

STUDY AND PREPARATION OF HIGH ALUMINA INSULATING CASTABLES

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

Bachelor of Technology
In
Ceramic Engineering
By

Pragati Gupta
Roll No. - 111CR0099

Under the supervision of

Prof. Ritwik Sarkar



Department of Ceramic Engineering
National Institute of Technology, Rourkela
2015



**National Institute of Technology
Rourkela, Odisha, India**

Certificate

This is to certify that the thesis entitled “ **Study and Preparation of high alumina Insulating Castables**” submitted by Ms. Pragati Gupta (111CR0099) in partial requirements of the award of Bachelor of Technology degree in Ceramic Engineering at 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 this thesis has not been submitted to any other Institute/ University for the award of any degree or diploma.

Date: 11.05.2015


Dr. Ritwik Sarkar

Associate Professor
National Institute of Technology
Rourkela, 769008

ACKNOWLEDGEMENT

With deep regards and profound respect, I avail this opportunity to express my deep sense of gratitude and indebtedness to Dr. Ritwik Sarkar, Associate Professor, Dept. of Ceramic Engineering, NIT Rourkela, for his inspiring guidance, constructive criticism and valuable suggestion throughout the project. I gratefully acknowledge his constant encouragement and help in development of my interests.

I am grateful to Dr. Swadesh Kumar Pratihari, HOD, Dept. of Ceramic Engineering nit rourkela, for providing me with an excellent setup and giving me all necessary facilities that helped me throughout the course of my work. I would also like to thank all the other Department faculty and staff who directly and indirectly helped me to complete my project.

Finally, I am grateful to my parents, lab assistants, PhD and M Tech scholars and last but not the least my friends who encouraged and helped me during the total period of time without whose support my project would not have been complete.



Pragati Gupta

Roll No. - 111CR0099

Department of Ceramic Engineering
National Institute of Technology, rourkela
Rourkela (769008)

OBJECTIVE

The major objective of this project is to develop an insulating castable which is a part of unshaped refractory. And study their change in bulk density, shrinkage behavior, cold crushing strength by applying different temperature range and how does low density (1.56 gm /cc) of castables affects its strength. X-RD analysis is also done for those samples which are exposed to 1500⁰C to determine different phases present in insulating castable.

CONTENTS

Chapter		Page No.
	Abstract	6
1	1. Introduction 1.1 Refractory 1.2 Unshaped refractory	7- 17 8-13 13-17
2	2. Literature review 2.1 Bonding and recent progresses in monolithic refractory 2.2 Refractory calcium aluminate cement/ high alumina cement 2.3 Insulating refractory	18-24 19-20 20-24 24
3	3. Experimental procedure 3.1 Raw materials used 3.2 Procedure	25-32 26-28 28-32
4	4. Result and discussion 4.1 Bulk density 4.2 Volume shrinkage 4.3 CCS measurement 4.4 X- ray analysis	33-40 34-35 35 36 37-40
5	5. Conclusion	41-42
6	6. References	43-45

ABSTRACT

Refractories are non-metallic inorganic materials. It can withstand high temperature load. It has many applications in glass, cement lime, iron and steel industries. Hence, unshaped refractories has taken place of shaped one. Castables are used as unshaped refractory in many areas. Refractory castables are prepared by mixing refractory grains, bonding agents, matrix components and additives by casting. We focuses here on the study and preparation of insulating castables. Insulating castables have many application in petrochemical industries and in process industries where it is used as a backup layer. It has very low density as well as low thermal conductivity that is why it is used in petrochemical and process industries to conserve heat energy and reduce fuel cost. Insulating castables are very brittle in nature, as it is made up of highly porous aggregates. Hence, it requires high water addition. To improve its strength slow vibration of 1.1 is done to reduce its water requirement. Different size range of the aggregates are used to make it more homogeneous. Cement is used as a binder in preparation of this castables.

CHAPTER-1

INTRODUCTION

1. INTRODUCTION

Ceramics are generally non-metallic, inorganic materials made up of metallic and non-metallic compounds bonded by covalent and ionic bonds^{1,3} Ceramic materials are inert and solid. Ceramic technology is the oldest technology more than 25000 years old and at the same time it is the most developing and modern technology. According to their method of preparation they can be classified as dense, lightweight ceramics. Ceramics have widest applications in many fields. It is classified in different classes amongst them refractories are of great importance.

1.1 Refractory

As per ASTM “refractories are inorganic non-metallic materials having those physical and chemical properties that make them applicable for structures and as components of system that are exposed to environment above 1000⁰ F”.² refractories are chemically and thermally stable at higher temperature that is why it has many applications in steel and glass manufacturing industries. Refractory constitutes the lining of the furnaces as it is heat resistant materials. Fire clays are generally used in the manufacturing of refractory.

Refractories are chosen according to the condition they are able to withstand. For example high alumina bricks are required for the preparation of slag lining in the blast furnace because for preparation of slag lining it is required to have corrosion resistant. Similarly for preparation of insulating castables SLA-92 is used as it has low density gives better insulating property.

The main requirements for refractories are

- high temperature withstanding character.
- must withstand high load / strength both at room temperature and high temperature.
- Corrosion resistant.
- abrasion/wear/erosion resistant.
- spalling resistant.
- thermal shock resistant.
- It should have low thermal conductivity as it should prevent the heat loss.

1.1.1 USES OF REFRACTORIES

Refractories are used as forming different linings in furnaces of different industries. In non-metallurgical industries refractories are used as it is installed on fired heaters, ammonia primary and secondary reformers, air heaters, coke calciner, hydrogen reformer, utility boilers, incinerators, catalytic cracking units, sulfur furnaces, ducting, stacks etc. Majority of these given equipments operate under high temperature (ranging from 900⁰ F to 2900⁰ F) and high pressure.

Thus the uses of refractories can be summarized as following.

- Glass industries
- Iron and steel industries

- Tiles industries
- Petrochemical industries
- Aluminium industries.
- Cement industries.
- Oil industries.

1.1.2 CLASSIFICATION OF REFRACTORIES

1.On the basis of Shape

- **Shaped refractories :** These types of refractories have different shapes and sizes. Which are further divided in special shapes and standard shapes as per the applications. Special shapes are made for particular furnaces and kiln whereas standard shapes have dimensions that are conformed by the most refractory manufacturers and these types of refractories are generally applicable to kilns or furnaces of the same types. These are usually fireclay bricks.

Example-Bricks

- **Unshaped refractories:** these types of refractories do not have definite shape and size. This class of refractory material is known as monolithic refractories. All unshaped refractories have the the property to form joint-less refractories which gives it better corrosion resistance and better life, complex shape can be casted or produced., and easy installation.

Example- Ramming masses, gunning masses, castables, mortar, fettling mix etc.

2. On the basis of chemical nature:

➤ **Acidic:** These type of refractories are used in those areas where atmosphere and slag are acidic in nature. These type of refractories get corroded in basic environment.

Examples- fire clay refractories, SiO₂ based refractories , alumina based refractories.

➤ **Neutral:** These types of refractories do not get affected by acidic and basic environment. These types of refractories are made from weakly acidic and basic refractories.

Example- Carbon based refractories, Zirconia based refractories, carborundum refractories etc.

➤ **Basic:** These types of refractories are used in those areas where atmosphere and slag both are basic in nature. These type of refractories get corroded in acidic environment.

Example- MgO based refractories, Dolomite refractories, CaO based refractories etc.

3. Based on heat duty:

These types of refractories are based on the % of Al₂O₃ content. Higher the heat duty better is the refractory.

