PROJECT REPORT ON

EFFECTS OF PARTICLE SIZE DISTRIBUTION ON THE PROPERTIES OF ALUMINA REFRACTORIES

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE

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By

MEENA SEEMA N.

Under the guidance of

Prof. Ritwik Sarkar

Department of Ceramic Engineering

National Institute of Technology

Rourkela-769008

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National Institute of Technology
Rourkela-769008

CERTIFICATE

This is to certify that the thesis entitled, "**Effect of Particle Size Distribution on Properties of Alumina Refractories"** submitted by **Miss Meena Seema N.** a part of requirements for the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by her under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/ Institute for the award of any Degree or Diploma.

Date:-

(Prof. Ritwik Sarkar)
Department of Ceramic Engineering
National Institute of Technology
Rourkela-769008

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Meena Seema N.

(Roll No. -107CR037)

Department of Ceramic Engineering

National Institute of Technology

Rourkela-769008.

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ABSTRACT

Particle size distribution and packing of particles are important to achieve high packing density and also fired density of any ceramic product. Mono-sized particles can only achieve upto a certain extent of packing density and for further increase in packing multiple sizes are required. The basic concept of packing of multiple sizes of grains is that finer grains go into the inter-particle voids of the coarser particles and thus improve the packing density. In the present study alumina based refractory is planned with using three different alumina grains. Effect of different grain size distributions using these three different sized grains on the fired properties of the pressed and fired shapes are studied. Six different distributions were used in the study, which were pressed and fired at 1550, 1600 and 1650ºC. The fired products were characterised for volumetric shrinkage, bulk density, apparent porosity and cold crushing strength.

CHAPTER – 01

INTRODUCTION

1.1 GENERAL INFORMATION

Refractories, a key input for iron and steel making, assume ever increasing role due to high stress on production of high quality sophisticated steels, which alone accounts for the consumption of nearly 70% of total refractories produced [1]. Refractories in general, are the non-metallic materials that are hard to melt at high temperature and having enough mechanical strength and heat resistance to withstand rapid temperature change, including repeated heating and cooling and resistance to molten slag and metals. Generally refractories are classified into two categories i.e.

(i) The shaped refractories, available in the form of different brick shape, and include the oxide and non-oxide system.

(ii) The unshaped refractories, which includes mortars, castables, plastics etc. [2].

Three types of refractory materials exist depending upon their chemical nature as given below:-

1) **Acidic refractor**y: - It should not be allowed to come in contact with basic products to avoid the reaction.

   Examples – *Fire clay, Silica, Quartz, Ganister sand, Semi-silica etc.*

2) **Basic refractory: -** Should not come in contact with acidic products.

   Examples – *Bauxite, lime, magnesite, dolomite,* ***alumina****, zirconia.*

3) **Neutral refractory: -** Can be used in conjunction with acidic or basic products.

   Examples – *Chromite, graphite, carbon, carbide, mullite, kyanite.*

1.2 ALUMINA REFRACTORIES

Refractories of this group are not used so extensively because they are costly. As a general rule the refractoriness increases with increase in the Al_2O_3 content of alumina silicate refractories. Raw materials for high alumina refracrory bricks which are chemically or ceramic bonded is mostly precalcined for giving lower firing shrinkage. Dry pressing is normally used for moulding the mixture of high alumina material and granular grog, though hand moulding and pneumatic ramming is also done to produce some special shapes [3].

1.3 ALUMINA (Al_2O_3)

1.3.1 BACKGROUND

Aluminum Oxide (Al_2O_3) or alumina is one of the most versatile of refractory ceramic oxides and finds use in a wide range of applications. It can exist in several crystalline phases which all revert to the most stable hexagonal alpha phase at elevated temperatures. This is the phase of particular interest for structural applications [4].

Alpha phase alumina is the strongest and stiffest of the oxide ceramics. Its high hardness, excellent dielectric properties, refractoriness and good thermal properties make it the material of choice for a wide range of applications [4].

High purity alumina is usable in both oxidizing and reducing atmospheres to 1925°C.Weight loss in vacuum ranges from 10^{-7} to 10^{-6} g/cm^2.sec over a temperature range of 1700°C to 2000°C. It resists attack by all gases except wet fluorine and is resistant to all common reagents except hydrofluoric acid and phosphoric acid. Elevated temperature attack occurs in the presence of alkali metal vapors particularly at lower purity levels [4].

It is found in nature as corundum in emery, topaz, amethyst, and emerald and as the precious gemstones ruby and sapphire, but it is from the more abundant ores such as bauxite, cryolite and clays that the material is commercially extracted and purified [4].

Corundum exists as rhombohedral crystals with hexagonal structure. The unit cell is an acute rhombohedron of side length 5.2Å and plane angle ~55°. It is the close packing of the aluminum and oxygen atoms within this structure that leads to its good mechanical and thermal properties [4].

1.3.2 EXTRACTION ROUTES

The most common process for the extraction and purification of alumina is the 'Bayer' process. The first step in the process is the mixing of ground bauxite into a solution of sodium hydroxide. By applying steam and pressure in tanks containing the mixture, the bauxite slowly dissolves. The alumina released reacts with the sodium hydroxide to form sodium aluminate. After the contents of the tank have passed through other vessels where the pressure and temperature are reduced and impurities are removed, the solution of sodium aluminate is placed in a special tank where the alumina is precipitated out. The precipitate is removed from the tank, washed, and heated in a kiln to drive off any water present. The residue is a commercially pure alumina [4].

Other extraction processes are used including pyrogenic treatment of bauxite with soda, and the extraction of aluminum hydroxide from metakaolin via either the chloride or sulphate.

The yield of alumina from these processes can approach 90%. For advanced ceramics uses, the alumina manufactured by these processes requires further purification. This is often achieved by recrystallization from ammonium alumina [4].

