A thesis submitted in the partial fulfillment of the requirements for the degree

of Bachelor of Technology

By

Akash Satapathy 107CR016



A thesis submitted in the partial fulfillment of the requirements for the degree

of

Bachelor of Technology

By

Akash Satapathy 107CR016

Supervisor: Dr. Ritwik Sarkar





## NATIONAL INSTITUTE OF TECHNOLOGY ROURKELA

### **CERTIFICATE**

This is to certify that the thesis entitled, "*Effect of different binders on High Alumina Self-Flow Castable*" submitted by <u>*Mr. Akash Satapathy* (107CR016)</u> in partial fulfilments for the requirements for the award of <u>*Bachelor of Technology*</u> degree in <u>*Ceramic Engineering*</u> 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 matter embodied in this thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

Date:

Dr. Ritwik Sarkar Associate Professor

Department of Ceramic Engineering

National Institute of Technology

Rourkela – 769008

### **ACKNOWLEDGEMENT**

With deep respect, I avail this opportunity to express my gratitude to Dr. Ritwik Sarkar, Associate Professor, National Institute of Technology, Rourkela for his inspiration and guidance and valuable suggestion throughout this research work. His vast knowledge in the field of Science and Technology helped to enlighten me. It would have been impossible on my part to come out with this project report without him.

I would like to express my gratitude to Dr. Japes Bera, HOD, Ceramic Engineering and all other faculties for constant support and valuable suggestions. I would also take this opportunity to express my gratitude to the non-teaching staff for their help and kind support.

I would also like to thank IFGL Refractories for the help and support in carrying out experiments and providing Raw Materials. I am also thankful to the Research Scholar in the Department of Ceramic Engineering for helping out in labs and analysis.

And lastly I am thankful to my parents and friends for their constant support.

Akash Satapathy

107CR016

### **ABSTRACT**

Effect of binder and its amount on the high alumina self flow castable has been studied. Self flow castable has been prepared fixing the distribution coefficient q at 0.21, in the continuous particle size distribution model as proposed by Dinger-Funk. High alumina cement and silica sol were used at 4wt% and 6 wt% as binder and water is used till self flowing consistency was achieved in the castable mix. The Castable cubes were prepared as per conventional processing and heat treatment was done at 950°C and 1550°C. All the dried and heat treated samples were characterized for bulk density, volume shrinkage and cold crushing strength. The matrix phase of the 6% binder containing samples were tested for phase analysis using X-ray Powder Diffraction (XRD) technique, which showed mullite phase in sol containing castable and grossite in cement containing one.

# LIST OF TABLE

Sl.No.	Table	Page	
1	List of Alumina Aggregate used in various fraction	11	
2	List of the chemical analysis of the Raw Material	12	
3	CPFT for various particle Size	13	
4	Percentage aggregate for each fraction	13	
5	Batch Composition for Cement Based Castable	14	
6	Water Consumption during Casting	20	

## **LIST OF FIGURE**

Sl.No.	Figure	<b>Page</b> 18	
1	Plot of CPFT v/s Particle Size in Log-log Scale		
2	XRD analysis of Polychem (HAC Binder)	19	
3	Peak Match List of Polychem	19	
4	XRD of Sol based castable fired at 1550°C	21	
5	Peak Match list of Sol based Castable fired at 1550°C	21	
6	XRD of Cement based castable fired at 1550°C	22	
7	Peak Match list of Cement based Castable fired at 1550°C	22	
8	Bulk Density v/s temperature plot	23	
9	Volume v/s temperature plot	24	
10.	Cold Crushing Strength v/s temperature plot	26	

## **LIST OF ABBREVIATION**

Sl.No.	Abbreviation	Full Form	
1	HAC	High Alumina Cement	
2	PSD	Particle Size Distribution	
3	XRD	X-Ray Diffraction	
4	BFA	Brown Fused Alumina	
5	WTA	White Tabular Alumina	
6	WFA	White Fused Alumina	
7	CPFT	Cumulative Percent Finer Than	
8	BD	Bulk Density	
9	CCS	Cold Crushing Strength	
10	CA	CaO.Al <sub>2</sub> O <sub>3</sub>	
11	CA <sub>2</sub>	CaO.2Al <sub>2</sub> O <sub>3</sub>	
12	CAH <sub>10</sub>	CaO.Al <sub>2</sub> O <sub>3</sub> .10 H <sub>2</sub> O	
13	C <sub>2</sub> AH <sub>8</sub>	2 CaO.Al <sub>2</sub> O <sub>3</sub> .8H <sub>2</sub> O	
14	C <sub>3</sub> AH <sub>6</sub>	3 CaO.Al <sub>2</sub> O <sub>3</sub> .6 H <sub>2</sub> O	
15	C <sub>12</sub> A <sub>7</sub>	12 CaO.7Al <sub>2</sub> O <sub>3</sub>	

