ball & china clay (50 wt%)-feldspar (25wt%)-quartz (25wt %).

Figure no (4) indicates the variation of tensile strength of pellets with the increase of temperature.

Four composition of samples are fired at different temperature 1350<sup>0</sup>c. and 1450<sup>0</sup>c And tensile strength was observed.

At 1350<sup>0</sup>c , tensile strength of 2nd composition i.e china clay&ball clay (25 wt%)-feldspar(25 wt%)-quartz(50 wt%) has lowest strength ( 2500.5 N) . And tensile strength of fourth composition i.e china clay (50 wt %)- quartz(25wt%)- feldspar( 25 wt%) has found highest strength (8421 N).

It was showed that from first composition to second composition strength decrease from 4233.5 N to 2500.5 N. Then second composition to third to fourth composition, strength increase from 2500.5 N to 6096 N to 8421 N.

Strength has increased mainly due to presence of quartz which is the solid materials are not melt near the vitrification range. This solid materials is the skeleton pores are filled during liquid formation which gives strength during firing of body.

At 1450<sup>0</sup>c , tensile strength of 2<sup>nd</sup> composition i.e china clay (25 wt%)-feldspar(25 wt%)-quartz(50 wt%) has lowest strength ( 1872.5N). And tensile strength of fourth composition i.e Ball & china clay (50 wt %)- quartz(25wt%)- feldspar( 25 wt%) has found highest densit ( 6182N).

It was also showed that from first composition to second composition strength decrease from 2158.5 N to 1872.5 N. Then second composition to third to fourth composition, strength increase from 1872.5 N to 4560 N to 6182 N. but as compared to the four composition of 1350<sup>0</sup>c , strength has been reduced at 1450<sup>0</sup>c.

Hence , we can explained that as we increase temperature from 1350<sup>0</sup>c to 1450<sup>0</sup>c, the formation of glass phase as well as mullite phase increases , and due to this formation of glass ,tensile strength are getting decrease. Because glasses are brittle.

## 5.5 Dilatometry analysis :

**Dilatometry analysis** is the test to identify the sintering stages of whiteware ceramics. And to measure the shrinkage of whiteware bodies. The heating profile consisted of initial heating from room temperature (RT) to 1250 °C at 10°C/min in presence of oxygen atmosphere.

