International Journal of Novel Research in Engineering and Applied Sciences (IJNREAS) 1(2)
April 2014©Avi-D Publishers 2014

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CHARACTERIZATION AND EVALUATION OF REFRACTORY PROPERTIES OF IGBETI DOLOMITE

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ABSTRACT

Any nation aspiring to be self reliance and become technologically dependent must be taken in sourcing and developing its local raw materials. For such a nation, substantial parts of her home-grown research endeavors should be directed towards her industrial and technological needs. This is the idea behind this work titled "characterization and evaluation of refractory properties of Igbeti Dolomite". Investigation reveal that Dolomite samples from Igbeti (Oyo State) is suitable for lining of furnace and kilos where the materials being melted requires basic environment and operating temperature is below 1778^{o}C. This is consequent upon the fact that it's chemical composition and refractory properties are within the acceptable standard range. The material was found to be basic as it contains high percentage of CaO and MgO. Refractoriness was found to be 1778^{O}C, bulk density 2.84g/cm^3, porosity of 19.28% and permeability of 88mm/s. The value of linear shrinkage was 0.2%, 23 cycles for thermal shock resistance and 650KN/cm^2 for cold crushing strength. It is concluded that the refractory produced of Dolomite from Igbeti deposit is suitable for lining of steel making furnace where basic environment is required and operating temperature is below 1778^{O}C.

Keywords: Refractory, Dolomite, Refractoriness, Basic, Calcitic, Properties.

1.0 INTRODUCTION

For any nation to have full economic freedom, her people, government, tertiary institution, research institutions and organized private sector must be interested and pursue vigorously local production of machines and parts with significant locally sources input.

To enhance national development, the foundry industry must be re-examined with a view of looking inwards for her materials needs. ***According to Adeyemo (2000),*** over 70% of our casting needs are still imported, over five decades after our political independence. This shows the low level of commitment to the development.

Two factors are accentuating the development of good refractories using the local raw materials. The first one is the growing number of metallurgical industries that are in dire need of these refractories, while the other factor is the advent of foreign exchange market, a situation that has lead to higher and unaffordable cost of processing the refractory materials needed by these industries ***(Ibitoye and Afonja, 1997).***

The major need of a foundry industry ranges from metal scarps, moulding sands, pattern wood, refractory linings e.t.c. Metal scraps are usually bought from scrap dealers. Such dealers normally collect scraps from industries and also depend on scavengers who pick up the scraps from dump sites and along the streets. The geographical location of this country placed her in a position where woods of different variety, are available in abundance. However, refractory lining, which is also a major requirement for foundry industry has been found to be grossly in-adequate. It is therefore necessary to look inward and research into the suitability of locally available raw materials for refractory linings for our foundry industry. Investigation has also revealed that, most of the foundry industries in Nigeria are still importing basic refractory lining despite the fact that the required materials for its production are locally available.

Refractories are heat resistant materials that can withstand high temperature without rapid physical and chemical deterioration. They are in-organic, non metallic and heat-resistant materials with different properties. Refractory may be acidic, neural or basic on the basis of chemical composition.

Majority of earlier indigenous researchers in this filed concentrated their efforts on acidic refractoriness while research work on neutral and basic refractoriness are still in-adequate hence the need for this work.

2.0 EXPERIMENTAL PROCEDURE

2.1 Sourcing of Materials

The basic raw materials used in this work are sample of Dolomite Procured from Igbeti (Oyo State) in the South –Western geo political zone of Nigeria (Lat 8.75^{0}N, Long 4.13^{0}E)

2.2 Equipment

The equipment and tools used in carrying out various tests on the samples collected include tape, weighing machine, hydraulic press, glass wares, sieve, collecting pan, spoon, stirrers, instron tester, muffle furnace, atomic absorption spectrometer (AAS), cylindrical steel mould, rammer, arbor press, time clock, rotary kiln, filter paper, pair of tongs and radiation pyrometer.

2.3 Methodology

From the Dolomite site at Igbeti, sample weighing 25kg were randomly collected from five points at three different locations spread at a distance of about 50 meters apart with an area of about 3,000 square meters. The estimation of the area was done by measuring the length and with of the sample area using folding tape. The area covered was then calculated.

2.3.1. Preparation of Test Pieces

The samples collected were crushed by means of 100Tonnes hydraulic press. The crushed samples were then calcined in a rotary kiln ran at a speed of 0.7rpm and heated to a temperature of 1,600^oC (Calcination Temperature of Dolomite) for a period of two hours to facilitate complete conversion of $MgCa(CO_3)_2$ which is unstable to CaO + MgO which is more stable and refractory.