1.3.3 TYPICAL ALUMINA CHARACTRISTICS

- Good strength and stiffness
- Good hardness and wear resistance
- Good corrosion resistance
- Good thermal stability
- Excellent dielectric properties (from DC to GHz frequencies)
- Low dielectric constant
- Low loss tangent

1.3.4 APPLICATIONS OF ALUMINA

With such a wide range of composition and properties, alumina ceramics find a wide range of applications [4]. Some of the major application areas can be grouped as:-

- **High Temperature and Aggressive Environments**

  Its high free energy of formation makes alumina chemically stable and refractory, and hence it finds uses in containment of aggressive and high temperature environments.

- **Wear and Corrosion Resistance**

  The high hardness of alumina imparts wear and abrasion resistance and hence it is used in diverse applications such as wear resistant linings for pipes and vessels, pump and faucet seals, thread and wire guides etc.

- **Biomedical**

  High purity alumina is also used as orthopedic implants particularly in hip replacement surgery.

- **Metal Cutting Tools**

  The high "hot" hardness of alumina have led to applications as tool tips for metal cutting (though in this instance alumina matrix composites with even higher properties are more common) and abrasives.

- **Milling Media**

  Alumina is used as milling media in a wide range of particle size reduction processes.

- **Microwave Components**

  The high dielectric constant coupled with low dielectric loss particularly at high frequencies leads to a number of microwave applications including windows for high power devices and waveguides.

- **Electrical Insulation**

  The high volume resistivity and dielectric strength make alumina an excellent electrical insulator which leads to applications in electronics as substrates and connectors, and in lower duty applications such as insulators for automotive spark plugs.

1.4 RAW MATERIALS USED IN ALUMINA REFRACTORIES

There are following raw materials which are generally used in alumina refractories:-

- Tabular alumina
- White fused alumina, and
- Reactive alumina

1.4.1 TABULAR ALUMINA

Tabular alumina is made from aluminum hydroxide derived from bauxite by the Bayer process. The aluminum hydroxide from the Bayer process is pelletized into balls about 2.5cm in diameter.

After going through the drying process, these balls are fed into the top of a shaft kiln and are gradually heated as they go down the furnace. The aluminum hydroxide is heated and shrunk at high temperature (around 2035ºC, just below the fusion point of alumina) with or without the addition of additives (i.e., MgO and B_2O_3). High temperature sintering helps the large crystals to plug up the pores, and densifies close to the theoretical density [5].

The soda content is an important ingredient in the tabular alumina formation. Tabular alumina is normally used in refractories for extremely high temperature.

1.4.2 WHITE FUSED ALUMINA

White fused alumina is produced by melting calcined alumina at above 2040ºC in an electric arc furnace. Although this is the pure form of fused alumina, the products available on the market are more porous and as such, the bulk density is lower than other alumina raw materials. It has high refractoriness, abrasion resistance as well as chemical inertness, but has not been found to be suitable or cost effective for most refractory applications [5].

1.4.3 REACTIVE ALUMINA

Reactive alumina plays an important role in monolithic refractories. Several varieties of reactive alumina are available in a broad range of particle sizes. The selection of reactive alumina depends on the rheological effect of the desired form of the particles, particularly in castable refractories. The size of the refractory particles plays an important role in the flow-ability, setting and ultimate chemical reaction in the refractory body.

Type of alumina which are sinterable to nearly theoretical density at relatively low temperature i.e. at range of 1550- 1600°C, that's why alumina is called reactive alumina with respect to sintering [5].

1.5 APPLICATIONS OF ALUMINA REFRACTORIES

High alumina refractory bricks are used in blast furnace stoves and other regenerative furnaces, cement and lime rotary kilns, electric arc furnace roof, ladles and glass melting furnaces. Due to their non-wetting characteristics the chemically bonded alumina refractory bricks are specially used in alumina melting and holding furnaces. Porous high alumina lances for blowing steel with inert gases in ladles and to use chamotte ground dense refractories for lining blast furnace shaft are the other uses of high alumina refractories under development [3].

Blast furnace hot stove wall and air ducts are lined with mullite-corundum type high alumina refractories (containing > 78% alumina) made by mixing kaolin with grade chamotte (containing alumina powder of 10-60 µm size), briquetting and firing at 1750ºC for 80 hours. Natural sillimanite blocks (a high alumina refractory) is used in glass industry [3].

CHAPTER - 02

LITERATURE REVIEW

2.1 POWDER PROCESSING AND PACKING OF POWDERS

2.1.1 INTRODUCTION

Powder processing has a wide scope, meaning preparing powder by size reduction, giving a green shape etc. The use of powder may end in the form of powder itself, such as polishing powders. Mostly, the powders are utilized for making shapes, which are strong and made useful for many applications. Powders are used for being melted and given shape, as in glass making or the green shape may be treated at high temperature for developing strength such as is done in most other ceramic industries. Here we have chosen to deal with principles of packing, as dense packing is desirable in many cases [6].

What ordinarily one can hold in hand as discrete particles is called pebbles. Lesser than that size may be called powders. The lower limit is the nano-size [6].

2.1.2 SIGNIFICANCE OF PARTICLE PACKING

The porosity as well as the pore characteristics of compacted mass consisting of solid particulate system is the manifestation of the packing of the particles. Flow of fluids through a tower packed with a solid particulate material, filtration of a slurry containing fine solids, passage of combustion gases through a shaft kiln charged with solid lumps, rate of drying of moist cakes and crystals, sintering of pressed items of ceramic and metallic systems are all governed by the total porosity of the bed and also the size, shape and distribution of the pores [7].