# **CONTENTS**

CERTIFICATE		. I
ACKNOWLEDGE	EMENT	. 11
ABSTRACT		Ш
LIST OF TABLES	AND FIGURES	. IV
LIST OF ABBREV	/IATIONS	. v
CHAPTER 1: INT	RODUCTION	. 1
	1.1 GENERAL INTRODUCTION	. 2
CHAPTER 2: LIE	TRATURE REVIEW	. 4
	2.1 CASTABLE AND PARTICLE SIZE DISTRIBUTION	5
	2.2 SELF FLOW CASTABLE	6
	2.3 SILICA SOL BASED CASTABLE	7
	2.4 CEMENT BASED CASTABLE	8
CHAPTER 3: EXF	PERIMENTAL PROCEDURE AND CALCULATION	10
	3.1 BATCH FORMULATION	11
	3.2 SAMPLE PREPARATION	15
	3.3 CHARACTERIZATION	16
CHAPTER 4: RES	SULTS AND DISCUSSION	17
	4.1 RAW MATERIAL CHARACTERIZATION	18
	4.2 SAMPLE CHARACTERIZATION	20
	4.3 PROPERTIES	23
CHAPTER 5: CO	NCLUSION	27
CHAPTER 6: REF	ERENCE	29

# CHAPTER

# INTRODUCTION

## 1.1 General Introduction

The ever-increasing demand and application of unshaped refractories to replace conventionally shaped refractories due to many major advantages (1) have inspired scientists and manufacturers to continuously investigate these materials in depth and to improve their quality and performance. Since the early 1960s, the availability of purer calcium aluminate cements (CACs) with higher alumina content has made the unshaped refractory system purer and allowed them to be applicable for various high-temperature applications (2). Among the various unshaped refractories, castables lead in all the areas of research, development, manufacturing and application. Physical, mechanical, chemical and thermo-mechanical characteristics of various castable systems as well as their processing and bonding mechanisms are the focus of such investigations.

Refractory castables are combinations of refractory aggregates, matrix components, bonding agents, and admixtures. The proportions of each component used vary in each castable composition to achieve the desired physical and chemical properties and characteristics for the intended castable application. Among different bonding agents calcium aluminate cement is the commonly used hydraulic binder in refractory castables compositions, but presence of CaO in the cement results low melting phases in Al<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> refractory system [1, 2]. Other important concerns for cement containing castables are the curing and dewatering steps, which must be carefully conducted in order to reduce explosive spalling [1]. A significant increase in the life and performance of alumina based castables has been made possible by decreasing the cement-content, thus avoiding / reducing the formation of liquid phase at application temperatures, which in turn improves the corrosion resistance and creep strength. Following this scientific backup, low cement castable (LCC), ultra low cement castable (ULCC), no cement castable (NCC) and self-flow castables (SFC) have already become popular in the refractory industries [3-5].

Further developmental work resulting in a new bonding system with superfine materials prepared through the sol-gel route, appeared in the industry, which opened a new horizon for refractory technologists. When combined with other solid particles, sol can be linked together in branched chains, in a process known as gelation (6, 7), which can be induced by water removal. During the drying step,

The hydroxyl groups (Si-OH) on the surface of the particles generate siloxane bonds (Si-O-Si), which results in a three-dimensional network (7). So, in a refractory system formation of a three dimensional network gel structure from a sol that surrounds the refractory aggregates results strength in the system on drying. Sol reduces / removes cement in the castable composition and on subsequent heating develops strength via ceramic bonding by low temperature sintering resulting in superior properties [8-10]. Silica and alumina sols have been tried for this purpose to a great extent and successful commercialization has also been achieved mainly by using silica sol.

# CHAPTER

# LIETRATURE REVIEW

# 2.1 Castable And Particle Size Distribution

Refractory is anything which saves energy as they withstand high temperature and high load even under extreme condition. ASTM C71 defines refractories as "non-metallic materials having those chemical and physical properties that made them applicable for structures or as components of systems that are exposed to environments above 1000°F".refractory can be shaped or unshaped.

Unshaped refractories have no predefined shape or size and are supplied to user industries as a mixture of different aggregates, binders and additives. They can take the shape of a single(mono) structure (lithus) without any joints and are also called monolithics. Among different unshaped refractories, materials that are cast/poured, with/without vibration are castables.

EN 1402 part 1 defines unshaped refractory materials as "mixture of aggregates and bonds, prepared ready for use after the addition of one or more liquids. They may contain metal, organic or ceramic fibre material." Application of a monolithic is entire based on its chemical composition and particle size distribution.

Particle packing is of two types: discrete packing and continuous packing. For monolithic refractory, packing plays a significant role. Unlike shaped refractory where packing determines the filled density only for unshaped refractory packing affects both the density and flowability. Hence instead of discrete packing (as useful for shape products) continuous packing is used for monolithics.

Grading is represented in log- log scale of cumulative percent finer than (CPFT) with particle size. This is the original equation of Andreassen

 $CPFT = (d/D)^q x 100$ 

And Andreassen was modified by Dinger and Funk.

The original equation doesn't indicate any definite minimum size of particle and assumes particles to be up to infinitesimal small, which is practically impossible. Dinger and Funk introduced a new parameter that is

the size of the minimum particle and makes the model practically feasible and applicable. The modified model is -

CPFT=  $[(d^{q}-d_{m}^{q})/(D^{q}-d_{m}^{q})].100$ 

Where

d= Particle Size of a fraction

D= maximum Particle Size

q= quotient

d<sub>m</sub>= minimum particle size

The target is to reduce the water requirement to reduce porosity and increase density. The need for packing is that it decides the castable is vibratable/ self flow mass and optimum packing requires less water for placement. The distribution factor dictates the Particle Size Distribution and the flow behavior of the castable.

