

## STUDY ON DOLOMITE BRICKS WITH PPLC (POSITIVE PERMANENT LINEAR CHANGE)

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### Abstract

Dolomite brick is the most important stable refractory under the working conditions of Argon Oxygen De-carbonization (AOD). For AOD applications, the Dolomite bricks should have positive Permanent Linear Change (PLC)[1,2] on re-heating. A positive PLC dolomite brick has higher life and most suitable for joint less refractory lining with no cobble stoning defect. So the development of dolomite bricks having positive PLC during applications is a challenge for refractory manufacturing industry. In the present investigation, dolomite refractory bricks were prepared by varying different additive with an intention to get the positive PLC of the brick. It is well known that Iron Oxide is a good additive for dolomite brick sintering. It has also been reported that monoclinic zirconia (m-ZrO<sub>2</sub>) additive improves the BD, CCS and PLC of dolomite brick. In this background, first trial composition was containing a fixed amount of 1% m-ZrO<sub>2</sub> and different concentration and types of iron oxide. Mill scale containing Fe<sub>2</sub>O<sub>3</sub> was added in two different concentrations 0.5%, 1% and in another trial 0.25% of nano Fe<sub>2</sub>O<sub>3</sub> was added. Fe<sub>2</sub>O<sub>3</sub> containing bricks showed a good improvement in BD and CCS due to the better sintering of dolomite grains in presence of liquid formation during sintering by the presence of Fe<sub>2</sub>O<sub>3</sub>. The CCS value was in the range 750-800 kg/cm<sup>2</sup>. However, the PLC value was negative in the range of -0.2 to -0.35%. Depending on this result, new additive, [3,4]Chrome Green oxide was then used. It has been found that the PLC tends to be positive with increase in Cr<sub>2</sub>O<sub>3</sub> content. However, in presence of Cr<sub>2</sub>O<sub>3</sub> the m-ZrO<sub>2</sub> it was not performing well. Finally m-ZrO<sub>2</sub> was removed and a higher percentage of Cr<sub>2</sub>O<sub>3</sub> additive showed the positive PLC (+ 0.12%) of the dolomite brick. The brick showed a CCS value of 650 g/cm<sup>2</sup>. This is in the acceptable limit[5].

### Introduction

The method of operation has been significantly altered as a result of technological advancements in the production of iron and steel. All units experience an increase in furnace capacity, operating temperature, hot-metal temperature, and throughput. These extreme changes alongside the need of further developed rehearses for better assembling and application climate are requesting another age of recalcitrant material with further developed properties, execution and existence with eco- benevolence. In the metallurgical, glassmaking, and ceramic industries, refractories are used to line the interiors of furnaces, kilns, or other devices that process materials at high temperatures. They are formed into a variety of shapes for this purpose. Without refractory materials, many of the advancements in science and technology would not have been possible. It is almost impossible to dream of producing one

kilogram of any metal without refractory[6,7]. According to ASTM C71, refractory materials are "nonmetallic materials having those chemical and physical properties that make them applicable for structures or as components of systems that are exposed to environments above 1000 °F (538 °C)." In keeping with the shifting trends in steelmaking, particularly in ladle metallurgy, there has been an increase in demand for high-performing shaped refractory materials in recent years. The availability and performance of shaped refractories with superior high temperature mechanical strength, erosion, and corrosion resistance determine the higher campaign lives and variability of newer steelmaking operations[8].

### **Characteristics**

Dolomite bricks are less durable, more porous, and softer than magnesia bricks. They can be used at temperatures of up to 1650 degrees Celsius with or without a load.

A decent covering development is the main property of an essential block. In practice, if the kiln conditions are right, a coating will form[9]. The most significant distinction between the various kinds of brick utilized in this context is their capacity to retain the coating once it has formed. In reality, the refractory lining's clinker coating is not always present during the campaign. Thermal shock may be the cause of some or all coating loss. The brick underneath will experience severe thermal shock when the coating on the lining is destabilized because of the sudden temperature change [17].For an extensive stretch of time, standard dolomite was restricted to the area where stable covering existed over the obstinate coating, and closures were negligible.However, the development of zirconia- enriched grades has significantly



improved the thermal shock resistance of dolomite brick. Zirconia was chosen due to its relatively low dolomite-reactivity[11,12].

**Fig .1: Dolomite refractory**

### **Materials and Methods**

#### **Material used**

Mixture proportions for SCC differ from those of ordinary concrete, therein the previous has more powder content and fewer coarse aggregate. Also, SCC incorporates high range water reducers (HRWR, superplasticisers) in larger amounts and regularly a viscosity modifying agent (VMA) in small doses. The questions that dominate the choice of materials for SCC are:

1. Limits on the amount of marginally unsuitable aggregates, that is, those deviating from ideal shapes and sizes,[13]
2. Choice of HRWR,
3. Choice of VMA, and

#### 4. Relation, interaction and compatibility between cement, Cement

Cement may be a binder, a substance that sets and hardens independently, and binds other materials together. The most important use of cement is that the production of mortar and concrete for bonding of natural or artificial aggregates to make a robust artefact that's durable in the face of normal environmental effects. Concrete may be a combination of cement, aggregate and water. Cement may be a powder manufactured from limestone that's mixed with other aggregates, notably sands, gravels and stone, to supply mortars and concretes[14,15]. High-quality cements require raw materials of adequate purity and uniform composition. The overwhelming majority of cement utilized in the India is hydraulic cement, sometimes mentioned as Ordinary hydraulic cement or OPC; although there also are specialist cements, such as Sulphate-Resistant Cement (SRC) and High-Alumina Cement (HAC) which are often used for sub-surface works. Indian standards IS: 12269- [2013] outlines the specifications for OPC 53 grade cement[16].