These are classified as following:

➤ **Low heat duty refractories:** Al₂O₃ content 25-30%.

➤ **Medium heat duty refractories:** Al₂O₃ content 30%-35%.

➤ **High heat duty refractories:** Al₂O₃ content 35%-40%.

➤ **Super heat duty refractories:** Al_2O_3 content 40%-45%.

4. On the basis of porosity:

➤ **Porous refractories** :These types of refractories have low density and contain air pores which are used as a backup lining in petrochemical industries. Low density of these materials give high conservation of heat.

Example- Insulating castables.

➤ **Non- porous refractories:** These types of refractories have high density with low porosity. These type of refractories are used in working lining of blast furnaces. — —

5.On the basis of purity:

It depends on the presence of major composition in the refractories.

Example- Al_2O_3 -75%. it means 75% alumina is present in its composition.

1.1.3. Basic properties of refractories:

These can be classified as 3 different types

➤ **Physical properties**

- Apparent porosity
- Bulk density
- Strength
- Abrasion

➤ **Chemical properties**

- Corrosion
- Erosion

Thermal properties:

- Thermal Conductivity
- Thermal diffusivity
- Thermal shock

1.2 UNSHAPED REFRACTORIES

These type of refractory does not have any shape. Different sizes of materials are mixed with H₂O , and then pressed to form a single piece known as monolithics. Monolithic is the name given to all unshaped refractory materials which are introduced as some form of suspension that ultimately harden to form a solid mass. The regular examples of this types of refractories are plastic masses, Ramming masses, castables, gunning masses, fettling blend, mortars and so on.

It has many advantages:

- It reduces the joints or does not have any joints.
- Faster applications
- Better spalling resistance.
- Heat saving.
- Easy to handle, transport and install.
- Volume stability
- Reduced downtime for repair.
- Easy installation..
- Cheaper as no firing is required.

- Less skill requirement.

1.2.1 Different types of monolithic refractories:

- **Castable Refractories :** These are materials made from coarse, medium , and fine grains. They are bonded by a binder system. Generally HAC (high alumina cement) is used as a binder in this system. Different types of binders that are frequently utilized incorporate hydratable aluminas and colloidal silica. Castables are blended with water and afterward introduced by either pouring or pumping. Situation of the material then follows vibration.
- **Plastic Refractories :** These types of refractories are added with binder or tempered with water. These type of refractories have sufficient plasticity which can be rammed into place.
- **Ramming Refractories :** these type of refractories are same as plastic refractories but are much stiffer than plastic refractories.
- **Patching Refractories :** these are also same as plastic refractories though have a very soft plasticity allowing them to be rammed into place.
- **Coating Refractories :** Theses type of refractories are made to protect refractory lining usually against chemical attack. These are used to cover working surface of the lining , these are very thin in nature.
- **Refractory Mortars :** Mortars are made up of finely ground refractory materials

which are finally mixed with water to form a paste. They are generally used for bonding shaped refractory products such as bricks.

1.2.2 CASTABLES:

Refractory castables are prepared by mixing of different refractory grains of different sizes, matrix components and additives. The proportions of each component used are different for different castable compositions to achieve different targeted properties essentially required for various applications. Generally Calcium Aluminate Cement is used as a hydraulic binder in refractory castables compositions, but it reduces the liquid formation temperature of Alumina and Alumina-Silica refractory systems. The majority of castables are supplied as a dry mixture of different grains, fine and additives in bagged condition and during the time of installation it is mixed with a liquid (typically water) and poured, vibrated, pumped, or pneumatically placed to form a shape or structure. Because of hydraulic bonding or chemical setting they become rigid. The dewatering and curing steps must be carefully controlled to avoid explosive spalling.

In accordance to IS: 10570 classification of castables are as follows:

1. Conventional castables: Castables containing $\text{CaO} > 2.5\%$ on calcined basis

➤ **Dense castables-** These type of castables are produced by calcinated fireclay, high-alumina refractory aggregates, andalusite, bauxite, white-fused alumina and high-alumina cements. It is having higher density and is known for its strength and low maintenance.

➤ **Insulating castables-** These type of castables are made up of refractory light-

weight aggregates, special additives and high-alumina cements. these type of refractories are made up of lightweight aggregates. These type of refractory offer excellent consistency in density. These type refractories have low density and low conductivity, these properties help in conserving heat and reduce fuel costs, hence it is used as a back up layer in process industries and hot face layer in petrochemical industries. these are made by different size range of light weight aggregates to make it more homogeneous. Aggregates are highly porous in nature.

aggregates used in making insulating castables: vermiculite, perlite, extend-o-sphere, bubble alumina and expanded clay.

2.Low cement castables : Castables containing $1\% < \text{Cao} < 2.5\%$ on calcined basis.

3. Ultra low cement castables: Castables containing $0.2\% < \text{Cao} < 1\%$ on calcined basis.

4. No cement castables : Castables containing $\text{Cao} < 0.2\%$ on calcined basis .

1.2.3 Low cement and ultra low cement castables :

Addition of approximately 0.01 to 0.30 weight % deflocculants (such as alkali metal phosphates and carbonates) and 2.5 to 4wt% fine ($< 50\mu\text{m}$, but generally less than $1\mu\text{m}$) clay minerals result in the reduction of cement content without any reduction in strength.

Development of low cement castable was started with an aim to decrease the amount of water by promoting a homogeneous distribution of the cement so that the hydraulic bond could be fully utilized. Despite their better corrosion resistance than conventional castables, initially developed low cement castables was too sensitive to rapid heating. Chemically bonded water was released in a low

temperature range which results in explosive spalling. The internal water pressure built up during heating and outer layers closed off as installations of LCC's and ULCC's are dense materials with low permeability.

Low cement castables are useful for many applications including the aluminum furnaces, steel, iron and steel foundries and other non-ferrous metals, incinerators ,rotary kilns, and also for making precast shapes used in various high temperature applications.

CHAPTER-2

LITERATURE REVIEW

2.1 BONDING AND RECENT PROGRESSES OF MONOLITHIC REFRACTORIES:

The structural strength of a monolithic refractories is calculated by 3 forces i.e. force between the aggregates and bonding phase, the force of the aggregates itself, and the force of bonding phases itself.

2.1.1 HYDRAULIC BONDING :

Calcium aluminate cement, Silicate cement comes under this type of binder. Through the formation of hexagonal tabular or acicular, cubic granulars, alumina- gel and C_3AH_6 crystals.

2.1.2 CHEMICAL BONDING:

Sodium silicate, Phosphoric acid or phosphates and phenolic resins with hardener comes under these type of binder. It is formed by polymerization due to the chemical reaction between oxide aggregate refractory material and binder.

2.1.3 CERAMIC BONDING:

It is a low temperature sintering bonding, provided with metallic powder or fluxes i.e. borates, borax glass, metallic Si, Mg, and Al powders which lowers the sintering temperature. Hence this bond is formed.

2.1.4 ADHESIVE BONDING :

Organic binders are Phenolic resin, arabic gum, dextrin, sulfite pure liquor, polyvinyl alcohol and Vinyl polymer. By carbonization this binders form carbon bond.

2.1.5 COAGULATION BONDING :

These type of binders include ultrafine oxide powder, fine clay powder, alumina sol and silica sol. To overcome the repulsive force by attractive force electrolytes are added.