## 2.2 Self Flowing Castable

Self flow is achieved if the exponent is less than 0.25. Flow is improved by increasing the fine fraction. Self flow is achieved by adding more fines and super fines to the composition. Flow is the effect of particles being able to move relative to each other separated by liquid. Adding fine and superfine improves the packing hence reducing the water consumption which gives low porosity.

A successful use of particle size distribution analysis is dependent on a deflocculated system. Deflocculant is added to disperse the fines in the composition. Fine and superfine oxide powder increase the packing of the castable by filling up the void spaces and to form a viscous suspension which keeps the coarse particles or aggregates sufficiently apart and the castable flows under its own weight due to the effect of the gravity.

Also fine and superfine particles tend to be spherical, so they also enhance the flow property of the castable.

In the flow process an important parameter is the yield stress ( $E_{yield}$ ). Yield stress indicates the minimum stress required to cause the deformation in the flow process of the castable. Flow takes place when the yield stress is exceeded.

Castable potential energy  $E_{pot}$  determines the flow process. If the castable potential energy exceeds the yield stress then flow process occurs and it stops as soon as  $E_{pot} = E_{yield}$ . In actuality, castable keeps deforming even after the equilibrium is achieved between the castable potential energy and yield stress. This is due to transformation of some castable potential energy to the kinetic energy during the flow process. Castable which develop a significant velocity and shear rate during the process exhibit a high kinetic energy and inertia during  $E_{pot} = E_{yield}$ , which results in deformation superior to as expected. The shear rate influences the castable viscosity and is an influential rheological parameter. For non-Newtonian castable, part of potential energy is absorbed by the system rather than converting to kinetic energy.

## 2.3 Silica Sol based Castable

Colloidal silica is a water stable suspension when mixed with castable enhances the drying speed and sinterability of the product. Colloidal silica contains up to 50 wt % of nanometric spherical amorphous silica particle. Colloidal silica is self dispersive in nature due to spherical size and nano-metric size. Colloidal silica can be linked together in branched chains, in a process called gelation which can be induced by water removal. During drying step the hydroxyl groups (Si-OH) on the surface of the particle generate siloxane bonds (Si-O-Si) which results in a 3-D network. The resulting structure is highly permeable and allows the composition to be easily dried, reducing cracks and explosive spalling. During sintering due to high surface area of silica induce the formation of mullite in alumina based system which improves the chemical resistance and hot strength properties.

Colloidal silica based castable require water removal so that gelling mechanism could take place. Water removal decreases the inter particle separation distance, hence increasing the particle collision and siloxane bond formation. For colloidal silica sometimes MgO sinter is used. To destabilize the colloidal silica suspension and then increase the probability for bond forming collisions, MgO sinter was added. MgO favors anionic reaction mechanisms on its surface by the withdrawal of hydrogen ions from Si-OH groups, which provides a higher siloxane bond formation increasing the colloidal silica gelation rate.

Colloidal silica based castable tale lesser torque and lesser mixing time due to greater viscosity of colloidal silica than water and they don't promote chemical reaction during mixing.

## 2.4 Cement Based Castable

High Alumina Cement (HAC) is used in Cement Based Refractory Castable. The major phases present in HAC are CA,  $CA_2 \& C_{12}A_7$ . As it is Hydraulic in nature, HAC gives high initial strength when mixed with appropriate amount of water. Hydrate phases are formed which gives the strength. Amount of CaO present in the HAC determines the water consumption by the HAC. With the increase in temperature the strength gradually decreases as the hydrated phases dehydrate.  $C_{12}A_7$  is the most reactive in HAC and causes flash setting effect.CA is less reactive than  $C_{12}A_7$  which in turn is more reactive than  $CA_2$ . Both chemical and physical changes take place when water is added to the cement. Hydration converts the anhydrous powder into various hydrate phases. During firing these hydrate phases break down leaving very reactive products, which re-crystallize to anhydrous calcium aluminates.

CA is the major phase in most HAC. When water is mixed with CA a suspension is formed and solid begins to dissolve. Then hydrates of Calcium Aluminates are formed during this process. These hydrates

nucleate and grow, allowing more and more anhydrous CA to dissolve into the solution. This throughsolution mechanism leads to the formation of a strong microstructure of interlocking hydrate crystals.

At relatively lower temperature (below 20°C), the hexagonal hydrate,  $CAH_{10}$  is formed but at higher temperature (above 30°C), this rapidly converts to the cubic phase  $C_3AH_6$ . At intermediate temperature another hexagonal hydrate,  $C_2AH_8$  is also formed and being meta-stable it is converted to  $C_3AH_6$ . Alumina gel and crystalline AH<sub>3</sub> are produced during this hydration mechanism. Substantial amount of CAH<sub>10</sub> and  $C_2AH_8$  is formed initially which later converts to  $C_3AH_6$  and AH<sub>3</sub>.

Hydration at around 20°C favors the formation of  $CAH_{10}$ ,  $C_2AH_8$ , gibbsite and alumina gel. At higher temperature  $CAH_{10}$  gives away to  $C_3AH_6$  and gibbsite. Pastes hydrated above 40°C were found to contain only  $C_3AH_6$  and  $AH_3$ .