2.1.3 SOME DEFINITIONS [7]

- **Packing of particles: -** Packing of particles may be defined as the selection of proper size, shape and proportions of particulate material as well as the compaction device so as to obtain a system with the desired porosity.
- **Porosity: –** Porosity of a sample or a compact is the percentage of the total space not occupied by the particulate material.
- **Bulk Density: –** Bulk density is the mass of the particles per unit volume of sample or compact.
- **Apparent volume: –** Apparent volume is the volume of the sample occupied by unit true volume of the particles.
- **Packing fraction: –** Packing fraction is the fraction of the total sample volume occupied by the particulate material.

2.1.4 PACKING OF SPHERES

Packing of mono-sized spherical particles gives insight into the importance of configuration of particles around a central one, which control the degree of voids one arrangement creates [6].

2.1.5 PACKING OF BINARY AND TERNARY SYSTEM [6]

The apparent volume for mono-sized particle has about 40% of void volume given by Coarse and Fine, as apparent volume or the unmixed mono-sized particle would not be different till their packing configuration remains the same.

When fine particles are added to the coarse pack and vibrated, the void created by the coarse is expected to filled. The apparent volume would change proportional to the amount of fines added. On the other hand, when coarse particles are gradually added to the fine pack, they displace a

number of fines along with the voids in between and as such the change of apparent volume would be more efficient if the original fine pack has more voids. Thus the change of apparent volume would be proportional to the original pore volume.

According to Norton, for three sizes the percent void reduces to about 23%. The significance is that the same percent void would evolve along the same curved lines depicted and it increases as the percent medium fraction increases [9].

2.2 APPLICATIONS OF PARTICLE PACKING [10]

The subject of packing of particles cuts across several disciplines of science and engineering. These include chemical, civil, powder metallurgical engineering, pharmacy and ceramic engineering. Comprehensive surveys on the structure of porous media in relation to some of their properties, such as permeability, capillary hysteresis, etc have been made by Eisenklam and Dullien and Batra. Various applications of particle packing have also been outlined by several other authors. Some of the applications are given below:-

- Application in concrete technology
- Application in ceramic technology
- Application in powder metallurgy
- Packing towers
- Packaging and transport
- Preparation of catalyst pellets
- Pharmaceutical preparations and nuclear fuel elements.

2.2.1 APPLICATION IN CERAMIC TECHNOLOGY

A ceramic body undergoing excessive drying and fired shrinkages gives rise to problems like warping and discrepancies in size. Bodies made out of properly graded particles will not present these difficulties. Grading of grogs and other components to obtain bodies with maximum density and minimum warpage is very important in refractories industry. Tri-axial white-ware bodies made out of quartz, feldspar and clays, represent a three component system of coarse, medium and fine particles. Careful selection of these components gives dense products without warping.

2.3 PARTICLE SIZE AND PHYSICAL PROPERTIES [11]

Like all ceramic materials and castables, in alumina refractories a particle size distribution is used. This helps to have a better packing comparing with using mono size particles. Besides influencing packing, Particle size distribution effects flow, apparent porosity and hence strength in castables. Some researches have been done to find a relation between particle size and physical properties.

Figure-1 shows a schematic way to predict flow ability and strength according to particle size. Particle sizes are divided in to three categories: fine, medium and coarse. By various formulations in the triangle, different flow rheology and strength would be resulted.

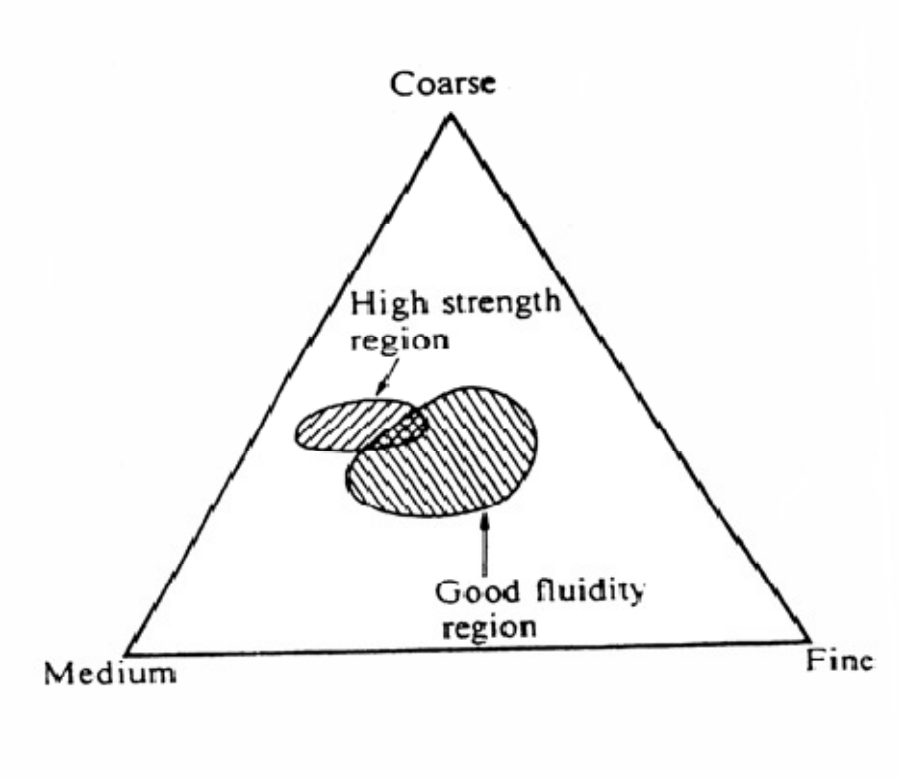

Figure 1 - Schematic particle size triangle

Hugill and Rees in 1930 suggested a batch composition coarse:medium:fine = 45:10:45 which remained very popular for a long time in formulation of batch composition in refractories [12].