#### Properties of coarse aggregates (Grit)

The test results for the properties of grit are presented in Table 3.5. The properties of grit indicated that it was suitable for use to produce the concretes. The bulk density, fineness modulus and relative density were within permissible limits specified by the Indian standards IS: 2386- [1963]. The bulk density of grit was 1764 kg/m<sup>3</sup>, which is lesser than that of sand. The bulk density of aggregates generally varies from 1200 to 1780 kg/m<sup>3</sup> [Kosmatka et al. 2002]. It includes the pores and voids existing in aggregates. The bulk density of aggregates is usually less than that of sand thanks to increased void content. However it's favourable that the difference in densities isn't very much like large difference between the relative densities of fine and coarse [17,18] aggregates results in increased segregation in concrete. The absorption of grit obtained was 1.58%. The absorption of fine aggregate generally varies within the range of 0.5 to 4.5% [Neville 2008]. Thus, the absorption of grit was in the lower range, which is good for concretes. A higher absorption value is indicative of greater pores in aggregates which may affect the strength and sturdiness of concretes[19,20].

**Table 1 : Properties of Coarse Aggregate-Grit**

Property	Value of Grit
Source	Hirri Mines (Bilaspur, C.G.)
Colour	Greyish Black
Specific Gravity	2.75
Fineness Modulus	5.76
Bulk Density	1764 kg/m <sup>3</sup>
Absorption	1.48%
Surface Moisture	0.1%

**MIX DESIGN FOR M20 GRADE CONCRETE****Design Stipulations:**

1. Grade of concrete (designation) = M20
2. Type of cement = OPC 53 Grade
3. Minimum nominal size of aggregate = 20 mm
4. Workability = 75-100 mm (slump)
5. Exposure condition = mild
6. Method of concrete placing = normal
7. Degree of supervision = good
8. Specific gravity of cement = 3.15
9. Specific gravity of coarse aggregate = 2.70
10. Specific gravity of fine = 2.56
11. Sieve analysis = zone 2 (IS 383-1970)



**Fig 1: Compression Testing Machine**

**Equipment:**

- Steel, iron cast, or other non-absorbent material moulds with size of (150mmX150mmX 750mm)
- Tamping rods: ASTM specify large rode (16mm diameter and 600mm long) and small rode (10mm diameter and 300mm long)
- Testing machine capable of applying loads at a uniform rate without interruption of shocks
- Scoop
- Trowel
- Balance with accuracy of 1g
- Power driven concrete mixer
- Table vibration in the case of using vibration to compact concrete in moulds

**Experimental Work**

PLC specimen samples were made with following raw materials[21,22], composition and experimental conditions as shown in the Table 3.1 to Table 3.8.

Raw materials used for this study: Calcined Dolomite, Fused magnesia large crystal (FMLC), Chrome Oxide green, Mill Scale, Zirconia, Nano Iron Oxide, Wax and Stearic Acid.

**Table 2: Experimental conditions**

<b>T1</b>		
Raw Materials	Granulometry (mm)	% Addition
Dolomite	(3-5)	10
	(1.6-3)	45
	(0.3-1.6)	10
	(0-0.3)	15
	Dust	5
FMLC 97	(0-1)	10
FMLC 97	Dust	5
Mill Scale	Dust	0.5
m-ZrO <sub>2</sub>	Dust	1
Wax	-	2.2
Stearic Acid	-	0.05

**Batch composition T1**, to T6 are mentioned on Tables T2 to T7 below:

**Table 3: Composition of T 1**

<b>T1</b>		
Raw Materials	Granulometry (mm)	% Addition
Dolomite	(3-5)	10
	(1.6-3)	45
	(0.3-1.6)	10
	(0-0.3)	15
	Dust	5
FMLC 97	(0-1)	10
FMLC 97	Dust	5
Mill Scale	Dust	0.5
m-ZrO <sub>2</sub>	Dust	1
Wax	-	2.2
Stearic Acid	-	0.05

**FMLC**

Fused Magnesia having large crystal size (500 - 1000µm), is an alkaline earth metal and the eighth-most-abundant element in the Earth's crust and ninth in the known universe as a whole. Magnesium is the fourth-most-common element in the Earth as a whole (behind iron, oxygen and silicon), making up 13% of the planet's mass and a large fraction of the planet's mantle. The free element (metal) is not found naturally on Earth, as it is highly reactive (though once produced, it is coated in a thin layer of oxide, which partly masks this reactivity). The free metal burns with a characteristic brilliant-white light, making it a useful ingredient in flares. The metal is now obtained mainly by electrolysis of magnesium salts obtained from brine.