# CHAPTER 3

# EXPERIMENTAL PROCEDURE

# 3.1 Batch Formulation

#### **Sampling of Raw material**

Step 1: Procurement of Raw Material

Aggregate: - BFA, WTA, WFA, Reactive Alumina, Tabular Alumina

Binder: - HAC binder (Polychem 75), Silica Sol (30% sol)

Additives:- Fume Silica, Sodium Hexametaphospate, Citric Acid

Step 2: Sieving the raw material to obtain various fractions.

Fraction (in mm)	Raw Material
- 5.6 to + 2.8	BFA
-2.8 to $+2.0$	Tabular Alumina
- 2.0 to + 1.0	Tabular Alumina
- 1.0 to + 0.5	WFA
-0.5 to $+0.25$	WTA
-0.25 to $+0.15$	WTA
-0.15 to $+0.075$	WTA
-0.075 to $+0.001$	Reactive Alumina

Table 1: List of alumina aggregate used in various fractions

#### **Characterization of Raw Materials**

Step 1: XRD of the HAC binder was done to identify the phases present. This was done by X- Ray Powder Diffraction Technique.

Step 2: Characteristics of raw material as provided by supplier

Oxide	Polychem	BFA	WTA	WFA
$Al_2O_3$	71.48%	95.66%	99.25%	98.92%
SiO <sub>2</sub>	0.4 %	0.4%	< 0.12%	-
CaO	24.6%	0.1%	< 0.1%	-
MgO	0.2%	-	-	-
TiO <sub>2</sub>	-	0.64%	-	Trace
$Fe_2O_3$	-	0.4%	< 0.03%	0.06%
Alkali(Na <sub>2</sub> O and K <sub>2</sub> O)	0.1%	-	< 0.4%	0.08%

Table 2: List of the chemical analysis of the raw material.

#### **Batch Calculation**

Each batch of 2.5kg was made. Batch was calculated for each composition (cement binder 4% & 6%) and silica sol 4 % & 6%) using the Dinger & Funk Equation

CPFT=  $[(d^q - d_m^q)/(D^q - d_m^q)] \ge 100$ 

Where,

D= Maximum Particle Size (5.6 mm) dm= Minimum Particle Size (1µm)

d = Particle Size

q= quotient (0.21)

12 | Page

Fraction (in mm)	Percentage ( in %)
5.6	100 %
2.8	83.81 %
2.0	76.76 %
1.0	63.72 %
0.5	52.45 %
0.25	42.7%
0.15	36.37 %
0.075	28.8 %
0.001	0

#### Table 3: CPFT for various particle Size

Fraction (in mm)	Percentage
- 5.6 to + 2.8	16.19 %
-2.8 to $+2.0$	7.05 %
-2.0 to $+1.0$	13.04 %
- 1.0 to + 0.5	11.27 %
-0.5 to $+0.25$	9.75 %
-0.25 to $+0.15$	6.33 %
-0.15 to $+0.075$	7.57 %
-0.075 to $+0.001$	28.8 %

After calculating the CPFT, percentage aggregate for each fraction was calculated.

Table 4: Percentage aggregate for each fraction

Batch composition for cement based castable

Fraction (in mm)	Percentage	Additives / Binder	Percentage
- 5.6 to + 2.8	16.19 %	Sodium	
-2.8 to $+2.0$	7.05 %	Hexametaphosphate	0.1 %
-2.0 to $+1.0$	13.04 %		
-1.0 to $+0.5$	11.27 %	Citic Acid	0.05 %
-0.5 to $+0.25$	9.75 %		
-0.25 to $+0.15$	6.33 %	Fume Silica	4 %
-0.15 to $+0.075$	7.57 %		
-0.075 to $+0.001$	24.8 % (for 4% Cement		4 % (for 4% Cement
	based Castable)		based Castable)
	22.8 % (for 6% Cement	Cement Binder	6 % (for 6% Cement
	based Castable)		based Castable)

Table 5: batch composition for cement based castable

For silica sol based castable, 1% fume silica was used. Silica sol was added during mixing.

## 3.2 Preparation of samples

#### **Mixing**

During mixing the coarse aggregate was added first then gradually fine fractions were added and at the end binder was added. The mix was thoroughly mixed to homogenize the mixture. Silica sol was added during this step at the end for sol based castable.

#### **Casting**

The required consistency of water was added to have self flow in the batch prepared. Then the castable mix was poured into the lubricated moulds. The excess mix was scrapped off and smoothened by a trowel. The moulds were kept for 24 hours in humid condition for 24 hrs to avoid the immediate drying of the surface.

#### **Drying**

After de-molding samples were kept for 24 hrs for air drying and then the samples were dried in an oven at 110°C for 24 hours. Weight and dimensions were measured after oven-drying of the samples. The dimensions were measured using Vernier caliper (least count 0.02 mm).

#### <u>Firing</u>

Two of each sample were taken and fired at 950°C and 1550°C respectively for 2 hours. After firing the dimensions of the samples were measured.

# 3.3 Characterization

#### **Bulk Density Measurement**

The physical bulk density of the sample was measured using the linear dimensions of the sample.

The dry weight of sample was measured.

Bulk Density = Weight /(Length \* Breadth \* Height)

#### **Volume Shrinkage Measurement**

The volume shrinkage of sample before and after firing was measured.