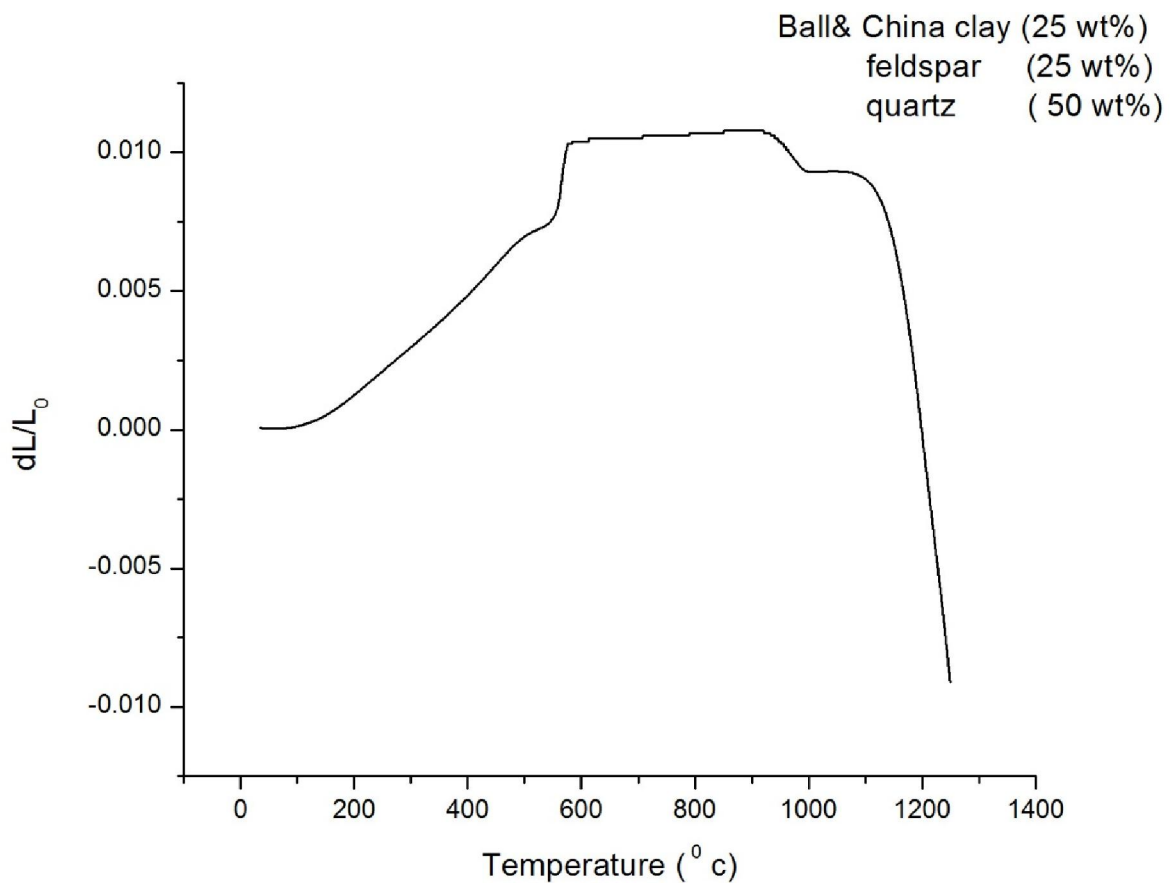


Figure No: 5  $dL/L_0$  vs temperature curve of ball & china clay (25wt%)-feldspar (25wt%)-quartz(50wt%) sample

Initially length is increased when increasing in temperature from 34.53<sup>0</sup>c to 580.01<sup>0</sup>c. The change in length in % during this temperature range was 40.9%. This is due to thermal expansion of material. This may be due to transform of clay to meta kaolin at around 530<sup>0</sup>c. Then there is constant length from 580.1<sup>0</sup>c to 926<sup>0</sup>c . hence neither contraction nor expansion has happened. Then from 926<sup>0</sup>c to 1000.5<sup>0</sup>c , there is no change in length . thus contraction will take place. This may be due to removal of lattice water, and metakaolin starts shrinkage, and converted into a spinel-like structure at 985<sup>0</sup>c. then from temperature 1000.5<sup>0</sup>c to 1091<sup>0</sup>c , there was constant in length. Again neither contraction nor expansion will take place. Beyond 1091<sup>0</sup>c , there was no change in length . shrinkage is obtained very rapidly . this may be due to formation of liquid phases when feldspar are getting melt. As the temperature further continues to increase, porosity is eliminated by glassy phases, which finally increase densification.

