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Experimental Investigation and Suitability of Alagutan Dolomite Deposit as Refractory Raw Materials

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***Abstract* – Refractory properties of dolomite deposit located at Alagutan (Lat 8^0 37′ N, Long 4^0 7′ E) Kwara State, Nigeria has been investigated. The need for this work arises from the dial need to re-examine our foundry industry with a view of sourcing her material needs locally and one of which is refractory lining. Samples of Dolomite were collected from Igbeti and subjected to various laboratory tests to determine the refractory properties of the material and determine its suitability for furnace lining in foundries. This investigation revealed the following results: permeability to air of 86mm/s, thermal shock resistance of 22cycles, refractoriness of 1778^{0}C, cold crushing strength of 652KN/cm^2, and linear shrinkage of 0.2%, bulk density of 2.76 g /cm^3, and apparent porosity of 21.17%. The results obtained compares compare favourably with standard values acceptable for refractory grade dolomite. The chemical composition of the samples was also found to lie within acceptable range for basic refractories. Conclusively, it was revealed that Alagutan dolomite is suitable for lining of furnaces requiring basic environment at less than 1778^{0}C operating temperatures.**

***Keywords* – Basic, Calcitic, Dolomite, Foundry, Refractory.**

I. Introduction

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, neutral or basic on the basis of chemical composition [1].

According to [2], refractory materials are non-metallic materials that have unusual high melting temperatures and maintain their structural properties at very high temperatures. Principally, they are composed of oxides of silicon and aluminium. Refractories are employed in great quantities in the metallurgical, glassmaking, and ceramics industries, where they are formed into variety of shapes to line the interiors of furnaces, kilns, and other devices that process materials at very high temperatures. Because of the high strengths exhibited by their primary chemical bonds, many refractory materials possess unusually good combinations of high melting point and chemical inertness. Clay based refractories are the fireclay materials and are made from clays containing the aluminosilicate mineral-kaolinite ($Al_2[Si_2O_5][OH]_4$) plus impurities such as alkalis and iron oxides. The alumina (Al_2O_3) content ranges from 25 to 45 percent.

In metallurgical, cement, glass and machine tools industries where kilns and furnaces are used for value addition process to materials, refractories are very necessary. Dolomite refractories are currently in use in some foreign countries such as china, France, England, India, etc. Dolomite refractories have wide applications in the steel industry where it is used in openhearth, basic oxygen converters and other steel refining systems [3].

Refractories belong to the class of ceramic materials which are employed for high temperature applications, usually above 1100^{o}C and are classified based on the impurity content and the alumina-to-silica ratio (Al_2O_3/SiO_2) [4].

The composition of refractories determines their activities in steel making slags. Refractories composed mainly of silica (an acidic oxide) should not be in contact with basic slags while those prepared from basic oxides (MgO,CaO) should be protected from contact with acidic slags, otherwise their particles in the lining will be slaked vigorously resulting in quick failure of the lining [5] .

Dolomite mineral is a double carbonate of calcium and magnesium having the formula $CaMg(CO_3)_2$. It is slightly hard, transparent and forms rhombohedron as its typical crystal habit. Dolomite used for refractory purpose should be hard and compact with uniform texture containing very low percentages of iron, silicon, Alumina, etc. This is because these impurities adversely affect the refractoriness of dolomite refractories [6] .

Plate I: Dolomite Crystal.

Many limestones contain a small amount of dolomite and the term dolomite as a rockname is usually confined to rock with more than 15% magnesium carbonate. Dolomite

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may exist as evaporates. Calcite limestones are readily dolomitized, and in modern sediments dolomites seems to form as a result of penecontemporaneous [7].

Dolomite is of economic importance to man by virtue of its versatile, physical, mineralogical and chemical properties that recommend them for numerous purposes and building materials. It is also used for making refractory furnace lining and a source of carbondioxoide [8]. Dolomite rocks are present as sedimentary layers in many areas and as product of regional metamophism of carbonate rocks. Dolomite is used as ornamental stone and raw materials to manufacture cement and magnesium oxide for refractories [9]. However, Dolomite is a kind of sedimentary rock resembling marble or lime stone but rich in magnesium carbonate. Nigeria has appreciable deposit of Dolomite at various locations though their properties differ from site to site on account of geological differences. Ironically, the bulk of basic refractory lining which is obtainable from dolomite is imported. The present economic state imposes the need for internal sourcing of this vital foundry consumable to meet up increasing demands. Over 70% of our casting needs are still imported, over five decades after our political independence. This is a reflection of low level of commitment to development [10].

Despite having extensive deposits of clay, dolomite and other mineral used for production of refractories in Nigeria, This country continues to depend on external sources of refractory materials for many of her industries [11]. Nigeria imported about 27 million metric tonnes of refractory materials in 1987 [12].