2.2 Refractory Calcium aluminate cement / High Alumina Cement

2.2.1 Cement:

Cement is a type of hydraulic binder. It gives the binding property by hydraulic reaction with water. It contains inorganic material, finally ground when mixed with appropriate amount of water forms a paste of cement and water and that shows setting and hardening properties and that paste shows through hydraulic reaction and then produce strength . Even retain its strength under water.

Calcium aluminate refractory is amongst the most widely used refractory now a days.

- **Portland cement-** it is cheapest of all cement, but because of undesirable phase changes that occur during heating it is limited to be used as a refractory material.

It has 4 major phases i.e. C_3S , C_2S , C_3A , C_4AF .

- **Cement fondu-** It is used as an alternative of portland cement. It does not undergo undesirable phase changes. It has low PCE value, highly acid resistant cement.

2.2.2 Calcium aluminate cement:

Calcium aluminate cement is a special hydraulic cement, which is differentiated from ordinary Portland cement by its high performance properties such as high chemical resistance, high corrosion resistance, slow setting but very rapid hardening, high resistance to acid attack and high refractory properties. Bauxite and calcareous materials are the main raw materials of calcium aluminate cement. The chemical composition of CAC containing Al_2O_3 which ranges between 40% and 80%.

Calcium aluminate refractory offers an advantage compared with conventional refractories in terms of flexibility, cost and speed of application.

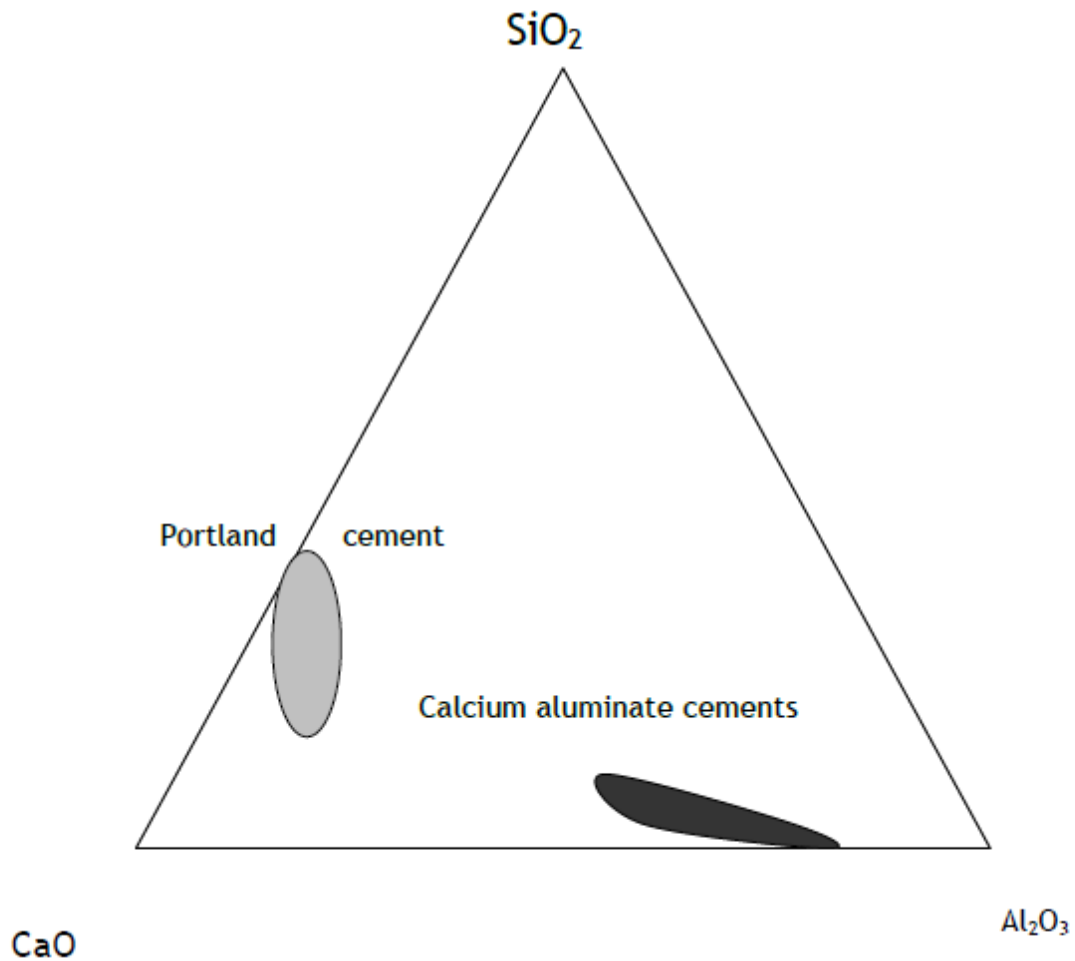


Fig-2.1. Composition range of CAC compared to portland cement in triple phase diagram of (CaO, Al_2O_3 , and SiO_2).

2.2.3 Different Phases present in CAC:

Major phase is CA (Calcium aluminate), and other minor phases present in CAC are CA_2 (calcium di aluminate), C_{12}A_7 (dodeca calcium hepta aluminate), C_4AF (tetra calcium alumino ferrite), C_2S (di calcium silicate), and C_2AS (di calcium alumino silicate).