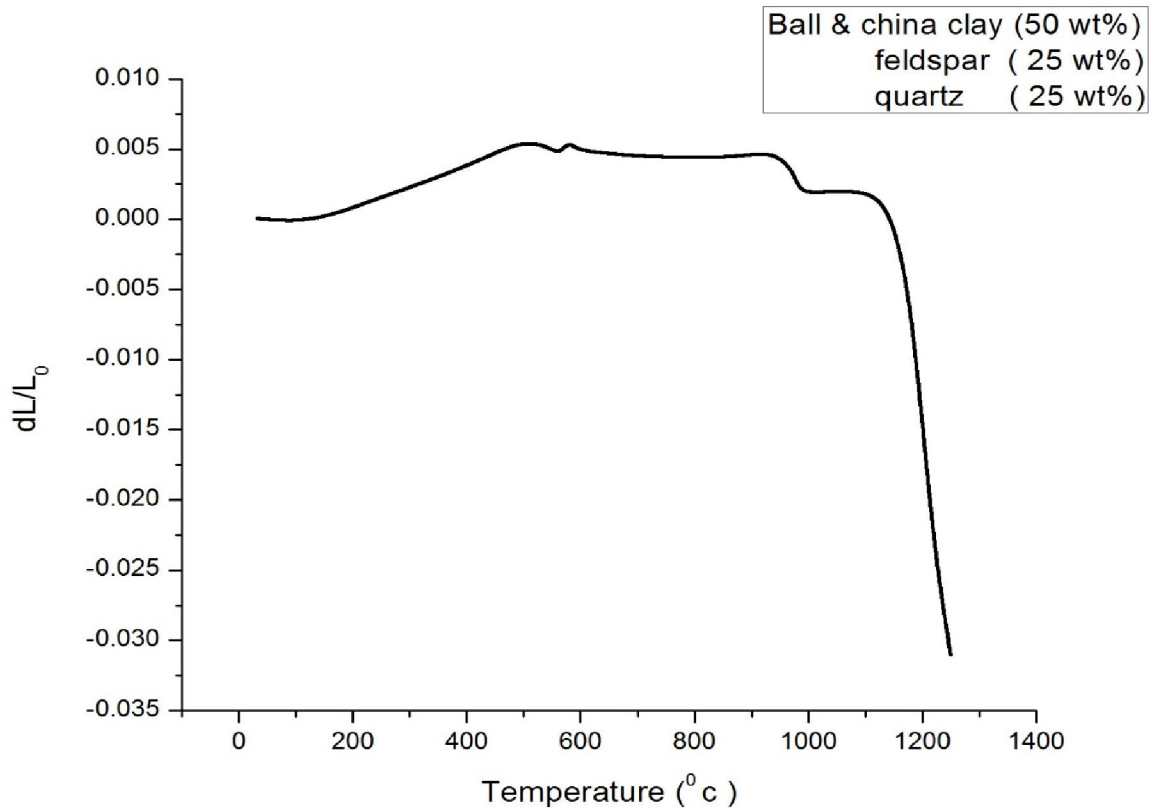


Figure No: (6)  $dL/L_0$  versus temperature of sample: ball&china clay (50 wt%)-feldspar(25wt%)-Quartz (25wt %)

According to figure no 6, there is very small change in length when the temperature is increased from 32.37<sup>0</sup>c to 503<sup>0</sup>c . The change of length in % is 21.36 % . This is due to thermal expansion. and expansion will take place may due to transform of clay to metakaolin at around 503<sup>0</sup>c. then from temperature 503<sup>0</sup>c to 563<sup>0</sup>c , no change in length. Thus low shrinkage was observed. And contraction will take place. Again from temperature 563<sup>0</sup>c to 582.3<sup>0</sup>c , very low thermal expansion was found. And then from 582.3<sup>0</sup>c to 936<sup>0</sup>c . The change in length was constant over this temperature range. Then from temperature 936<sup>0</sup>c to 1001<sup>0</sup>c . there is no change in length . thus contraction will take place. This may be due to removal of lattice water, and metakaolin starts

shrinkage, and converted into a spinel like structure at 985<sup>0</sup>c. Then from temperature 1001<sup>0</sup>c to 1099<sup>0</sup>c there was constant in length. Again neither contraction nor expansion will take place.

And beyond 1099<sup>0</sup>c . There was no change in length. Shrinkage is obtained very rapidly. This may be due to formation of liquid phases when feldspar are getting melt. As the temperature further continues to increase, porosity is eliminated by glassy phases, which finally increase densification.