Volume shrinkage =  $(V_B - V_A)/V_A * 100$ 

V<sub>B</sub>= Volume of sample after firing

 $V_A$ = Volume of sample before firing

#### **Crushing Strength measurement**

Crushing strength of the bricks was measured in a Compressive Tester.. The Crushing Strength was

be determined by

Cold Crushing Strength = (Load / Area)

Load in Kg and Area in cm<sup>2</sup>

#### **Phase Identification**

Phase identification was performed in a X-Ray Diffractometer (PW-1830, Philips, Netherlands made) using Cu K-α radiation.

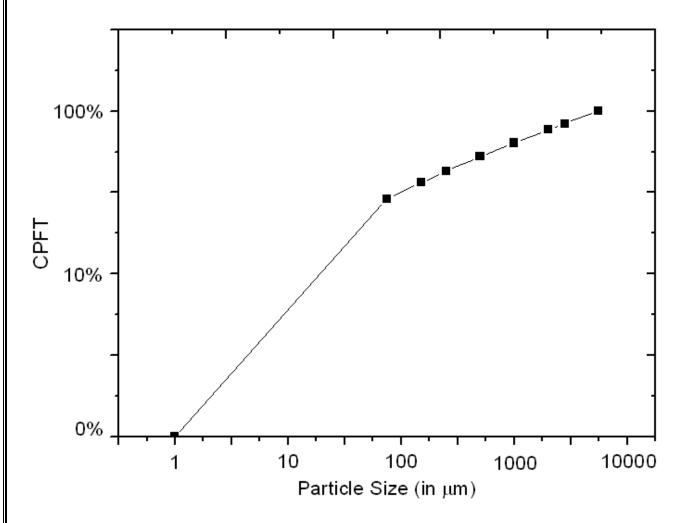
# CHAPTER

# RESULTS & DISCUSSIONS

# 4.1 Raw Material Characterization

#### Particle Size Distribution

The Particle size distribution formulated gives the plot of CPFT v/s Particle size in a log-log scale as shown below:-



*Figure 1: Plot of CPFT v/s PSD in a log – log scale* 

Figure 1 shows the plot of CPFT with the PSD with the value of q=0.21. The lowest particle size being 1µm shows 0% CPFT. There is an abrupt change in the slope of the plot due to high fine content (28.8 %).

#### XRD of High Alumina Cement

HAC was used as binder for cement based castable. XRD of HAC gave the idea about the phases present in the binder. XRD was done in Philips X-Ray Diffractometer (PW 1730, Holland)

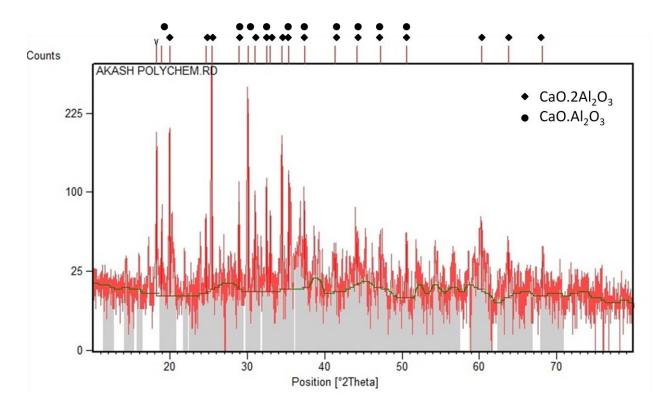
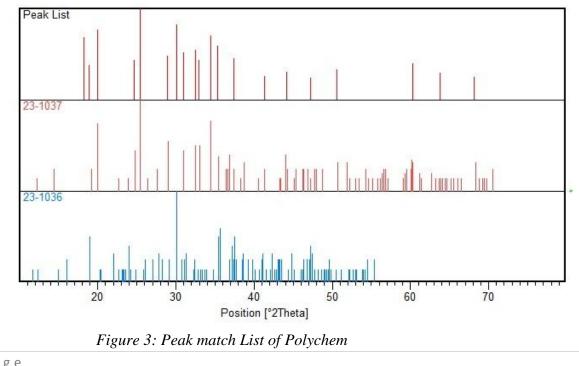


Figure 2: XRD analysis of Polychem (High Alumina Cement Binder)



**19 |** Page

23-1037 -  $CaO.2Al_2O_3(CA_2)$ 

23-1036 - CaO.Al<sub>2</sub>O<sub>3</sub> (CA)

From the XRD analysis of the high alumina cement binder as observed in figure 2 and 3, it was found that  $CaO.2Al_2O_3$  (CA<sub>2</sub>) was the major phase and the other phase was  $CaO.Al_2O_3$  (CA)

# 4.2 Sample Characterization

#### Water Required for Casting

Table shows the water required for casting of cement bonded castable and sol bonded castable.

Water Required for Casting ( in mL)		
Cement based castable	Sol based castable	
190 mL	75 mL	
200 mL	50 mL	
	Cement based castable 190 mL	

#### Table 6: Water Consumption in Casting

Table 6 shows cement based castable shows higher water consumption than sol based castable as

sol was used for casting of sol based castable. Water is added to get the desired flow property.

The water required for casting of cement based castable increases with increase in high alumina

cement content due to increase of CaO content in the composition.

For sol based castable the water requirement decreases as the percentage of sol increases (100 ml to 150 ml) as low amount of water will be required to get the desired flow property.