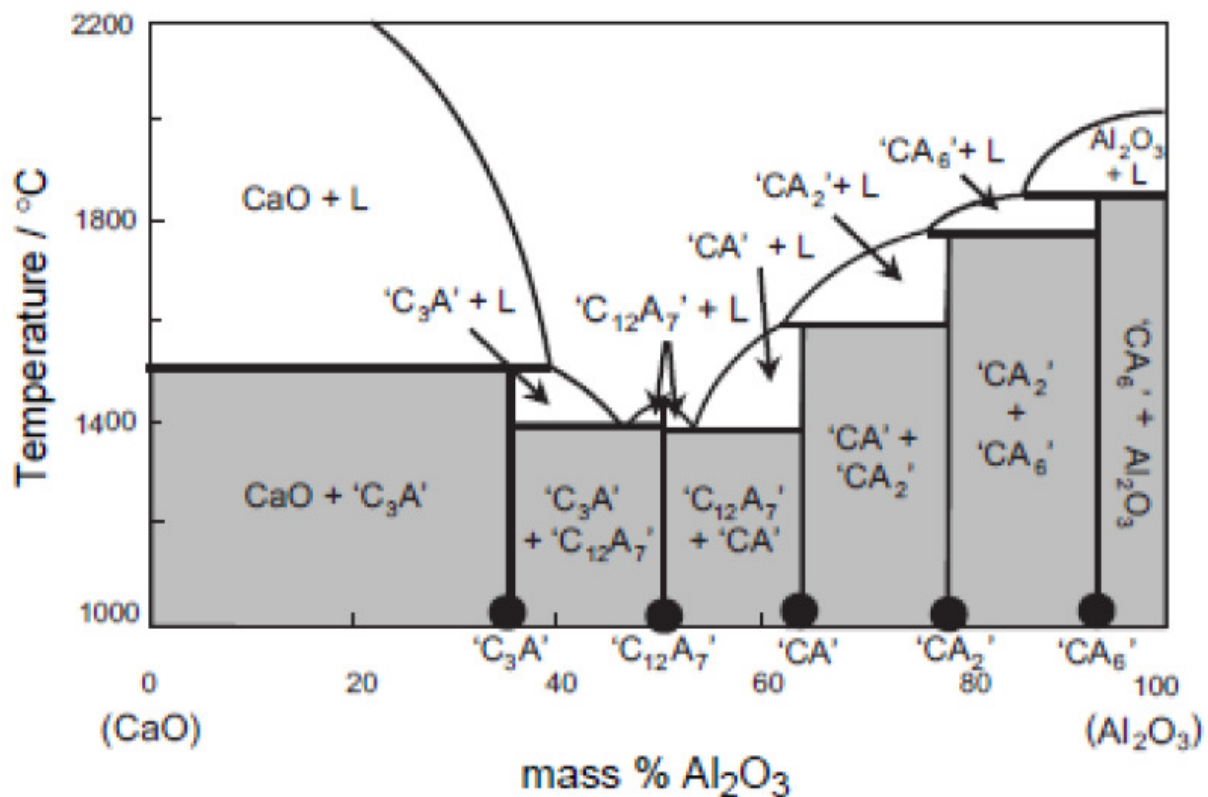


Fig. 2.2 Phase diagram of CaO and Al₂O₃ system

2.2.4 Hydration behaviour of CAC:

Hydration converts the anhydrous cement powder into different phases. It is a reaction between different phases of CAC and water, which is responsible for strength development in cement. During firing of these hydrated phases break down occur, leaving very reactive products, which later re-crystallizes to anhydrous Calcium aluminates. This anhydrous calcium aluminate produces a framework of sintered material at a temperature much lower than that which would have been required to sinter the anhydrous calcium aluminate powder. It gives the information about the nature of Calcium aluminate hydrates, the way in which they are broken and their morphology.

C_3AH_6 (a form of hydro garnet) and $\gamma-AH_3$ (gibbsite) is the most stable hydrates at all temperature. However the development of these steady hydrates happens through the nucleation of metastable stages CAH_{10} , C_2AH_8 and indistinct stages. The change of metastable stages into stable stage is called 'conversion'.

TABLE 2.1 HYDRATION REACTIONS OF CA PHASE ARE AS FOLLOWS:

TEMPERATURE RANGE	REACTION
$T < 20^{\circ}C$	$CA + 10H \rightarrow CAH_{10}$
$20^{\circ}C < T < 30^{\circ}C$	$2CA + 11H \rightarrow C_2AH_8 + AH_3$
$T > 30^{\circ}C$	$3CA + 12H \rightarrow C_3AH_6 + 2AH_3$

- CAH_{10} and alumina-gel gets dehydrated at approximately $100^{\circ}C$.
- C_2AH_8 breaks around $1000C$.
- Both converts to C_3AH_6 .
- $C_3AH_6 \rightleftharpoons C_{12}A_7$ at temperature $300^{\circ}C$ approximately.
- $C_{12}A_7 \rightleftharpoons CA$ at temperature greater than $900^{\circ}C$

HYDRATION OF CA_2 - It is a minor cphase present in many calcium aluminate cement.

At $T \sim 20^{\circ}C$ CAH_{10} , C_2AH_8 , Gibbsite, and alumina gel is formed .

At $T > 40^{\circ}C$ C_3AH_6 and AH_3 is formed.

Due to the reaction of Al_2O_3 and dehydration products of calcium aluminate hydrates amount of CA_2 increases. By adding CA or $C_{12}A_7$ reactivity of CA_2 can be increased.

Due to exothermic reaction at increasing temperature more reactive phases hydrates at faster rate.

Thus at higher temperature rate of reaction increases.

HYDRATION OF $C_{12}A_7$ -

In refractory calcium aluminate it is the most reactive phase found. Even in the presence of gypsum it may flash set.

2.3 INSULATING REFRACTORIES :

Refractory manufacturers mark another advancement in furnace construction by developing insulating refractories. Its value has been already proved in a variety of applications. These type of refractories is recently being used in a continuous automatic furnaces, full line of small standard furnaces, and in both muffle and direct fired type of car bottom and annealing furnaces.

Fire clay refractory bricks also have high insulating value. These bricks are light in weight and soft in nature and can be easily cut. It can withstand high temperature. Because of its high insulating value and light weight it is being preferred for a thinner furnace wall and has low cost too. But they contain different impurities which sometimes adversely affects its refractory applications. Insulating fire brick at elevated temperature allows for self supporting structures. To prevent the heat loss these refractories work well as they have a very low thermal conductivity. These insulating fire bricks used on the outer side of the walls, under the slab and floor bricks. These are recommended for use as primary hot face refractory linings as back-up insulation behind other refractories in furnaces, kilns. flues and similar other high industrial equipment.

CHAPTER-3

EXPERIMENTAL PROCEDURE

3.1 RAW MATERIALS USED:

1.SLA-92 (Super light-weight aggregates) in the size range of

- 3-6
- 1-3
- 0-1

It is used as a main aggregates in preparing insulating castables. It has high porosity, in the range of 70%-75% porosity. So it has low density and high water demand. It is a CA_6 based raw materials. The key properties of these CA_6 based insulating aggregates are high purity, high refractoriness, low thermal conductivity at above $1200^{\circ}C$ and high thermal spalling resistance. In phase analysis SLA-92 has CA_6 about 90% with only minor amount of corundum and CA_2 . Use of these materials will help the environmental friendly solutions and energy saving concepts which are increasingly become the focus of attention for all the industries.

Different size range of the SLA-92 is given to increase its homogeneity and to increase its strength.

CHEMICAL ANALYSIS (MASS %) of SLA-92 ARE:

Table: 3.1

Al_2O_3	91.00%
CaO	8.50%
SiO_2	0.07%
Fe_2O_3	0.04%
Na_2O	0.04%

2.Cement- Cement is used as hydraulic binder. As water demand of SLA-92 is very much higher because of 70-75% of porosity, Cement is used to reduce its high water demand and it improves the

strength of the castables. Main phases present in it is CA and CA₂.

3.WTA (white tabular alumina)- It contains 100 % of alumina. It is formed in ‘ball’ like shape prepared by pressing the calcined alumina obtained from Bayer’s then it is sintered at very high temperatures of 1900-2000 degrees Celsius. The resultant product is called tabular alumina as it is formed in large ‘tablet’ like structures that’s why it is called 'tabular alumina'. There is an alignment of the tabular alumina crystals which provide a large strength to the insulating castables. This gives excellent mechanical strength and abrasion resistance to insulating castables . It also reduces the high water demand of insulating castables.