The balanced chemical equation for the calcinations process is:

$$MgCa(CO_3)_2 \xrightarrow{Heat} CaO + MgO + 2CO_2\uparrow \quad ....1$$

The dead burnt Dolomite was allowed to cool. Thereafter, it was sieved into a mesh size of 250mm (medium sized grain). To prevent hydration: the sieved, calcined Dolomite was mixed with 12% tar (binder) and thoroughly stirred to form a homogeneous plastic paste.

Cylindrical steel rammer shown is plate I and specified in fig. I was then used to prepare test pieces properly rammed and extruded using an arbor press. The test pieces were then dried in a muffle furnace at intervals of 100^oC for every 10 minutes until a temperature of 1200^oc was attained. The samples were then soaked at 1200^0C for 8 hours and allowed to cool in the furnace for 24hours.

Plate 1: Cylindrical steel mould / rammer

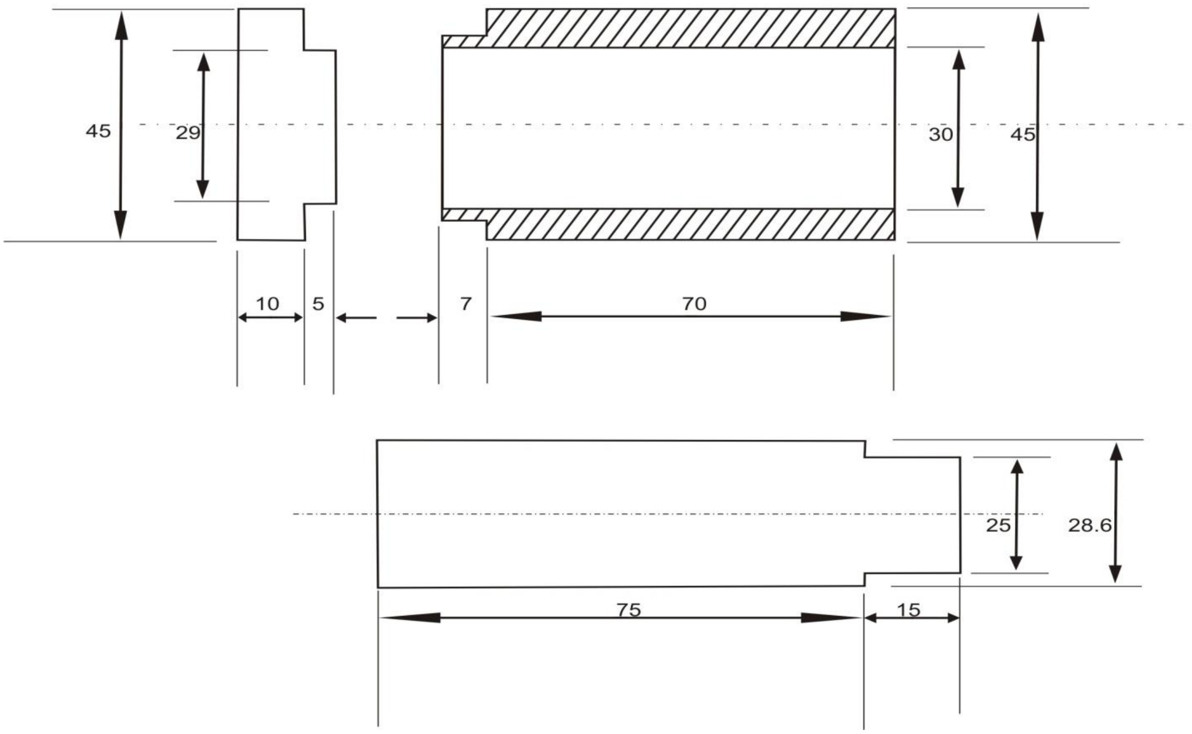

Fig I: Cylindrical Steel mould/Rammer

2.3.2. **Chemical Analysis**

The chemical composition of the samples were determined using the Atomic Absorption Spectrometer AAS. The process involves mixing of the samples in water, filtering and investigating the percentage of various oxides contained in it.

2.3.3 **Refractory Properties**

30 test pieces were prepared from the sample collected from the three locations mapped out on the Dolomite deposit being investigated (Igbeti). Three test pieces were used to conduct each of the tests carried out on the sample.

Plate II: Extruded Text pieces

The refractory properties investigated include permeability, thermal shock resistance, refractoriness, cold crushing strength, linear shrinkage, bulk density, apparent porosity and Loss and Ignition.

2.33.1. **Permeability to Air**

The permeability of materials to air is defined as the area of flow of air in milliliters per second through 1 square centimeter of the materials under a pressure gradient of 1 centimeter head of water per centimeter thickness (Chester, 1973).

Three test pieces prepared to specification of 30mm diameter and 50.8mm height from a standard rammer tube specified in figure I were air dried for 24 hours and then dried at 110^{o}C for 12 hours in an oven. 2000cm^3 of air was then allowed to pass through the test piece and the jar containing water. The pressure difference between the surfaces was measured by a manometer. The time taken for 2000cm^3 of air to pass through the test piece was recorded.