Gugel and Norton (1962) arrived at the batch composition involving fireclay grog giving lowest porosity as -

	Coarse	**Medium**	**Fine**
Size (Tyler mesh)	4-8	35-65	-100
Wt. Proportion	44.8	13.8	41.4

The minimum porosity was 23%. Addition of a fourth component with water as lubricant and application of pressure of 15,000 psi resulted in a much lower porosity of 14.5% [13].

Karisson and Spring (1970) working also with fire clay grog of irregular shape found that even fine particles added to coarse particles causes expansion of bed. For a three-component system, the best proportion suggested was 40:30:30 [14].

McOeary (1961) experimented with packing of spherical glass shots in glass containers, and subjected them to mechanical vibration. He made some useful observations, as follows [15]:

- Mono-sized spheres get packed in orthorhombic arrangement. The density becomes 62.5% of theoretical density.
- For a typical two component system with coarse 0.124 and fine 0.0065˝, the best packing was achieved for the weight ratio 77:23.

For ternary system the maximum density occurred at volume percentages 66:25:9 of size ratio-77:7:1. For a four component, the best density reported was 95.1% of theoretical density with diameter ratios 316:38:7:1 and volume compositions 60.7:23.0:10.2:6.1.

2.4 OBJECTIVE OF THE PRESENT STUDY

To study the effect of particle size distribution on properties of alumina refractories for six different batch compositions having different grain sizes distributions (i.e. coarse : medium : fine) and fired at different temperatures, namely 1550, 1600 and 1650^{o}C.

CHAPTER – 03

PROBLEM STATEMENT

1. To study the effect of particle size distribution on properties of alumina refractories at the six different batch composition or different ratio of alumina (i.e. coarse:medium:fine) at three different temperature.

   We have six batches which contains different wt% of tabular alumina (coarse), white fused alumina (medium) and reactive alumina (fine) as different aggregates containing six different ratio of coarse:medium:fine which helps to find the difference between the above batches after sintering at different temperatures, 1550ºC, 1600ºC and 1650°C. The soaking time at pick temperature in each process is 2 hours.

2. Determination of Shrinkage, Apparent Porosity, Bulk Density, CCS and observing the variation.

CHAPTER - 04

EXPERIMENTAL WORK

4.1 THE RAW MATERIALS USED

- Tabular Alumina (2-3mm)-Coarse
- White Fused Alumina (0.5-1mm)-Medium
- Reactive Alumina (10-50µm)-Fine

First of all 6 batches of 600 grams each were made in which different percentage of Coarse:Medium:Fine. The composition of above six batches is given here:-

1. 45:15:40
2. 45:10:45
3. 55:10:35
4. 40:30:30
5. 50:20:30
6. 45:25:30

4.2 COMPACTION INTO REFRACTORY BRICKS

First weighed amount of various alumina fractions (coarse, medium, fine) was taken in an enamel tray according to the required composition. Dry mixing was done for coarse and medium so that the mixture became uniform. After dry mixing and addition of 6% PVA (Poly Vinyl Alcohol) solution as binder. After mixing it for 5 min, the fine aggregates (powder) are added and mixed.

Now sub-batches of 100gm from each batch are separated to form bricks. Bricks was formed with a die pressed by Hydraulic Press (**Carver press USA)** at pressure 10 ton on 19.495 cm^2 surface area with a holding time of 1 min. Stearic acid is used as lubricant for die and punch.

The die, punch and base were cleaned with acetone. Stearic acid was used for lubrication of die and punch. Die was put on the base plate. Weighed amount of powder was put in the die. The punch was inserted and slightly pressed down to touch powder sample. Powder was pressed at 10 ton load. Pressed article was carefully taken by ejection process. The dimensions of the green brick were measured with the help of vernier caliper.

4.3 DRYING OF REFRACTORY BRICKS

After preparing the bricks, they were dried for 24 hrs at 110^0C in an oven.

After drying dimensions were measured for each brick using Vernier Caliper.

4.4 SINTERING OF REFRACTORY BRICKS

Now, from each batch 6 bricks were taken and sintered at 1550^0C, 1600^0C and 1650^0C with soaking time of 2 hrs. At each temperature 2 bricks were sintered from each batch. After sintering dimensions of the brick was measured using vernier caliper.

4.5 SHRINKAGE, BULK DENSITY AND POROSITY OF SINTERED BRICKS

Shrinkage is calculated by using dimensions of bricks. The bulk density and apparent porosity of the sintered bricks were determined by Archimedes principle using water. Dry Weight is measured and then the bricks were kept in distil water and then vacuuming is done for about 45 min-1 hr. After that suspended weight is measured using apparatus in which brick is suspended in water and weight is measured. After taking suspended weight, soaked weight is taken. Hence

the dry weight, soaked weight and suspended weight were measured. The shrinkage, bulk density and apparent porosity were calculated by the formulas:

- **Shrinkage:-**It is the

$$\text{Volume Shrinkage} = \frac{\text{Volume of green sample} - \text{Volume of fired sample}}{\text{Volume of green sample}}$$

$$VS = \frac{\text{VG} - \text{VF}}{VG}$$

Where,

V_G = Volume of green sample

V_F = Volume of fired sample

- **Bulk Density:-**It is the weight per unit volume of the refractory including the value of the open pore spaces.

$$\text{Bulk Density } = \frac{\text{Dry weight}}{\text{Soaked weight} - \text{Suspended weight}}$$

$$B.D. = \frac{D}{W - S}$$

- **Apparent Porosity:-**It is the percentage of the volume of the open pore spaces to the total volume of the sample.

$$\text{Apparent porosity} = \frac{\text{Soaked weight} - \text{Dry weight}}{\text{Soaked weight} - \text{Suspended weight}}$$

$$A.P. = \frac{W - D}{W - S} \times 100$$

Where,

W = Weight of the specimen in air including the moisture in its open pores, in grams

D = Weight of the dry sample, in grams

S = Weight of the specimen suspended in water, in grams

4.6 COLD CRUSHING STRENGTH OF SINTERED BRICKS

It is the ability to withstand structural load coming over them at normal temperature.