4. SHMP (Sodium Hexa Meta Phosphate) – It is used as a deflocculant in the mixer.

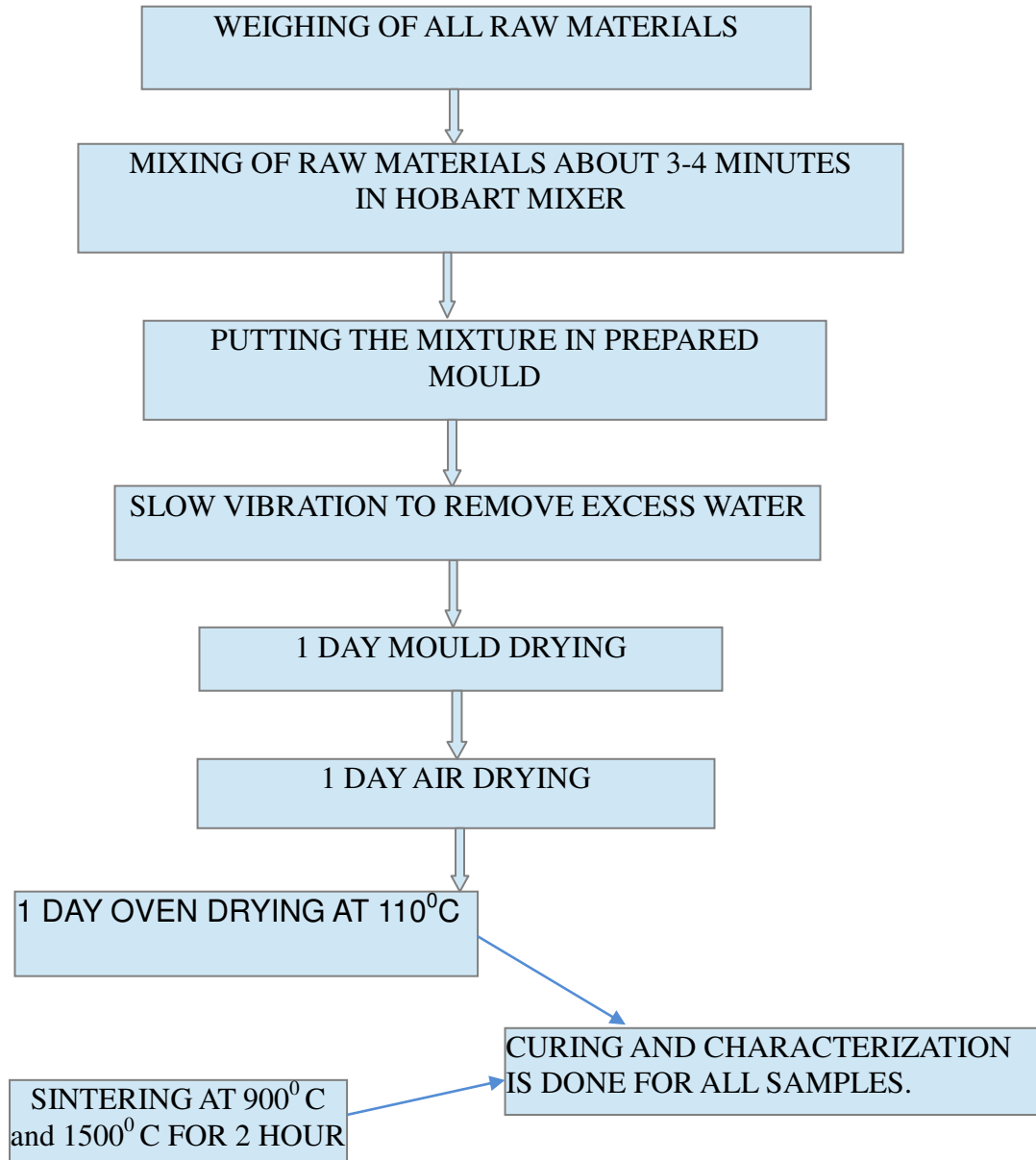
5. Citric Acid- It is used as a plasticizer in the mixer. It used to increase homogeneity and to avoid bleeding of the mix.

3.1.1 BATCHES OF EACH COMPOSITIONS USED (in wt%):

Table 3.2

RAW MATERIALS	I st batch (in 500 gm)	II nd batch (in 500 gm)	III rd batch (in 500 gm)	IV th batch (in 500 gm)	V th batch (in 600 gm)
SLA-92					
3-6	40%	20 %	30%	30%	10%
1-3	20 %	40 %	30%	10%	30%
0-1	10 %	10%	10%	10%	10 %
Cement	20.00%	20.00%	20.00%	20.00%	20.00%
WTA fines	10.00%	10.00%	10.00%	30.00%	30.00%
SHMP	0.50%	0.50%	0.50%	0.50%	0.50%
Citric acid	0.10%	0.10%	0.10%	0.10%	0.10%

3.2 PROCEDURE :



3.2.1 RAW MATERIAL COLLECTION:

Different raw materials like SLA-92 were taken from Almatris company in the size range of 3-6, 1-3, 0-1 and, white tabular alumina, cement (CA25R) is taken.

3.2.2 CASTING AND SAMPLE PREPARATION :

Batches of given raw materials as given in the table 3.2 is prepared.

The batch mixing was done in a Hobart Mixer (as shown in fig. 3.2) for 10-15 minutes, until a homogenous batch was prepared. Required amount of water was added to the batch during batch mixing and then it was homogeneously mixed. Then SHMP and Citric acid as given in the table 2 are mixed together then poured in the mixer to make it more homogeneous and then the batch was casted in 50mm X 50mm X 50mm cubic steel molds (as given in fig. 3.3) .



Fig. 3.2 Hobart Mixer



Fig. 3.3 Steel Mold

to keep the temperature under control casting different batches, molds were covered with a wet cloth The molds were left untouched as such for 24 hours and then wet cloths are removed and mold

drying is done for 24 hrs after that demoulding was done. The castables formed were then kept open for air drying for another 24 hours and then it is dried at 110°C in an oven. After 24hrs it is taken out from oven and weight and their dimension is measured from Weighing machine and Vernier Callipers respectively.

3.2.3 FIRING:

There are 3 samples for each batches of 5 (I, II, III, IV, V) it is marked 1/I, 2/I, 3/I, 1/II, 2/II, 3/II, 1/III, 2/III, 3/III, 1/IV, 2/IV, 3/IV, 1/V, 2/V, 3/V.

- 2/I, 2/II, 2/III, 2/IV, 2/V samples are kept at 900°C .
- 3/I, 3/II, 3/III, 3/IV, 3/V samples are kept at 1500°C .

3.2.4 DETERMINATION OF DENSITY AND VOLUME SHRINKAGE :

BULK DENSITY- The weight of all the dried samples were taken by weighing machine and its dimensions were measured with the help of vernier callipers and its volume (length x breadth x height) is calculated. Bulk density is then calculated by dividing dried weight by samples dimensional volume. It is taken average for all samples.

Bulk Density=weight/ (length x breadth x height) gm/cc.

VOLUME SHRINKAGE-

Volume shrinkage is measured by following

$$(\text{Volume at } 110^{\circ}\text{C} - \text{Volume at } 900^{\circ}\text{C} \text{ or Volume at } 1500^{\circ}\text{C}) / \text{Volume at } 110^{\circ}\text{C} .$$

3.2.5 DETERMINATION OF CCS (COLD CRUSHING STRENGTH):

CCS is the measure of maximum compressive stress required to break a material. It was

measured by using the machine as given in fig. 4

CCS = maximum load at which fracture occurs / area of the sample.



Fig 3.4 Testing machine for CCS measurement

3.2.6 XRD FOR PHASE ANALYSIS:

To determine the phases present in the fired castables XRD analysis was done. The main aim was to observe whether calcium aluminate, alumina, calcium silicate is present in the castables or not. XRD measurements were performed at a $10^{\circ}\text{C}/\text{min}$ scan rate using a Rigaku, Japan make x-ray diffractometer (as shown in fig. 3.4) and with diffraction angle 2θ range of 20° to 60° .

Samples after firing at 1500°C were first crushed to powder and collected and then sent for X-ray

analysis. The fining of the particle size was done to expose more number of atoms to the incoming X rays so that a sufficient intensity could be obtained after diffraction. The X-ray analysis data was matched with the standard JCPDS software to identify the phases.