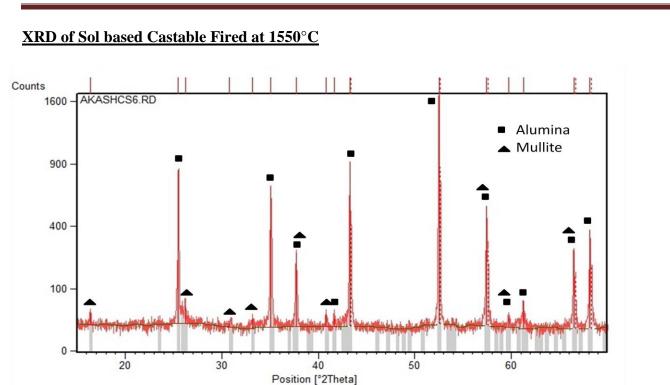


Figure 4: XRD of Sol based Castable fired at 1550°C

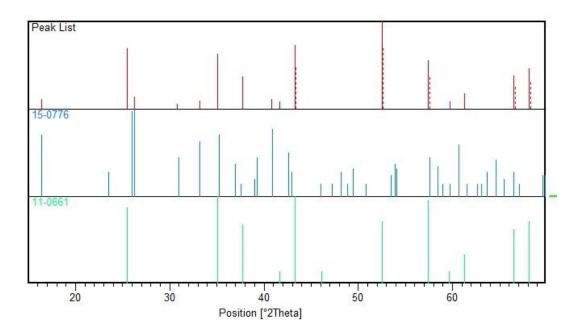


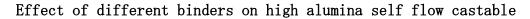
Figure 5: Peak Match list of Sol based Castable fired at 1550°C

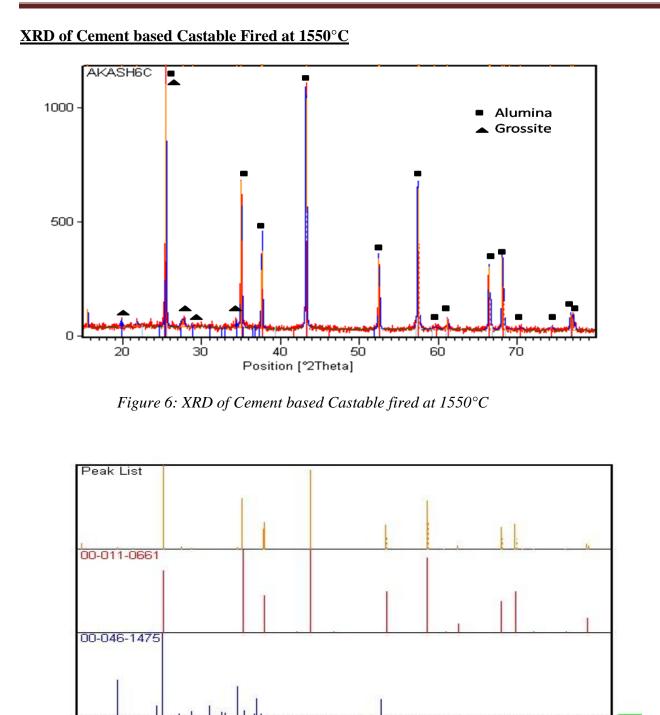
11-0661- Al<sub>2</sub>O<sub>3</sub>

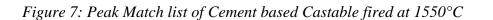
 $15-0776 - 3Al_2O_3.2SiO_2$ 

In the figure 4 and 5, the XRD analysis shows that the major phase present in sol based castable is alumina and the other phase present is mullite. Mullite may be formed due to the reaction between fine alumina particles and  $SiO_2$  coming from sol in the matrix phase.

**21 |** P a g e







50

60

70

00-011-0661 - Alumina (Al<sub>2</sub>O<sub>3)</sub>

00-046-1475- Grossite (CaO.2Al<sub>2</sub>O<sub>3)</sub>

In the figure 6 and 7, the XRD analysis shows that the major phase present in cement based castable is

40 5 Position [°2Theta]

alumina and the other phase present is grossite.

20

30

**22 |** P a g e

# 4.3 Sample Characterization

#### **Bulk Density**

Figure 8 shows the dependence of bulk density with temperature for different binders at different weight percentage. Bulk density of various compositions is plotted at 110°C, 950°C & 1550°C

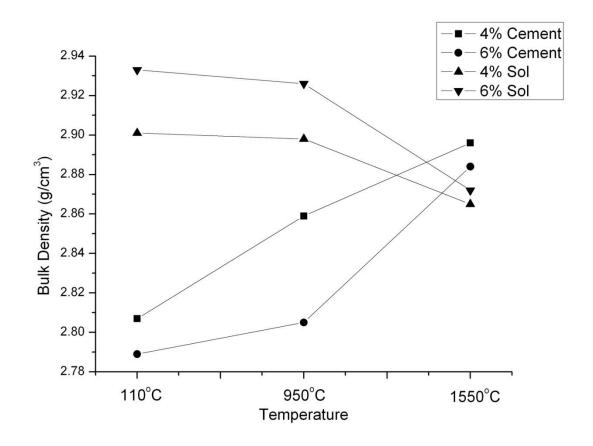


Figure 8: Bulk Density with Temperature Plot

The bulk density gradually increases for cement based castable due to gradual removal of pores and densification, and that of sol bonded castable decreases due to gradual expansion because of mullite phase formation.

At 110°C, 4% cement based castable shows more bulk density than 6% cement based castable due to high porosity in the oven dried samples as required water for casting 6% cement based castable is higher than 4% cement bonded castable.