Cold crushing strength of the refractories has been measured by placing a suitable refractory specimen on flat surface followed by application of uniform load to it through a bearing block in a standard mechanical or hydraulic compression testing machine. The load at which crack appears the refractory specimen represents the cold crushing strength of specimen. The load is applied on the sample in the flat position. The load is applied uniformly during the test.

$$\text{Cold Crushing Strength} = \frac{\text{Weight withstand}}{\text{Cross section area}}$$

$$CCS = \frac{W}{A}$$

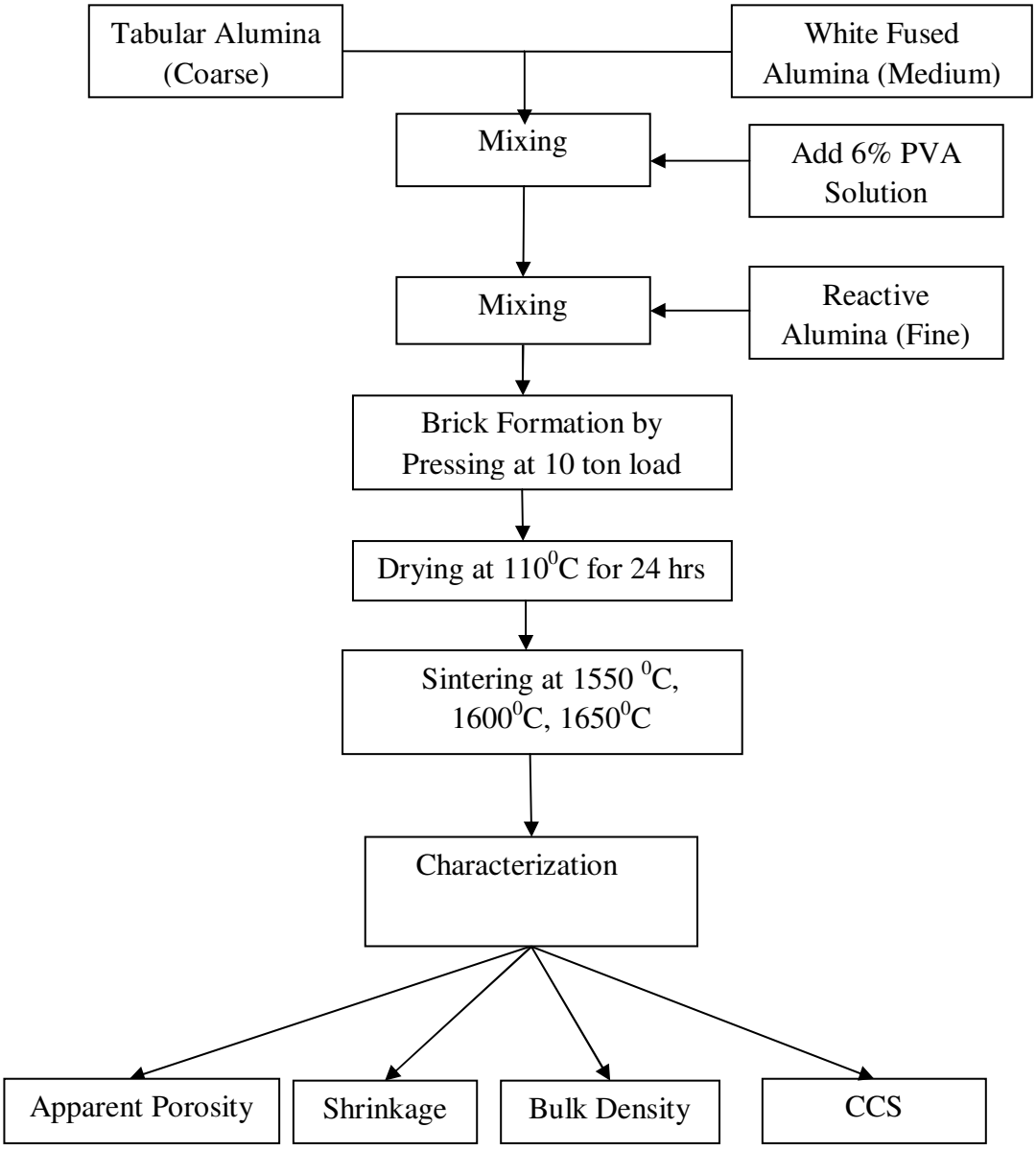

Figure 2 - Flow chart of the whole procedure

CHAPTER - 05

RESULTS AND DISCUSSION

5.1 SHRINKAGE

Temp	45:15:40	45:10:45	55:10:35	40:30:30	50:20:30	45:25:30
1550	1.204	1.93	1.152	0.841	0.505	1.281
1600	1.725	2.673	1.697	0.843	1.199	1.663
1650	2.167	3.489	3.495	1.87	2.275	1.753