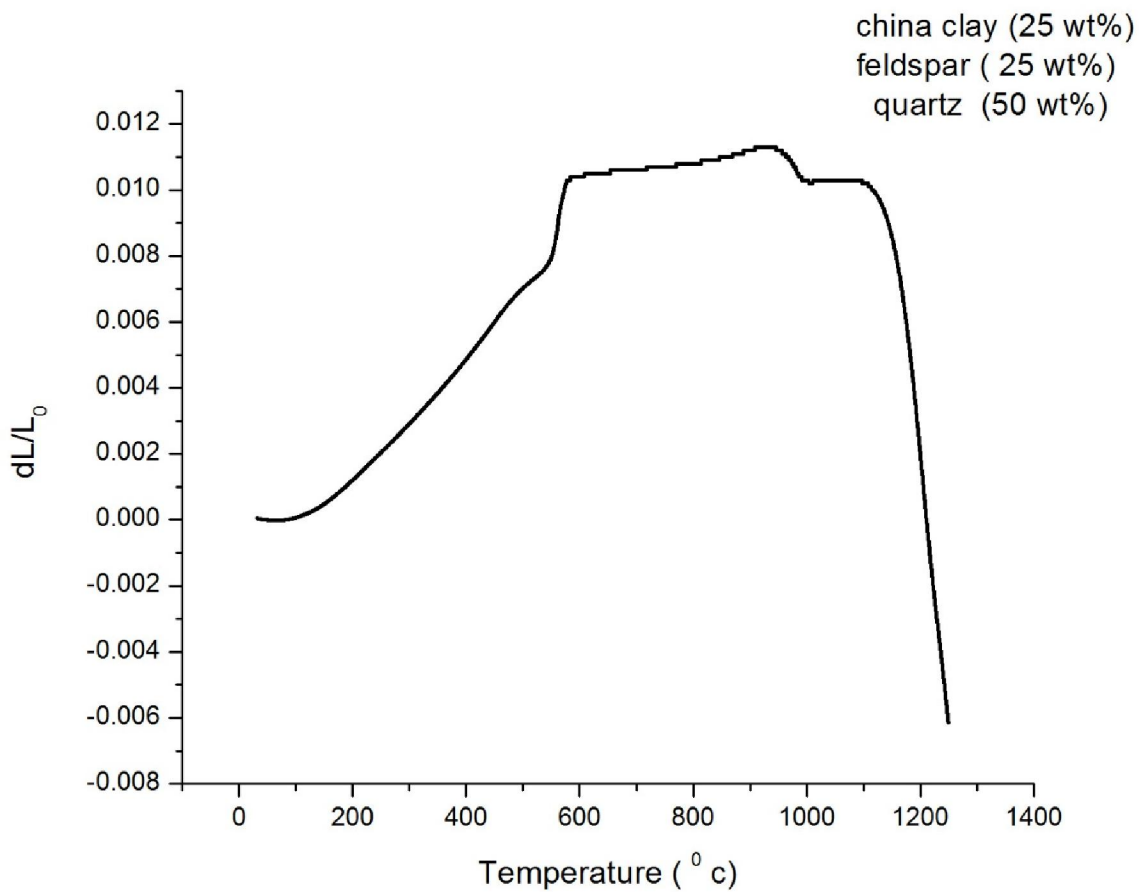


Figure No: (7)  $dL/L_0$  versus temperature of sample : china clay (25 wt%)-feldspar(25wt%)-Quartz (50wt%)

According to figure no (7). There is very large change in length when the temperature is increased from  $31.53^{\circ}\text{C}$  to  $583^{\circ}\text{C}$ . The change of length in % value is 44 % over wide range of temperature range. This is due to thermal expansion. And expansion will take place may due to transform of clay to metakaolin at around  $530^{\circ}\text{C}$ . then from  $583^{\circ}\text{C}$  to  $912^{\circ}\text{C}$ . The change in length was constant over this temperature range. Hence neither expansion nor contraction was observed. Then from temperature  $912^{\circ}\text{C}$  to  $995^{\circ}\text{C}$ . There is no change in length. thus contraction will take place. This may be due to removal of lattice water, and metakaolin starts shrinkage, and converted into a spinel like structure at  $985^{\circ}\text{C}$ . then from temperature  $995^{\circ}\text{C}$  to  $1111^{\circ}\text{C}$  there was constant in length. Again neither contraction nor expansion will take place.

And beyond  $1111^{\circ}\text{C}$ . there was no change in length. Shrinkage is obtained very rapidly. this may be due to formation of liquid phases when feldspar are getting melt. As the temperature further continues to increase, porosity is eliminated by glassy phases, which finally increase densification.