Fig. 3.4 X-Ray Diffractometer

CHAPTER – 4

RESULTS AND DISCUSSION

4.1 BULK DENSITY: It is taken average of all samples for each batches.

BATCH NO.	BD (gm/cc) at 110 ⁰ C	BD (gm/cc) at 900 ⁰ C	BD(gm/cc) at 1500 ⁰ C
I	1.17	1.16	1.18
II	1.22	1.17	1.2
III	1.18	1.17	1.2
IV	1.3	1.28	1.44
V	1.38	1.29	1.46

Sample V has the highest bulk density as WTA fines content is higher than I, II, III. In I, II, III SLA-92 content is higher but it is a porous material so their density is also lower in case of I, II, III. WTA fines content in IV and V is same, but SLA-92 in the size range of 1-3 is higher than 3-6 in case of Vth batch. So it makes it more homogeneous and its density is also higher. Same applies with I, II, III batches. So density is in the order of I<III<II<IV<V.

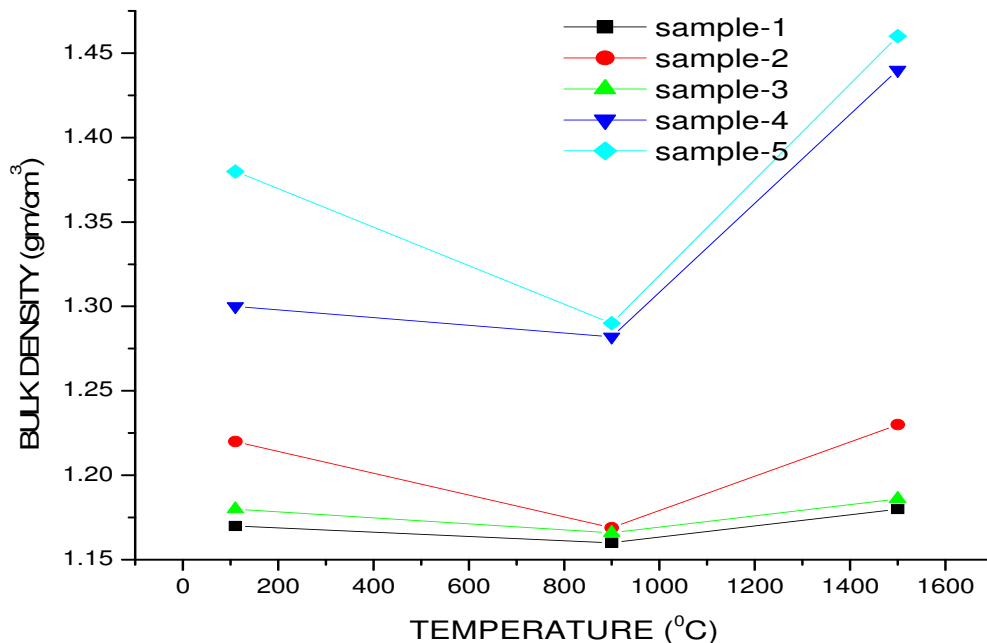


Fig. 4.1 Comparison of Bulk Density for all 5 batches (I, II, III , IV, V)

Bulk density is higher at 110⁰ C and then lowest at 900⁰ C, and then again increases and highest at 1500⁰C. Reason for this change is as following.

- Cement contains CA and CA₂ as main phases.

Hydration reactions of CA is given in the table 2.1. CAH₁₀ and AH₃ gets dehydrated at 100⁰ C .

C₂AH₈

breaks at approximately 100⁰C . both converts to C₃AH₆ . C₃AH₆ at approximately 300⁰C gets converted to C₁₂A₇. Total hydraulic bond lost at above 600⁰C. So its strength is lower at 900⁰C. But at above 1400⁰C it forms another ceramic bond i.e. CA gets converted to CA₂ and CA₂ to anorthite (Ca₀.SiO₂.2Al₂O₃). Due to sintering temperature which make it very much stronger as ceramic bond is stronger than hydraulic bond and no bonds. So its density is higher at 900⁰C, Highest at 1500⁰C and lower at 900⁰C.

4.2 VOLUME SHRINKAGE:

Table 4.2

BATCH NO.	SHRINKAGE AFTER 900 ⁰ C	SHRINKAG AFTER 1500 ⁰ C
I	1.82%	2.27%
II	0.68%	0.78 % [^]
III	1.20%	1.50%
IV	0.65%	0.77%
V	0.60%	0.74%

As we can see from the table 4.2 shrinkage decreases with increasing density .

So its order is V < IV < II < III <I. As higher is the density higher will be the strength and lower is the shrinkage.

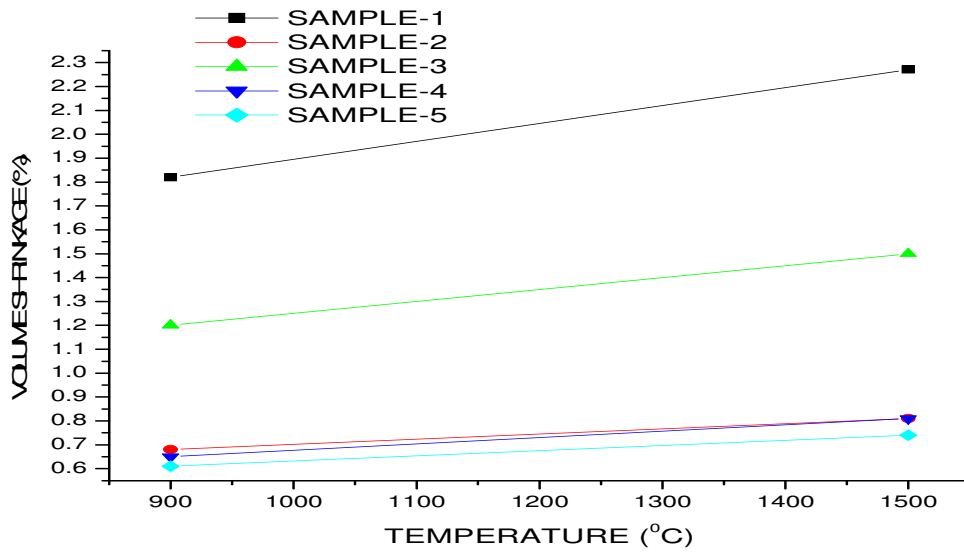


Fig 4.2 Shrinkage behaviour with increasing temperature.