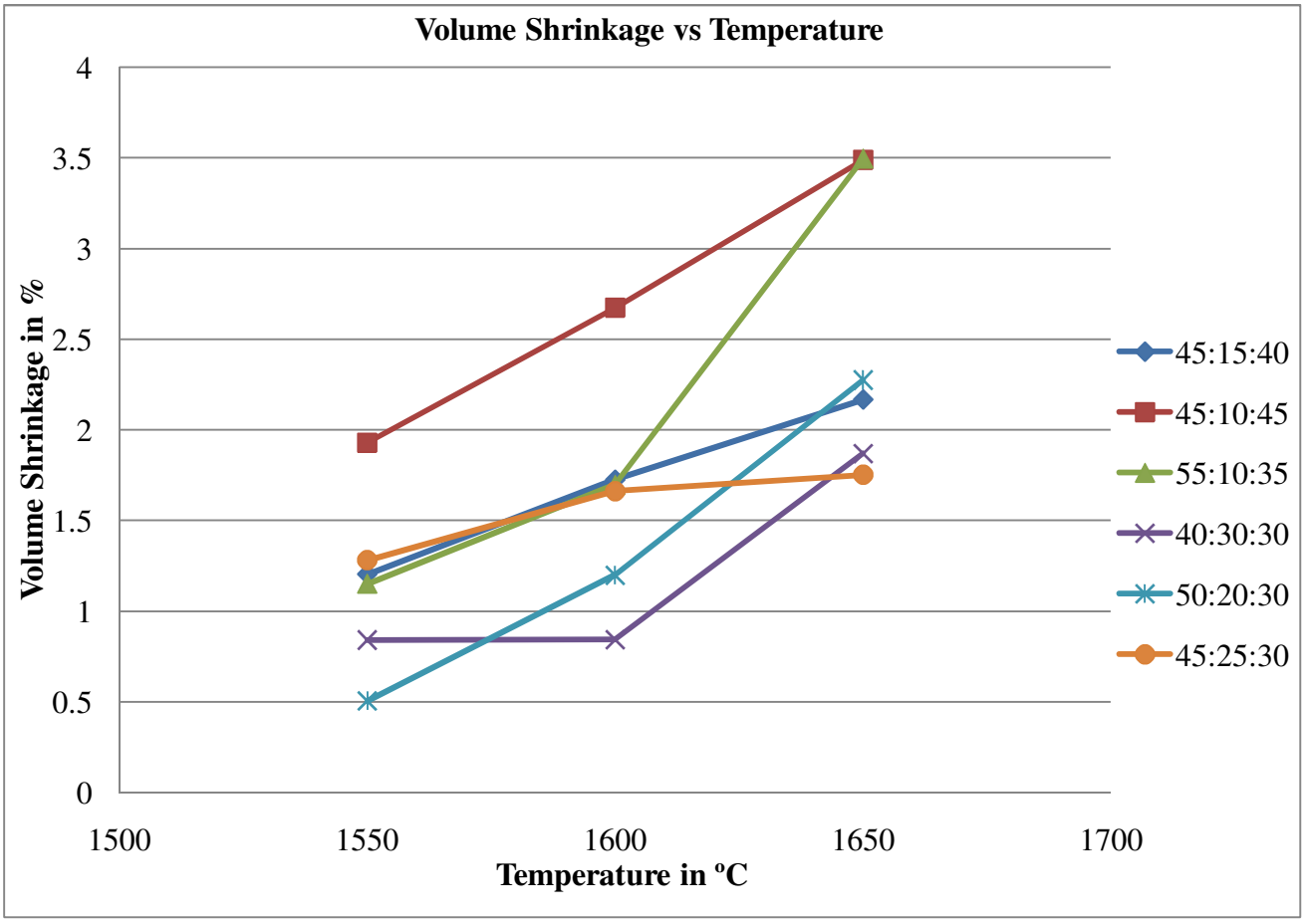

Figure 3-Volume Shrinkage vs Temperature

There is a general trend of increasing shrinkage value with increasing firing temperature for all the compositions. Batch containing composition 45:10:45 showed highest values for all the temperatures. Also lesser amount of fines shows relatively lower shrinkage value due to lesser extent of sintering.

5.2 APPARENT POROSITY

Temp	45:15:40	45:10:45	55:10:35	40:30:30	50:20:30	45:25:30
1550	27.507	26.34	26.878	25.148	27.061	22.891
1600	21.708	20.305	24.322	19.86	21.32	19.187
1650	17.863	14.691	21.133	18.729	21.289	18.576