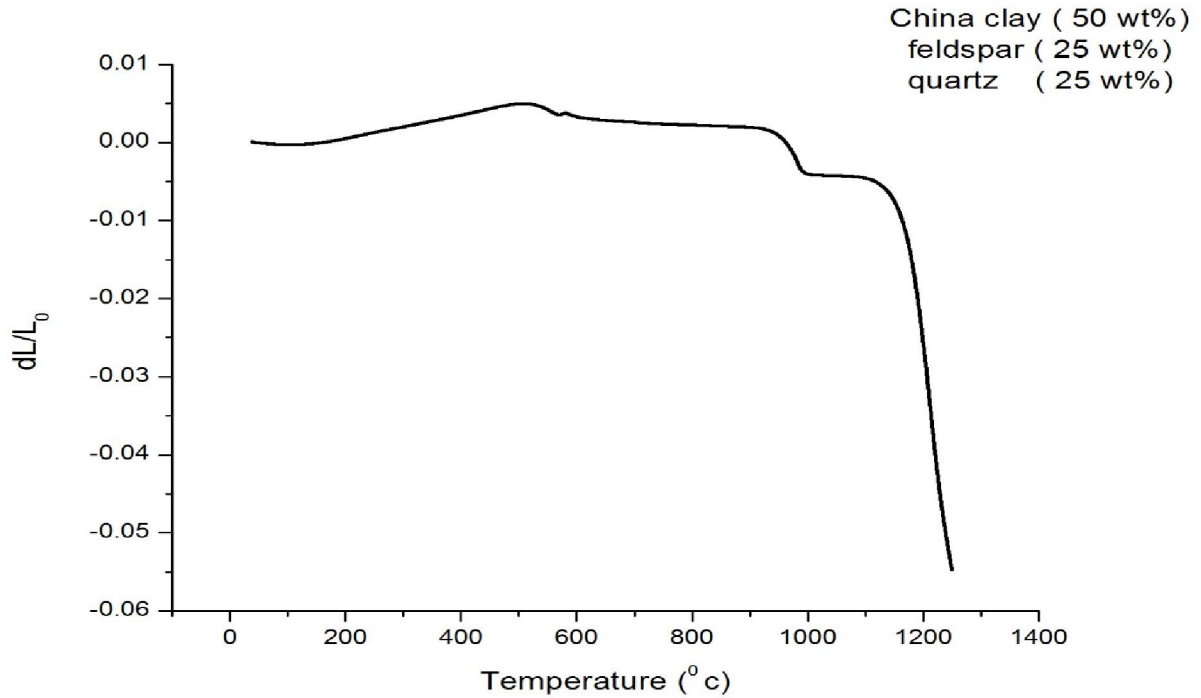


Figure No: (8)  $dL/L_0$  versus temperature of sample: china clay (50 wt%)-feldspar (25wt%)-Quartz (25wt%)

According to figure no 8. There is very small change in length when the temperature is increased from 32.37<sup>0</sup>c to 497<sup>0</sup>c . The change of length in % is 19.4 % . This is due to thermal expansion . And expansion will take place may due to transform of clay to metakaolin at (450-550<sup>0</sup>c). Then from temperature 497<sup>0</sup>c to 573<sup>0</sup>c , no change in length. Thus low shrinkage was observed. And contraction will take place. Then from 573<sup>0</sup>c to 930<sup>0</sup>c . The change in length was constant over this temperature range. Neither contraction nor expansion will take place. . Then from temperature 930<sup>0</sup>c to 997<sup>0</sup>c . There is no change in length . Thus contraction will take place. This may be due to removal of lattice water, and metakaolin starts shrinkage, and converted into a spinel like structure at 985<sup>0</sup>c. Then from 997<sup>0</sup>c to 1104<sup>0</sup>c . The change in length was constant over this temperature range.again neither contraction nor expansion will take place. And beyond 1104<sup>0</sup>c .

There was no change in length. Shrinkage is obtained very rapidly. This may be due to formation of liquid phases when feldspar are getting melt. As the temperature further continues to increase, porosity is eliminated by glassy phases, which finally increase densification

**5.6. X-Ray Diffraction:** The position ( $^{\circ}2\theta$ ) range in which the analysis was done,  $5^{\circ}$ - $60^{\circ}$  and the rate was  $15^{\circ}/\text{min}$ . The intensity vs position data was obtained for 6 samples. The data was analyzed using **X'Pert HighScore** and phases were identified by tallying them with **JCPDs**.