4.3 CCS (COLD CRUSHING STRENGTH) :

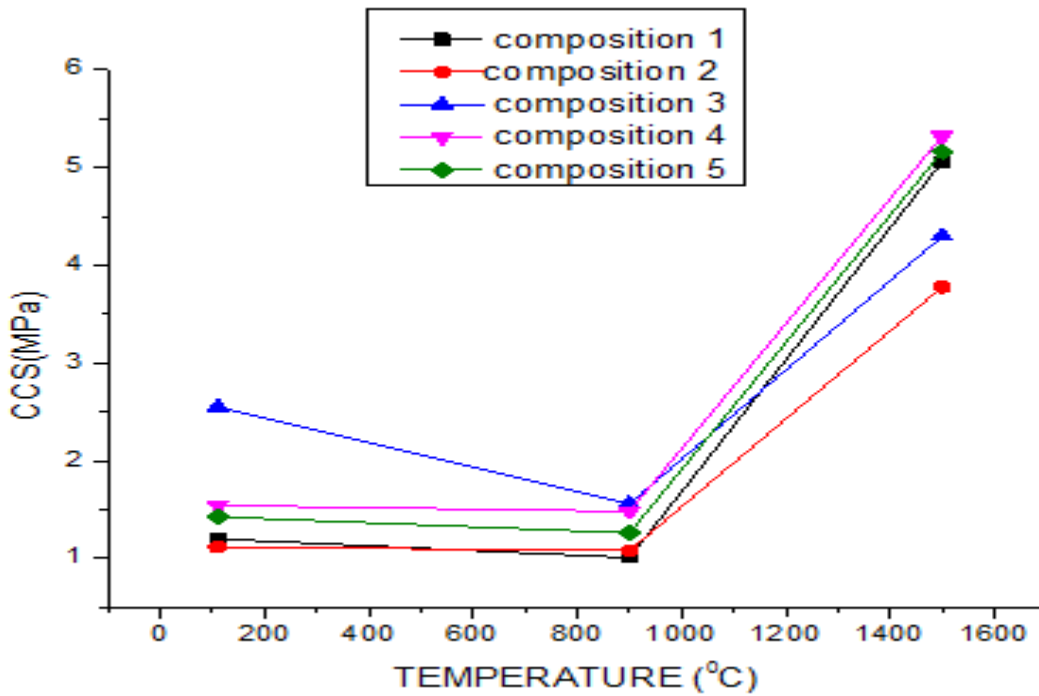


Fig. 4.3 CCS behavior of all 5 batches with increasing temperature

Table 4.3

SAMPLES	PEAK LOAD (KN)	CCS (MPa)
1/I	3.03	1.2
2/I	2.5	1.01
3/I	12.66	5.06
1/II	2.87	1.12
2/II	1.73	1.08
3/II	9.5	3.78
1/III	6.47	2.55
2/III	3.87	1.56
3/III	10.86	4.3
1/IV	4.17	1.54
2/IV	3.73	1.48
3/IV	11.38	5.32
1/V	3.79	1.43
2/V	3.11	1.26
3/V	12.92	5.16

CCS is the measurement of maximum compressive stress required to break a material. It is calculated as the maximum load per unit area that the sample can take before breakage, and the formula is load/area in kg/cm². Here we have measured CCS for all samples. It is observed that at 110⁰ C the CCS value was higher due to presence of hydraulic bonding in the composition , and at 900⁰ C hydraulic bonding totally disappear which results in the formation of CA. Hence at this temperature CCS value is lower than 110⁰ C . At 1500⁰ C CCS value is highest because at this temperature another ceramic bond appear which is stronger than hydraulic bond due to sintering.

4.4 XRD ANALYSIS:

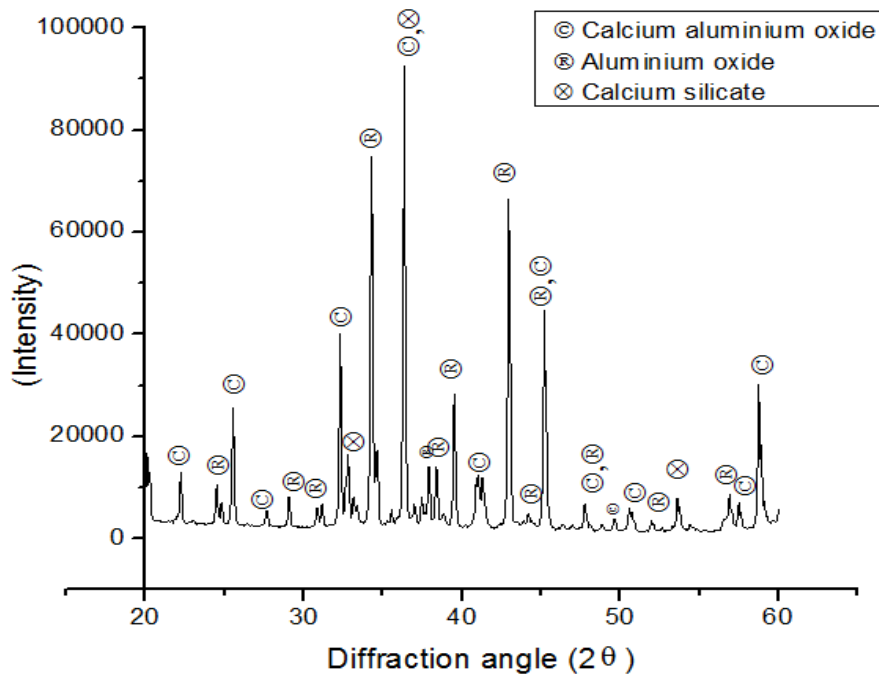


Fig.4.4 X-ray analysis for sample 2 (1500⁰ C)

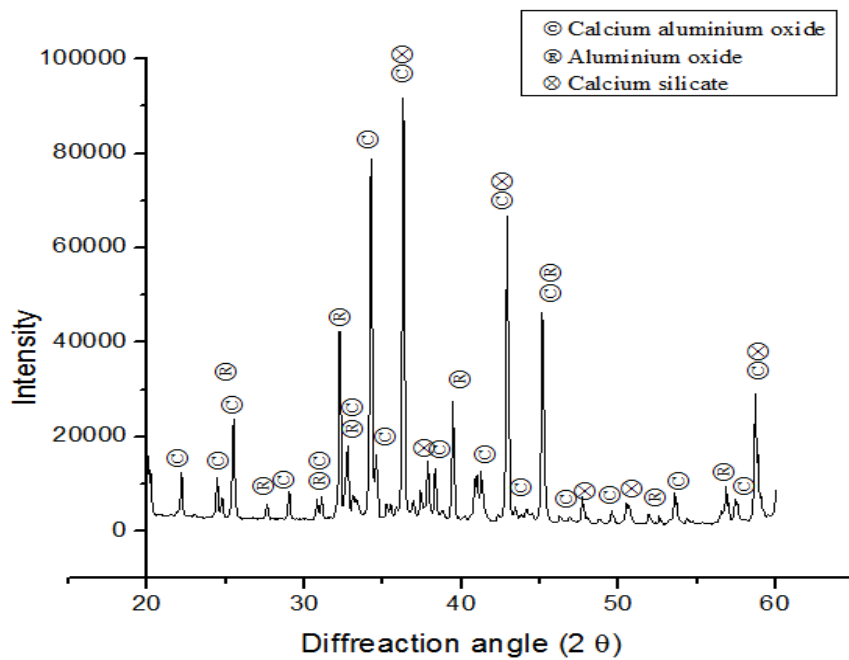


Fig.4.5 X-ray analysis for sample 3 (1500⁰ C)

F

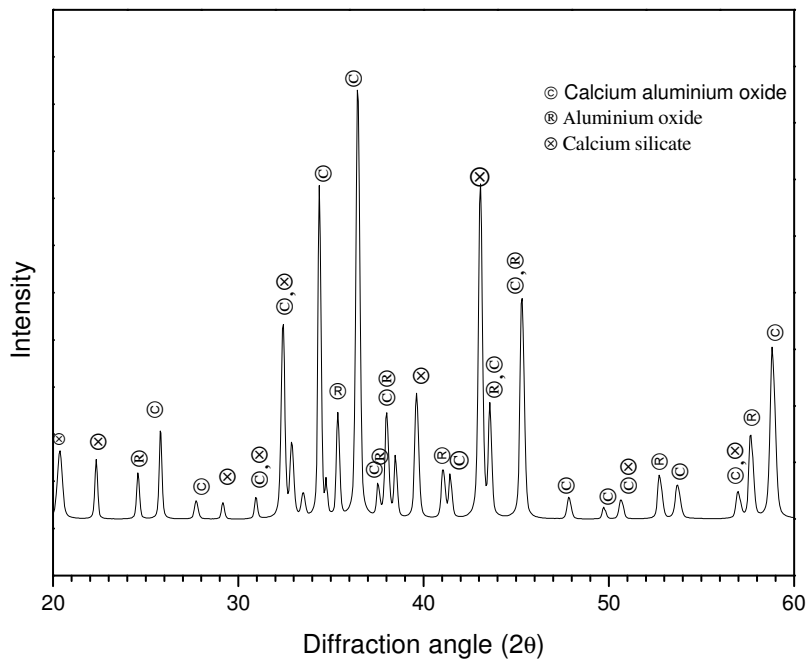


Fig.4.6 X-ray analysis for sample 4 (1500⁰ C)