Permeability was then calculated from equation 2.

$$P_A = \frac{V \times h}{A \times P \times t} \quad \ldots\ldots (2)$$

Where P_A = Permeability number

V = Volume of air (cm^3)

h = height of specimen (mm)

A = cross sectional area of specimen (mm^2)

P = Pressure of air in cm of water

2.3.3.2 **Thermal Shock Resistance**

Test pieces were placed in a furnace which has been maintained at 1000^{0}C. This temperature was maintained for 10 minutes. The specimen were removed with a pair of tong from the furnace one after the other and then cooled for 10minutes on a platform. The specimens were returned to the furnace for a further 10 minutes. This process was continued until the test piece cracked and fell into pieces. The number of heating and cooling cycles for each specimen was recorded.

2.3.3.3 **Refractoriness**

Test piece was placed on refractory plaque along with standard cones whose melting point was known. The plaque was then put inside the furnace and the temperature was raised at a rate of 100^{0}C per hour. The test piece was removed, cooled to room temperature and observed under a microscope for any sign of crack, each time a cone is bent over. Refractoriness of the tested sample is the number of the standard cone that has bent. The temperature equivalent of the cone number is determined from the Orton series.

2.3.3.4 **Cold Crushing Strength**

Three test pieces of calcined dolomite samples were prepared to a size of 30mm x 50.8mm length. The test pieces were fired in a furnace at 1100^0C and maintained at this temperature for 6 hours after which the samples were cooled to room temperature. The specimen was then placed on a compressive tester (Instron tester) and load was applied axially by turning the hand wheel at a uniform rate till failure occurs. The tens meter reading was then recorded.

Cold crushing strength was calculated from equation3.

$$CCS = \frac{P_{max}}{A} \qquad (3)$$

Where CCS = Cold Crushing Strength (KN/m^2)

P_{max} = maximum load (KN), and

A = Area (m^2)

2.3.3.5. **Linear Shrinkage**

The test pieces rammed into standard cylindrical forms of known length and diameter were dried in an oven for 24hours at 110^0C, the test pieces were then removed and measured. Using equation 4, the linear shrinkage was calculated.

$$L_S = \frac{L_D - L_F}{L_D} \times 100\% \qquad (4)$$

Where L_S = Linear shrinkage

L_D = Dried length, and

L_F = Fired length

2.3.3.6. **Bulk Density**

This is a useful property of refractories, which defines the materials present in a given volume. An increase in bulk density of a given refractory increases its volume stability, its heat capacity, as well as resistance to slag penetration.

Representative samples of calcined dolomite rammed into standard shape of 30mm x 50.8mm length were prepared. The specimen were air-dried for 24 hours and then oven-dried at 110^oC, cooled in a desiccator and then weighed. The specimen was then transferred to a beaker containing water and heated for 30mins to expel the trapped air.

The specimen was then cooled and soaked weight taken. The specimen was then suspended in water contained in a beaker placed on a balance. The suspended weight was then taken Equation 5 was used to calculate the bulk density.

$$BD = D \times \frac{Pw}{W-S} \quad \text{-----------------------} (5)$$

Where BD = Bulk density (g/cm^3)

D = dried weight (g)

W = soaked weight (g)

Pw = density of water (g/cm^3)

S = suspended weight

2.3.3.7 **Apparent Porosity**

This is a measure of the volume of the open pores, into which a liquid can penetrate, as a percentage of the total volume. This is an important property especially in cases where the refractory is in contact with molten charge and slags. A low apparent porosity is desirable since it would prevent easy penetrations of the refractory. Prepared test pieces were air-dried for 24 hours and then oven-dried at 110^oC, cooled and then transferred into a desiccator and weighed. The specimen was then transferred into a 250ml beaker in an empty vacuum desiccator. Water was then introduced into the beaker until the test piece were completely immersed. The specimen was allowed to soak in boiled water for 30 minutes being agitated from time to time to release trapped air bubbles. The specimen was transferred into an empty vacuum desiccator to cool, excess weight (soaked) W, was determined. The specimen was then weighed suspended in water using beaker placed on balance. Hence, suspended weight S was obtained.

The apparent porosity was calculated using equation 6 below

$$A.P = \frac{W-D}{W-S} \times 100\% \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots.. \quad (6)$$

Where A.P = Apparent porosity

W = soaked weight (g)

D = dried weight (g) and

S = suspended weight (g)

2.3.3.8 **Loss on Ignition**

50g each of the samples collected from each of the three locations of the Igbeti site were dried at 110^oC and cooled in the desiccator. A porcelain crucible was cleaned, dried and weighed. The crucible containing the calcined dolomite sample was placed in a muffle furnace and heated to a temperature of 900^oC for three hours. The crucible and its contents were cooled in a desiccator and then weighed to an accuracy of 0.001g.