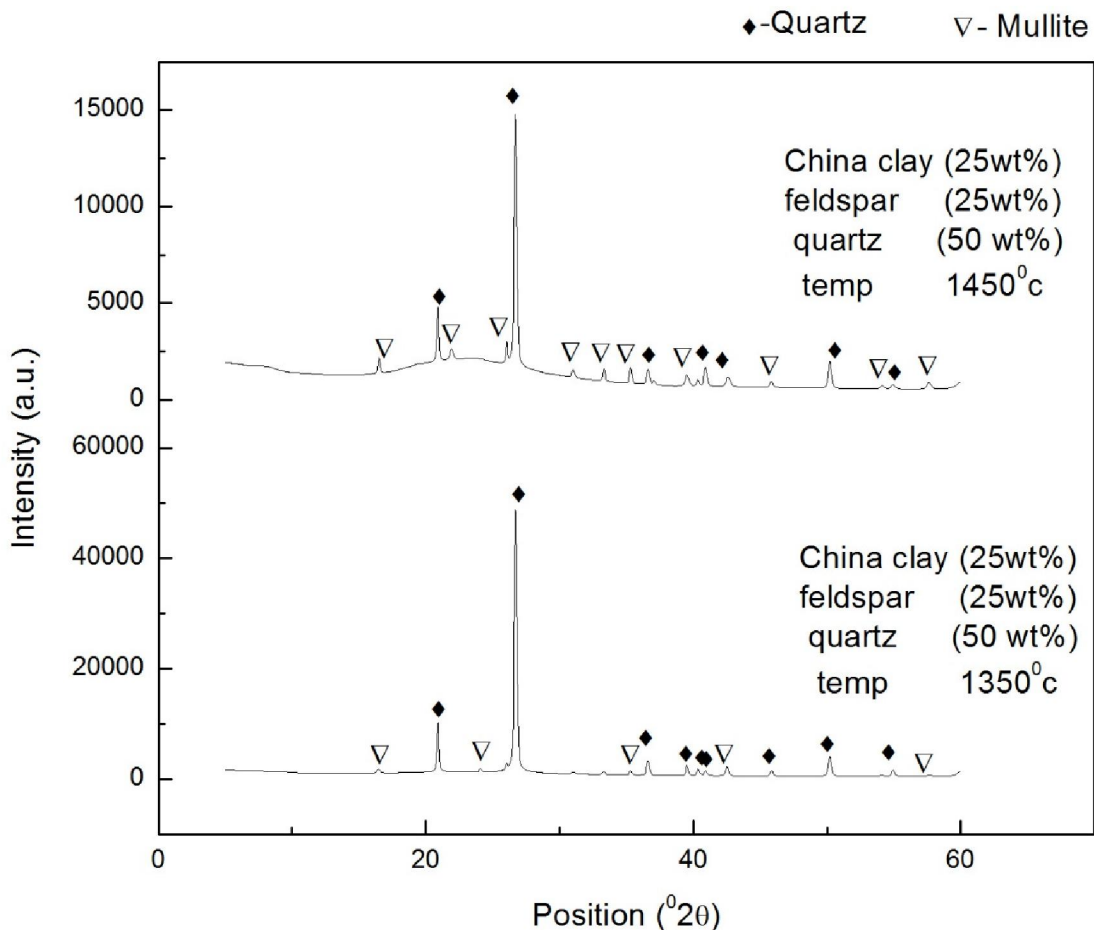


Figure No: 8 XRD of two sample having same composition at different temperature 1350<sup>o</sup>c & 1450<sup>o</sup>c.

Phase has been identified as major phase present is quartz and minor phase present is mullite in both temperature at 1350<sup>0</sup>c and at 1450<sup>0</sup>c .

According to figure no 8 , at 1350<sup>0</sup>c , intensity of quartz is maximum as increasing temperature up to 1450<sup>0</sup>c intensity of quartz are getting reduced. so we can explained that quartz has been consumed as there was formation of mullite phase has been occurred at high temperature and the shown curve indicated that there is also glass formation took place.