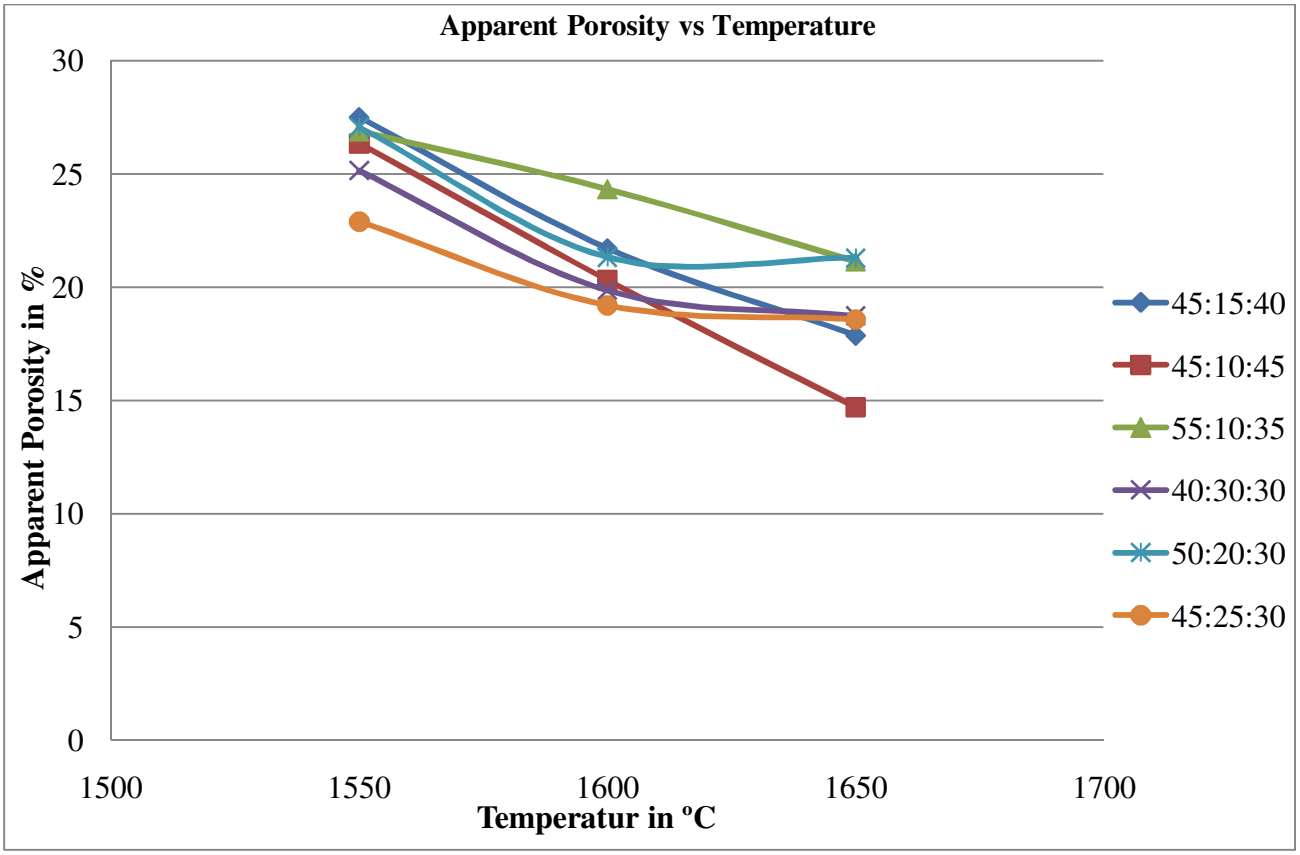

Figure 4- Apparent Porosity vs Temperature

There is a general trend of decreasing porosity value with increasing firing temperature for all the compositions. But no definite trend was observed with t he variation in the different batches. However batch containing 55:10:35 ratio shows always a higher porosity value for all t he temperatures, which may be due to the poor filling of the inter-granular voids by the fines in that bath as the batch contains highest amount of coarse and less fines.

5.3 BULK DENSITY

Temp	45:15:40	45:10:45	55:10:35	40:30:30	50:20:30	45:25:30
1550	2.707	2.799	2.681	2.671	2.504	2.805
1600	2.809	2.887	2.782	2.775	2.571	2.851
1650	2.841	2.941	2.826	2.92	2.651	2.889

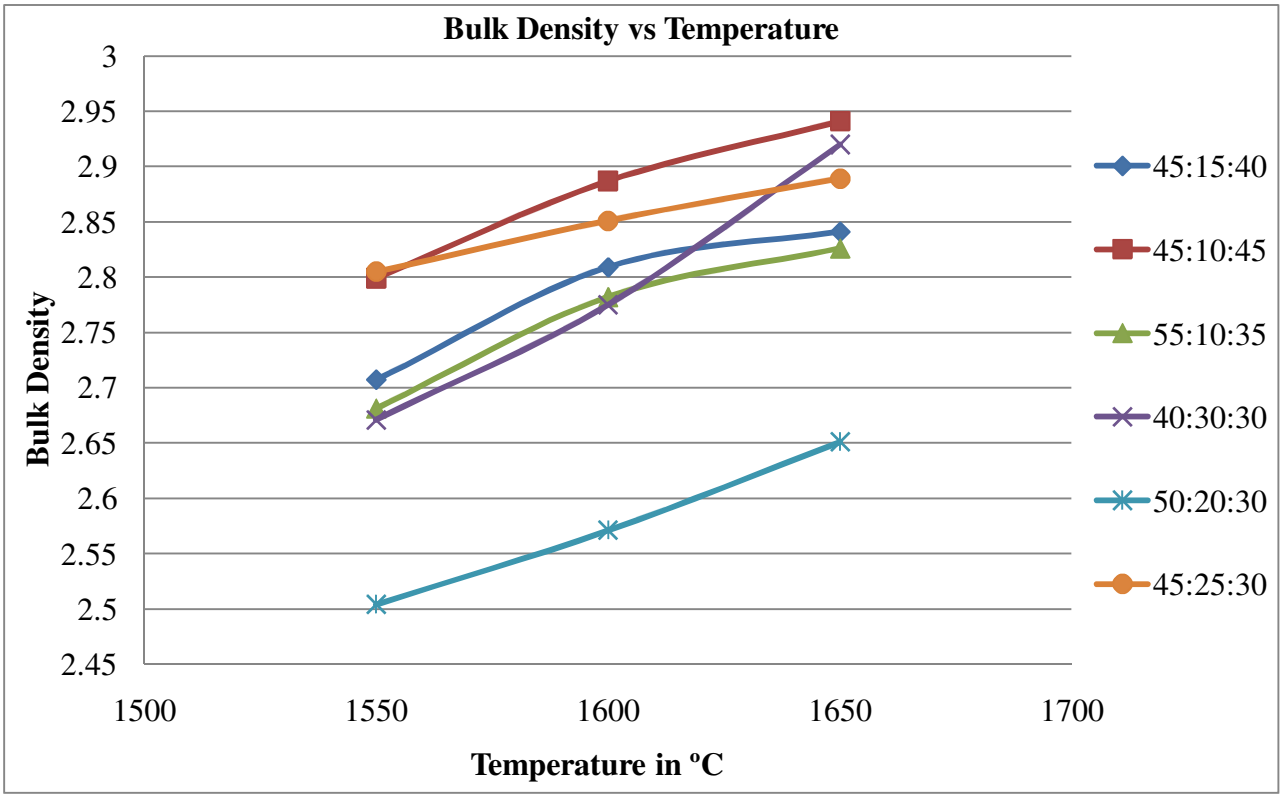

Figure 5- Bulk Density vs Temperature

There is a general trend of increasing density values with increasing firing temperature for all the compositions. Batch containing composition 45:10:45 showed distinct higher values for all the temperatures.