4% sol based castable shows lower bulk density than 6% sol based castable due to high porosity in the oven dried samples as required water for casting 6% sol based castable is lower than 4% sol bonded castable.

Sol bonded castable shows higher bulk density than cement bonded castable due to less water requirement for casting.

#### **Volume Shrinkage**

Figure 9 shows the variation of the volumetric shrinkage with temperature for different binders at different percentage. Volume shrinkage of various compositions is plotted at 110°C, 950°C & 1550°C

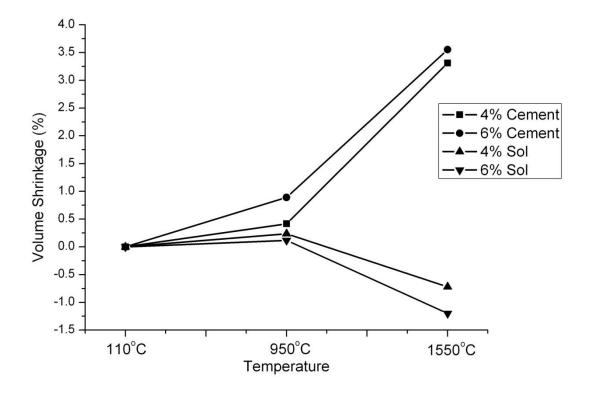


Figure 9: Volume Shrinkage with Temperature Plot

The cement based castable shows shrinkage at both 950°C and 1550 °C whereas sol based castable shows shrinkage at 950°C and expansion at 1550°C.

6% cement based castable shows higher shrinkage than 4% cement based castable at both 950°C and 1550°C.

At 1550°C as more liquid is formed in case of 6% cement based castable than 4% cement based castable, higher densification and better pore removal occurs. More liquid is formed due to higher percentage of CaO in the composition.

Both 4% & 6% sol based castable shows shrinkage at 950°C and then it gradually shows expansion may be due to mullite phase formation in the composition

At 1550°C as high amount of mullite is formed in case of 6% sol based castable than 4% sol based castable, it shows higher expansion. High amount of mullite is formed due to higher percentage of  $SiO_2$  in the composition.

#### **Cold Crushing Strength**

Figure 10 shows the variation of the volumetric shrinkage with temperature for different binders at different weight percentage. Volume shrinkage of various compositions is plotted at 110°C, 950°C & 1550°C

The value of cold crushing strength gradually increases for all castable composition with increase in the temperature. This is due to pore removal and densification.

At 110°C, the cement based castable shows higher strength than sol based castable due to presence of hydrated bonding phases which gives strength to the castable.

6% cement based castable gives higher strength than 4% cement based castable due to higher amount of the hydraulic binder.

Sol based castable shows lower strength than cement based castable as no bonding phases are present.

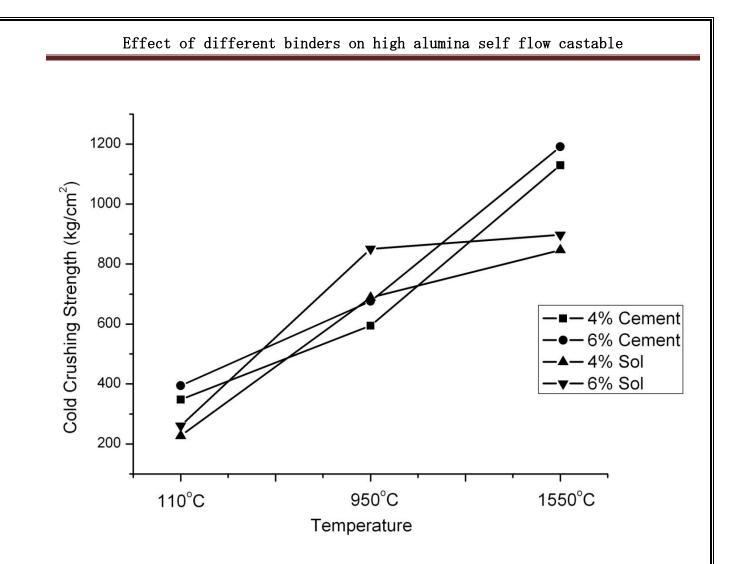


Figure 10: Cold Crushing Strength with Temperature Plot

Cement based castable shows low strength at  $110^{\circ}$ C than at  $950^{\circ}$ C unlike theoretical estimation, as because the major phase present in the HAC is CA<sub>2</sub> which shows lower hydraulic behavior hence gives low strength at  $110^{\circ}$ C.

At 1550°C, the cement based castable shows higher strength than sol based castable due to significant densification which gives higher strength.

# CHAPTER S

# CONCLUSION

# 5. Conclusion

Effect of binder and its amount on the high alumina self flow castable, with a distribution coefficient q at 0.21 as per the continuous particle size distribution model proposed by Dinger-Funk, has been studied.

Initial strength of the sol bonded castable was found to be lower than that of the cement bonding one due to the coagulation bond mechanism of sol as compared to that of the chemical hydrate bond formation in cement containing ones. However the cement containing ones showed relatively lower initial strength values may be due to the presence of higher amount of less hydratable grossite (CA<sub>2</sub>) phase and lesser extent of CA phase in the high alumina cement.