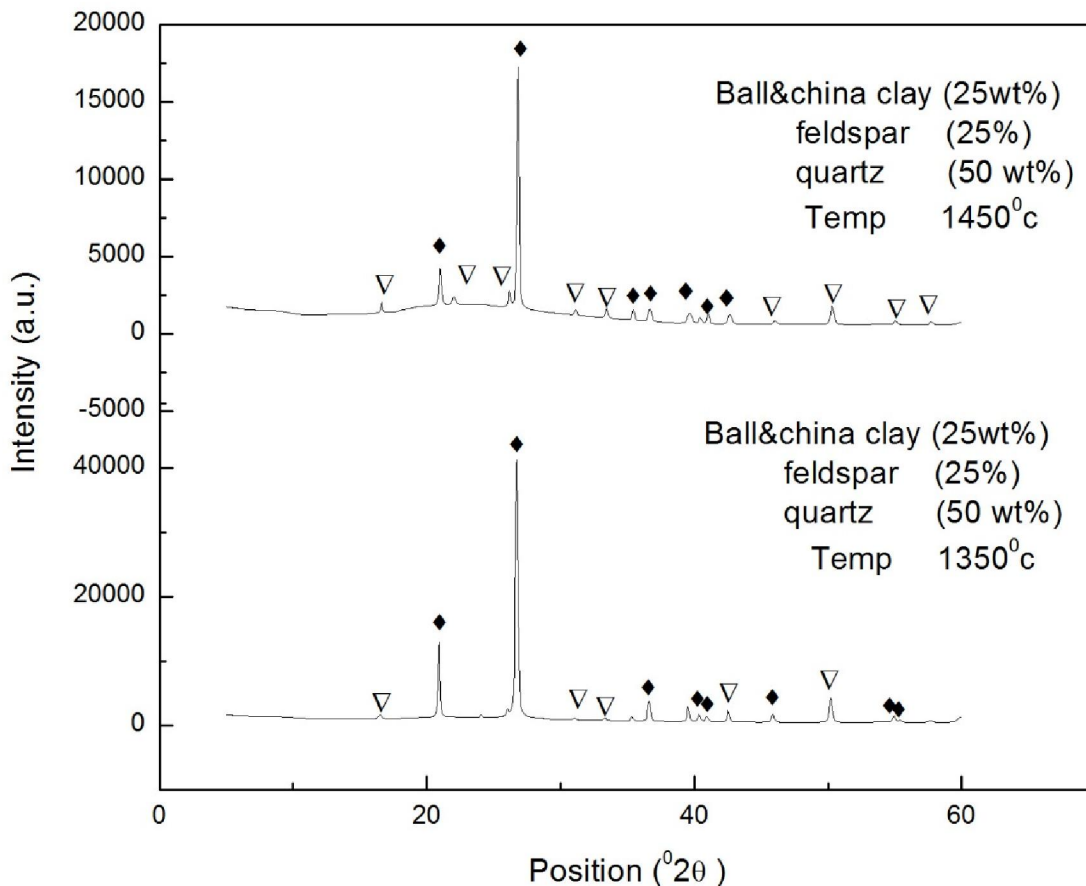


Figure No: 9 XRD of two sample having same composition at different temperature 1350<sup>0</sup>c & 1450<sup>0</sup>c.

Phase has been identified as major phase present is quartz and minor phase present is mullite in both temperature at 1350<sup>0</sup>c and at 1450<sup>0</sup>c .

According to figure no 9 , at 1350<sup>0</sup>c , intensity of quartz is maximum as increasing temperature up to 1450<sup>0</sup>c intensity of quartz are getting reduced. so we can explained that quartz has been consumed as there was formation of mullite phase has been occurred at high temperature and the shown curve indicated that there is also glass formation took place.