The loss of ignition was calculated from equation 7.

$$\text{Loss on ignition} = \frac{M_2 - M_3 \times 100\%}{M_2 - M_1} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots(7)$$

Where M1 = Mass of porcelain crucible

M2 = Mass of sample and porcelain crucible, and

M3 = Mass of fired calcined dolomite Sample and porcelain Crucible

For each of the three samples collected, the tests were repeated and the average of the evaluated values recorded.

3.0 RESULT AND DISCUSSION

The result obtained from tests conducted are given in tables 1 and 2

3.1 Chemical Analysis

Chemical analysis reveals that the samples contain high percentage of CaO and MgO. This implies that the material will produce refractory lining suitable for use in a basic environment. According to ASTM (1982), a standard basic refractory grade Dolomite should contain minimum of 1 % ($Fe0+Al_20_3$). Using ASTM as a basis, the percentage composition of CaO and MgO in the sample is within the range acceptable for a basic refractory grade Dolomite.

Table 1: Chemical composition of Igbeti Dolomite

Types of oxide	**SiO_2**	**AI_2O_3**	**Fe_2O_3**	**TiO_3**	**CaO**	**MgO**	**Na_2O**	**K_2O**	**L.O.I**
Composition (%)	1.89	-	-	-	32.65	19.76	-	-	45.90
Standard	Above	Below	Below	Below	Above	Above			42-47

Value	1.0	1.5	1.5	1.5	30.0	20.0			

3.2 **Refractory Properties**

3.2.1 **Permeability**

The samples give a value of 88mm/s. According to Gupta (2008) and ASTM (1982), permeability number range of 25-90mm/s is acceptable for Dolomite refractories.

Refractories under the influence of gasses and liquid should be impervious as this would prevent leakage of gasses and penetration of liquids through the walls of the furnace. Thus the value obtained is good enough as it falls within acceptable range.

3.2.2 **Thermal Shock Resistance**

TSR value of 23 cycles was obtained. According to Gupta (2008) the acceptable value for refractory grade Dolomite is 25 to 30 cycles. The value obtained falls slightly out of this range. This implies that the sample has to be worked upon by blending with suitable additives to improve its TSR Value.

3.2.3 **Refractoriness**

A value of 1778^{o}C (PCE) was obtained for refractoriness. This value falls within range of 1540^{o}C to 2130^{o}C acceptable for refractory grade Dolomite based on ASTM.

3.2.4 **Linear Shrinkage**

LS Value of 0.2% obtained is within the acceptable range of 0.2% specified for refractory grade Dolomite according to ASTM (1982) standard.

3.2.5 **Bulk Density**

A bulk density value of 2.84cm^3 was obtained. The acceptable range of B.D value according to ASTM (1982) for refractory grade Dolomite is 2.40g/cm3 and 3.20g/cm^3. The B.D value obtained falls within this range.

3.2.6 **Apparent Porosity**

Investigation gave an A.P. value of 19.28% for the sample collected. According to Gupta (2008) and in conformity with ASTM (1982), refractory grade Dolomite should have A.P value within the range of 18-20%. The value obtained falls within this range.

Table 2: Refractory Properties of Igbeti Dolomite

Property	**Sample 1**	**Sample 2**	**Sample 3**	**Mean**	**Standard Value**
Permeability to Air (mm/s)	87	88	89	88	25-90

Thermal shock resistance	22	23	24	23	23-28
Refractoriness (^oC)	1785	1785	1763	1778	1540-2130
Cold crushing strength (KN/cm^2)	652	648	650	650	Above 400
Linear Shrinkage (%)	0.2	0.2	0.2	0.2	0-0.2
Bulk Density (g/cm^3)	2.82	2.84	2.86	2.84	2.40-3.20
Apparent Porosity	19.28	19.30	19.26	19.28	18-20

The control values used in table 1 and 2 are in accordance with the standard of ASTM

4.0 CONCLUSION AND RECOMMENDATION

From the result of the tests conducted on the sample of Igbeti Dolomite, it was discovered that the Dolomite from this site is basic. This is consequent upon the fact its composition reflects higher percentage of CaO and MgO, negligible percentage of SiO_2 and absence of AI_2O_3 and other acidic components.

In agreement with the submission of Olaiya (2011), the Dolomite from this site is also calcitic as it contains higher percentage of CaO.

The Refractoriness of the sample is estimated at 1778^{o}C. Since the melting temperature of steel is about 1500^{o}C, the Dolomite from Igbeti can be used for lining of the steel making furnace. The refractory from this site can also be used in other furnaces and kilns where basic environment is required and operating temperature is below 1778^{o}C. Consequently, it is recommended for such.

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