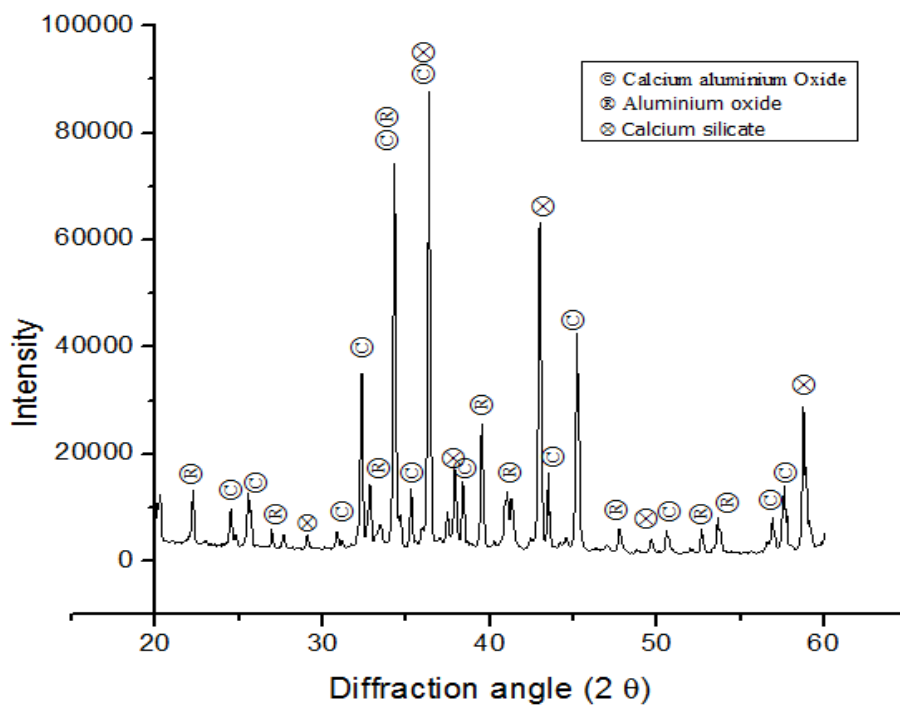


Fig.4.7 X-ray analysis for sample 5 (1500⁰ C)

X-ray diffraction analysis is done to know the phases present in the powdered sample. It is used to know the orientation of grains or single crystal and to know crystal structure of unknown material present in it. X-ray analysis is done for 4 samples which was fired at 1500°C i.e. for 3/II, 3/III, 3/IV, 3/V. It is observed that in fig.4.4, fig. 4.5, fig. 4.6 and fig. 4.7 the major phases present are CA_2 , CA_6 , alumina and calcium silicate.

CHAPTER-5

CONCLUSION

CONCLUSION

- Insulating castables concept based on light- weight aggregate SLA-92.
- Different raw materials cement and white tabular alumina is added to give strength to this insulating castable.
- It is popular because of its high heat storage value, environmental friendly and low cost of operation because of its low density (range between 1.2 to 1.5) and its low thermal conductivity.
- Density , CCS value decreases at 900⁰ C because of loss of hydraulic bonding. Hydraulic bonding present at 110⁰ C. Density and CCS value is highest at 1500⁰ C because a stronger ceramic bond is present there due to sintering.
- Volume shrinkage is calculated. XRD analysis is done to know the phases present in this insulating castables.
- Disadvantage of this refractory is that it is very brittle in nature.

CHAPTER-6

REFERENCES

REFERENCES

- 1..ASTM Volume 15.01 Refractories; Activated Carbon, Advanced Ceramics.
- 2.Abstracted from International Ceramic Monographs, Vol. 1, no. 2, pp. 772-93, 1994- Edwin Ruh .
3. “Lets make a castable”, by Bjorn Myhre, Refractories Applications & news Vol.3, no. 3-4 (2008)
- 4.Refractory Calcium aluminate cements- (Mrs. K.M. Parker,J.H. Sharp). Dept. of Ceramics Glasses and polymers, University of Sheffield.
5. Bonding and recent progresses of monolithic refractories (Z.Li*, G. ye*, P.R. China) vol no. 3
6. High performance refractory castables (R.G. Pileggi, A.R. Studart, M.D.M Innoanhini) vol 81.
7. Refractory castables an overview (P.pilate), (Belgium Ceramic Research Centre)
8. W. D. Kingery, “Factors Affecting Thermal Stress Resistance of Ceramic Materials,” by J. Amer. Cer. Soc., 38 (1) 3-15 (1955).
9. Insulating Refractories by W.M. Hepburn.
10. Refractory Metals and Hard Materials Impact Factor & Information, Publisher: International Plansee Society for Powder Metallurgy; International Tungsten Industry Association, Elsevier.
- 11.The Accelerated Drying of Refractory Concrete – Parts I and II, Volume 6, Issues 2 and 4 /The Refractory Worldforum
12. W. Vieira, W. E. Lee, K. G. Ahari,S. Zhang, C. Parr and H. Sarpoolaky, —Castable Refractory Concrete, *Intern. Mat. Rev.*, **46** [3] 145-167 (2001).
13. “Recent developments in Monolithic refractories”, by Subrata Banerjee, ACSB(1998).
14. US Patent 4762811 (1988).

15. Insulating Brick, retrieved 2012-07-04.
16. Modern Refractory Practices, Fifth Edition (Harbison–Walker Refractories: Pittsburgh, Pennsylvania, 1992), Page CR-2 , CR-3
17. Refractory Engineering. [ISBN 3-8027-3155-7](#). Die Deutsche Bibliothek. 2004.
18. The Hydration Products of a Refractory Calcium Aluminate Cement at Intermediate Temperatures, Nilforoushan, Mohammad Reza*; Talebian, Nasrien.
19. US Patent 5681786(1997)
20. “Calcium aluminate cement based castables for demanding applications”, presented at 1st Monolithics Conference, C.Parr, Thomas A. Bier, (1997).
21. “High alumina self-flow castable with different binders”, Ritwik Sarkar and Akash Satpathy, Refractories World Forum, 4 [2] 129-133 (2012).
22. A. Bhatia, B.E.: ‘Overview of Refractory Materials’, p- 10-12.
23. “Lea’s Chemistry of Cement and Concrete” by Peter Hewlett, ISBN:0750662565, Publisher Elsevier Science & technology Books, page no-726.
24. A. Nishikawa ” monolithic refractories technology” Pilbrico japan Co. Ltd, Tokyo, Japan ,1984.
25. “ Material design for new insulating lining concepts” by Dale Zacherl*, Dagmar Schmidtmeier, Rainer Kockegey-Loren, Andreas Buhr, Marion Schnabel Almatiss GmbH, Frankfurt/Main, Germany Jerry Dutton Stourbridge, United Kingdom.
26. US Patent 392214, (1976).
27. US Patent 6287999B1,(2001).
28. EP 0686611 B1,(1998).