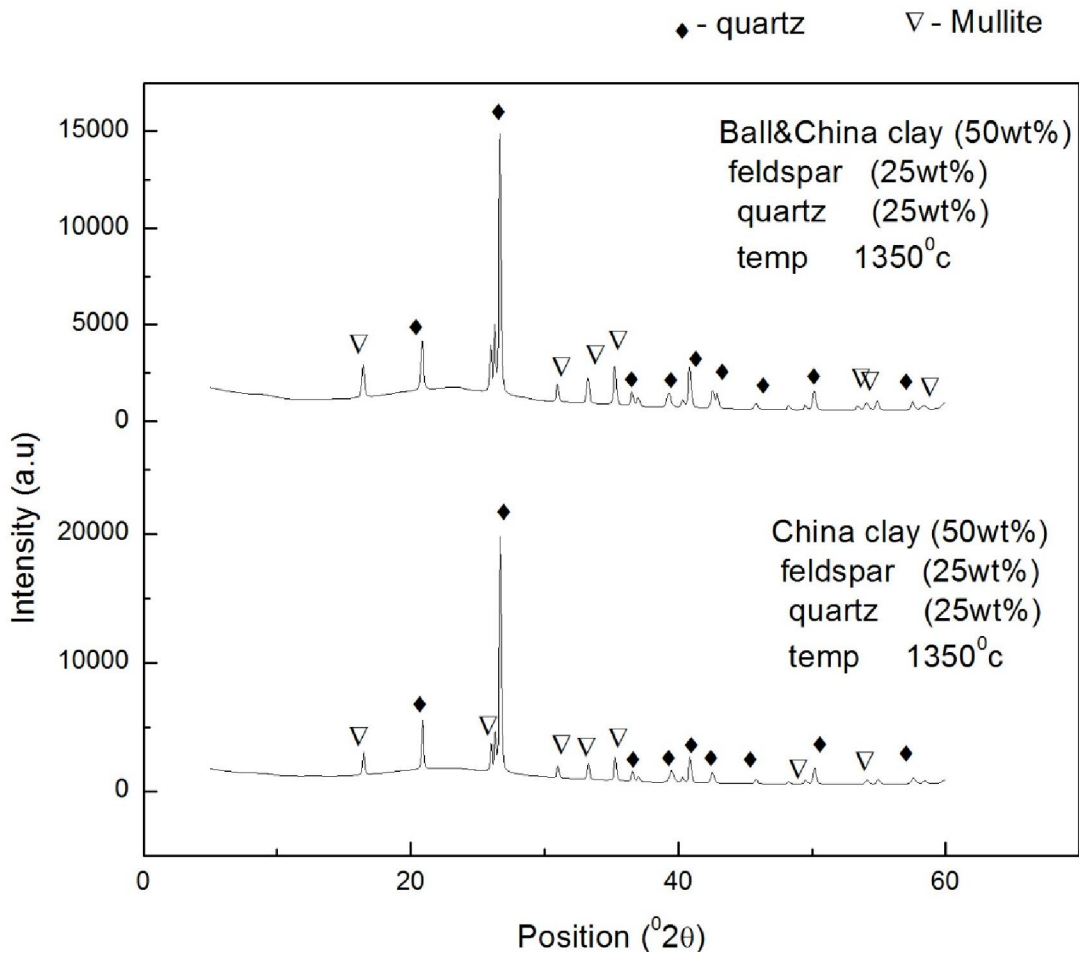


Figure No: 10 XRD of two sample having different composition at same temperature 1350<sup>0</sup>c.

Phase has been identified as major phase present is quartz and minor phase present is mullite temperature of 1350<sup>0</sup>c.

According to figure no 10 , at 1350<sup>0</sup>c there is number of quartz phase was present and some of them contain high peak intensity. And mullite contain less number of peaks and their peak intensity were also low. Maximum peak intensity contain in quartz phase is 26.74<sup>0</sup>.

Both composition shown similar behavior.

# **CHAPTER 6**

# **CONCLUSION**

## **5. Conclusion:**

1. The development of low clay whiteware bodies has been studied.
2. Bulk density and apparent porosity for different temperatures was obtained. As we increase temperature, bulk density values reduced and apparent porosity was getting increased.
3. Linear shrinkage for different temperatures was also obtained. We found that as we increase temperatures linear shrinkage were reduced.
4. Tensile strength was also reduced. As we increase temperatures, there was increase in glass formation as well mullite phase was observed. Due to this glass formation, strength turn to reduced. Because glasses are brittle.

# CHAPTER 7

# REFERENCE

## 6. Reference:

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