5.4 COLD CRUSHING STRENGTH

Temp	45:15:40	45:10:45	55:10:35	40:30:30	50:20:30	45:25:30
1550	647.488	683.728	557.256	541.931	547.9	536.108
1600	683.771	635.937	584.181	650.027	740.858	608.173
1650	678.141	756.461	687.892	616.26	752.975	727.802

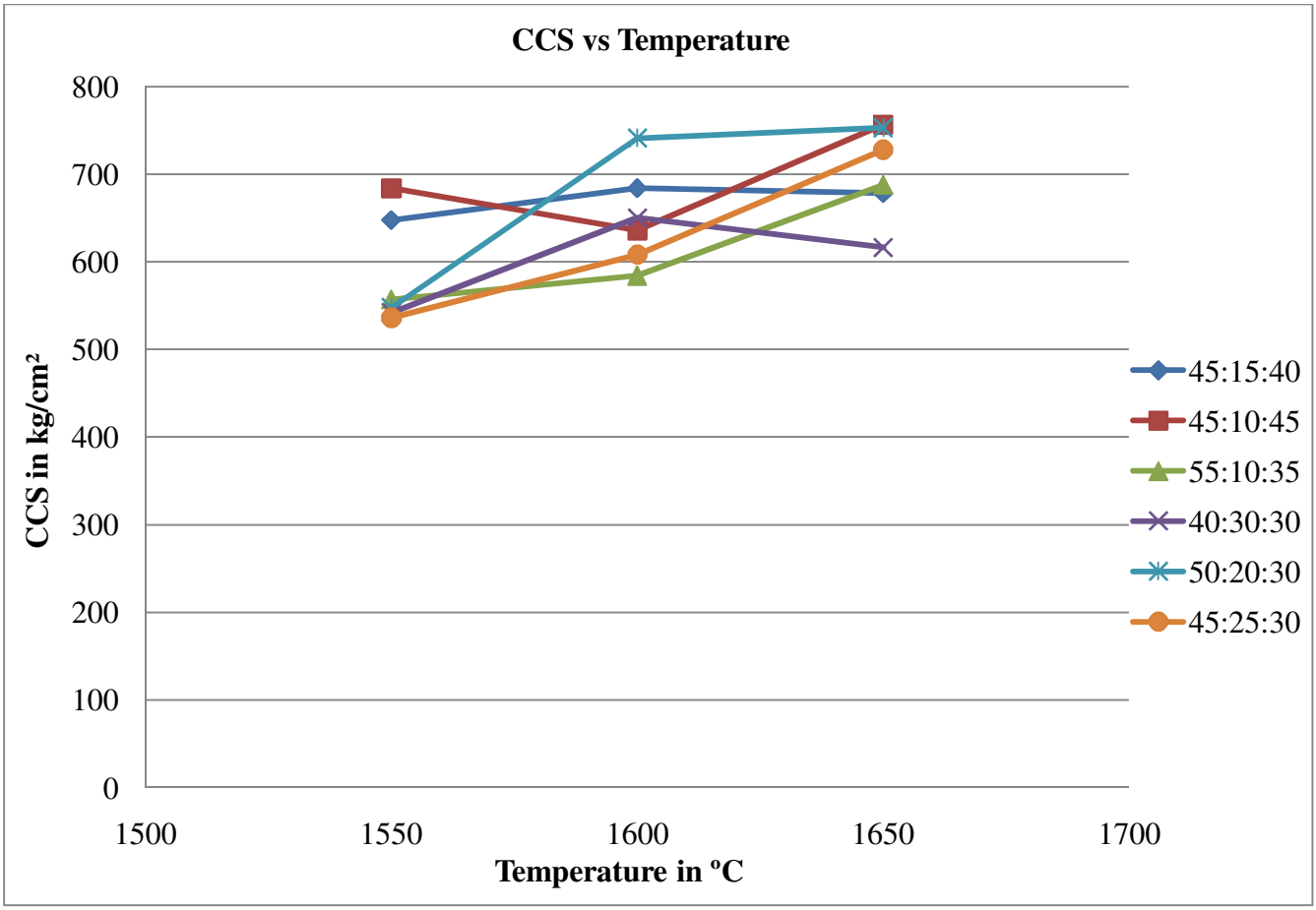

Figure 6- CCS vs Temperature

There is a general trend of increasing CCS values with increasing firing temperature for all the compositions. Batch containing composition 45:10:45 showed higher values. However only at 1600oC the batch containing 50:20:30 showed little higher value, which may require to be repeated.

CHAPTER - 06

CONCLUSION

1. In general there is increase in shrinkage, bulk density and strength, and decrease in apparent porosity is observed for all the batch compositions with increase in temperature.
2. The exact effect of grain size distribution, like increase or decrease of coarse/medium/fines is not very clear on the different properties.
3. 45:10:45 shows increased shrinkage, density and strength properties and decreased porosity.
4. Higher amount of coarse or lesser amount of fines has resulted in higher porosity level due to poor filling up of voids in between the coarse particles.
5. Packing of particles and its effect on the final properties are dependent on the grain sizes and their distribution. In the present study only one fixed grain sizes were used for coarse, medium and fine fractions and the above conclusion are made based on that. However for a complete study, it is required to vary the grain sizes of all the coarse, medium and fines and then compare all the results to identify the optimum distribution ratio for obtaining the best result.

CHAPTER - 07

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