Mullite phase is found in the sol bonded castable due to the reaction between silica particles from sol and fine alumina present in the matrix at high temperatures and the sol containing castables showed expansion or lower shrinkage values. Also formation of mullite has resulted in a reduction in density values at high temperatures.

Only grossite is found other than corundum phase in XRD of cement bonded castable may be due to un-reacted grossite phase from high alumina cement or due to formation of grossite phase by reaction between of calcium aluminate and fine alumina present in the matrix at high temperatures.

# CHAPTER 6

# REFERENCE

1. . W. E. Lee, W. Vieira, S. Zhang, K. G. Ahari, H. Sarpoolaky and C. Parr, "Castable Refractory Concrete," Intern. Mat. Rev., 46 [3] 145-167 (2001).

2. S. Banerjee, "Recent Developments in Monolithic Refractories," *Am. Ceram. Soc. Bull.*, **77** [10] 59-63 (1998).

3. J.S. Masaryk, Development and use of low cement self flow castables, in: Proc. UNITECR, Sao Paulo, Brazil, pp. 527–538 (1993).

4. K. Bhattachariya, P. Chintaiah, D.P. Chakraborty and M.S. Mukhopadhyay, Ultra low cement castables– a new generation of trough bodies for increased cast house life. *Interceram.* **47** [4] 249–251 (1998).

5. A.R. Studart, R.G. Pileggi, W. Jhong and V.C. Pandolfelli, Processing of zero cement self-flow high

alumina refractory castables by matrix rheological control. Am. Ceram. Soc. Bull. 77 [12] 60-66 (1998).

6.S. Banerjee, "Monolithic Refractories–A Comprehensive Handbook," Singapore: World Scientific/The American Ceramic Society, 311p (1998).

7. R. K. Iler, "The Chemistry of Silica: Solubility, Polymerization, Colloid and Surface Properties, and Biochemistry," New York, Wiley, 866p (1979).

8. S. K. Das, R. Sarkar, P. K. Mondal and S. Mukherjee, "No cement high alumina self flow castable", *American Ceramic Society Bulletin*, 82 [2] 55 – 59 (2003).

9. T. K. Mukhopadhyay, Ritwik Sarkar, S. K. Das, S. Ghosh and S. Ghatak, "Effect of synthetic mullite aggregate prepared by hydroxyl-hydro gel method on sol bonded clay based mullite castable", *American Ceramic Society Bulletin* (www.ceramicbulletin.org) *84 [11] p 9101-9106 (2005)*.

10. R. Sarkar, S. Mukherjee and A. Ghosh, "Gel bonded  $Al_2O_3 - SiC - C$  based blast furnace trough castable", *American Ceramic Society Bulletin* (www.ceramicbulletin.org) 85 [5] p 9101-9105 (2006).

11. H. S. Fang, C. H. Cha and S. Y. Yang, "Development of self flow castable" Proceedings of UNITECR59, vol, 1, pp 264-71, Kyoto, Japan (1995).

12.Bjorn Myhre and Aase M. Hundere, Elkem Refractories, "The use of particle Size Distribution in development of Refractory Castable".

13. B.N.Samaddar, "Principles of powder processing- packing of powders", IRMA journal Vol- XXXIV no.4,2001

14.D.R.Dinger and J.E.Funk, USA, "Particle Packing- III – Discrete versus Continuous Particle Sizes"
15. D.R.Dinger and J.E.Funk, USA, "Particle Packing- III – Compute Modelling of Particle packing phenomena"

16. "The use of particle Size distribution in development of refractory castable", presented at the XXV

ALAFAR congress, San Carlas De Bailocke Argentina, Dec 1-4,1996, Elkem Refractory

17.A.K.Chatterjee, "Revisiting calcium aluminates and aluminous cements", IRMA Journal Vol.XXXVII, No.1,2004

18.A.V.Briebach, Morgan Refractories Ltd., Neston, Wirral, Cheshire, "A review of Refractroy Hydraullic Cements ".

19. P.Pilate, J. Tirlocq, F. Cambier, Belgian Ceramic Research Centre, Mons Belgium, "Refractory Castables : an overview" cfi/Ber.DKG 84(2007) No.6

20. Rafael Salomão, Marina Rojas Ismael, Victor Carlos Pandolfelli , federal University of São Carlos – Materials Engineering Department "Hydraulic Binders for Refractory Castables: mixing, curing and drying" cfi/Ber.DKG 84(2007) No.9

21.Z.Li, G.ye, P.R.China , Luoyang Institute of Refractories Research paper on Refractories 2(1990)22.Subrata Banerjee, Answer Technology Inc, Wheaton, Illinois, USA featured article" Versatility of Gel-Bond castable/pumpable Refractories."

23. (Mrs.)K.M.Parker and J.H.Sharp Dept. of Ceramics, Glasses and Ploymers, University of Shiffield Review Paper "Refractory Calcium Aluminate Cements".

24. M.R.Ismael, R.D.dos Anjos, R.Salomão and V.C. Pandolfelli " colloidal silica as a nanotructured binder for refractory castables" Refractories application and news, volume 11, number 4

25. A.R.Studart,W.Zhong,R.G.Pileggi and V.C. Pandolfelli "processing of zero-cement self-flow alumina castables" The American ceramic society bulletin.

26. "Free-flowing castables- a prerequisite for wet-gunning of refractory castables" presented at VII-th International Metallurgical Conference in Ustron , Poland, May 1997, Elkem Materials