**Volume stability, expansion and shrinkage at high temperature**

The contraction or expansion of the refractory's can take place during service. Such permanent

changes in dimensions may be due to:

- a) The changes in the allotropic forms which cause a change in specific gravity
- b) A chemical reaction which produces a new material of altered specific gravity.
- c) The formation of liquid phase.
- d) Sintering reactions.
- e) It may also happen on account of fluxing with dust and slag or by the action of alkalis on fireclay refractory's, to form alkali-alumina silicates, causing expansion and disruption. This is an example which is generally observed in blast furnaces.

## Results and Discussions

### Observation of Iron oxide

It has been reported that Iron Oxide ( $\text{Fe}_2\text{O}_3$ ) plays an important role in Dolomite refractory's. It is established that  $\text{Fe}_2\text{O}_3$  helps in liquid phase sintering of dolomite, which results in a good densification of the brick. For that reason, the CCS increases with  $\text{Fe}_2\text{O}_3$  addition due to the increase in BD, decrease in AP. This in turn improves the hydration resistance. As well as  $\text{Fe}_2\text{O}_3$  containing liquid, which forms during sintering solidify as a coating upon the Dolomite grains. This coating again improves the hydration resistance. In this background, the first trial composition for the present study was composed of  $\text{Fe}_2\text{O}_3$  containing additives. Our target was to get positive PLC of the brick. The references [20] also stated that the  $\text{Fe}_2\text{O}_3$  containing dolomite brick will not produce shrinkage during PLC firing because the brick is already highly dandified.

### Role of Zirconia

It has also been reported that monoclinic Zirconia ( $m\text{-ZrO}_2$ ) additives in Dolomite brick improves the properties like crack arresting [97] by forming  $\text{CaZrO}_3$  bonded materials. The thermal expansion of  $\text{CaZrO}_3$  compared to  $\text{MgO}$  can lead to the formation of gaps between  $\text{Ca}_2\text{SiO}_4$ ,  $\text{CaZrO}_3$  and  $\text{MgO}$  grains. These discontinuities can increase toughness and thermal shock resistance by stopping crack propagation. On this basis, our first trials composition was based on 1 % monoclinic  $\text{ZrO}_2$  and mill scale additives with 0.5 % and 1% variation. Mill scale mainly contains  $\text{Fe}_2\text{O}_3$ .

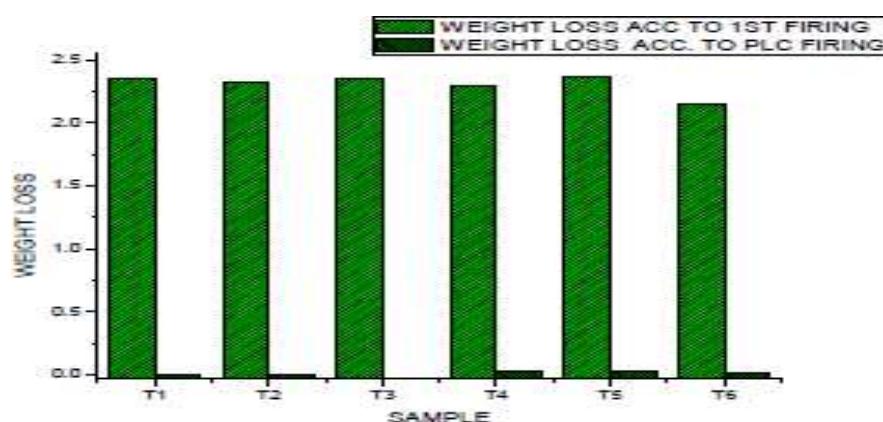
### Weight Loss

The weight loss during 1<sup>st</sup> firing and PLC firing. The weight loss during 1<sup>st</sup> firing is due to the evaporation of binder from the sample. During the PLC firing the dust raw material loses its grip from the outer surface and begins to come out in dust form, thus weight loss occurs.

**Table 4 :Weight loss of the specimen**

Sample No.	Weight loss acc. to 1 <sup>st</sup> firing	Weight loss acc. To PLC firing
T1	2.36	0.00
T2	2.33	0.00
T3	2.35	-0.03
T4	2.3	0.03
T5	2.37	0.03
T6	2.15	0.013

**Fig 2 Weight loss of different specimen**



### Conclusion

The major findings are:

- Able to achieve positive PLC (0.14%) by the addition of green chrome in the composition.
- CCS values of Trial Bricks T2, T3 containing iron oxide were excellent about 800 kg/cm<sup>2</sup>.
- Uniform distribution of nano-iron oxide accelerates the formation of CaZrO<sub>3</sub>.

Finally, according to the work done, it can be concluded that by using chrome oxide we can achieve positive PLC, and thus it was successful.

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