In view of all these facts, there is therefore every need to research more into the characteristics of the locally available dolomite deposits and ascertain their suitability for various useful purposes rather than relying on imported raw materials. This will go a long way to further reduce dependence of our foundry industry on imported refractory and consequently conserve our foreign exchange and hence give the nation's economy a boost.

The objective of the present study is to determine the composition as well as refractory properties of the Dolomite obtained from Alagutan and ascertain its' suitability as a lining material for foundry furnaces.

II. Materials and Methods

The basic material used in this work are samples of Dolomite procured from Alagutan (Kwara State) in the north central geo-political zone of Nigeria (Lat 8^0 37´ N, Long 4^0 7´E).

The various tests on the samples collected were carried out using tools and equipment which include the following:

- Folding tape
- Weighing machine
- Hydraulic press
- Glass ware
- Sieve
- Collecting pan
- Spoon
- Stirrers
- Instron tester
- Muffle furnace
- Atomic absorption spectrometer (AAS)
- Cylindrical steel mould
- Arbour press
- Time clock
- Pair of tongs
- Radiation pyrometer

III. Experimental Procedure

From the Dolomite site at Alagutan, sample weighing 25kg were randomly collected from five points at three different locations spread at a distance of about 50 metres within an area of about 3,000 square metres. The length and breadth of the sample area were measured by means of the folding tape and the sample area calculated.

Sample Preparation

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^{o}C (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 stable and refractory.

Plate II: Calcined/Fired Dolomite.

The balanced chemical equation for the calcinations process is as shown below:

$$MgCa(CO_3)_2 \xrightarrow{\text{Heat}} CaO + MgO + 2CO_2 \qquad (1)$$

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

The cylindrical steel rammer shown in plate III and specified in fig 1 was then used for preparation of test pieces. The test pieces were properly rammed and extruded with the aid of an Arbor press. The test pieces were then dried in a muffle furnace at intervals of 100^{o}C for every 10 minutes until a temperature of 1200^{o}C was attained. The samples were then soaked at 1200^{o}C for 8 hours and allowed to cool in the furnace for 24 hours.

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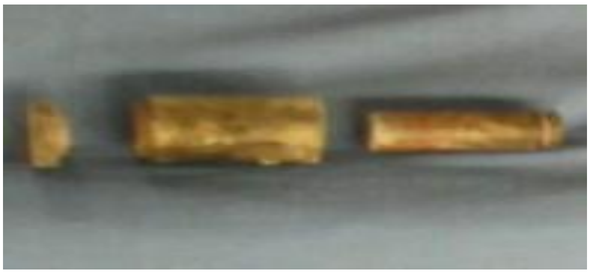

Plate III: Cylindrical Steel Mould

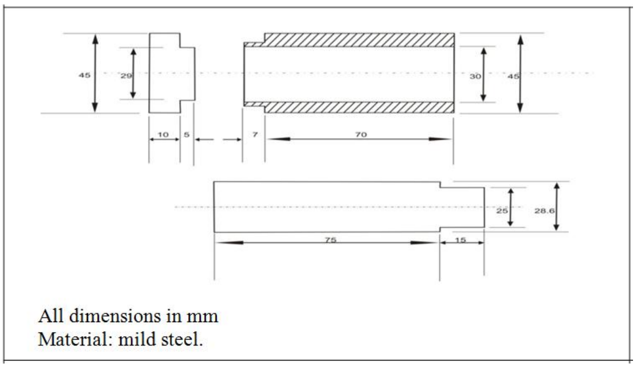

Fig.1. Cylindrical Steel mould

Chemical Analysis

The chemical analysis of the samples were carried out by means of Atomic Absorption spectrometer AAS. The process involves mixing of the samples in water, filtering and investigating the percentage of various oxides contained in it.

Refractory properties

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

Plate IV: Extruded test pieces

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

Permeability to air

The permeability of materials to air is defined as the area of flow of air in millimetres per second through 1 square centimetre of the materials under a pressure gradient of 1 centimetre head of water per centimetre thickness [13].

Three test pieces prepared to specification of 30mm diameter and height of 50.8mm from the cylindrical steel mould specified in fig. 1 were air dried for 24 hours and then dried at 110^{o}C for 12 hours in an oven. 2000 cm^{3} of air was then allowed to pass through the test piece and the jar containing water. The time taken for this volume of air to pass through the test piece was then noted and recorded. Permeability was then calculated from equation 2 [14].

$$P_A = \frac{v \times h}{A \times p \times t} \qquad (2)$$

Where, P_A = Permeability number
v = Volume of air (cm^{3})
h = height of specimen
A = Cross sectional area of specimen (mm^{2})
P = Pressure of air in cm of water
t = time taken (s)

Thermal Shock resistance

Test pieces were placed in a muffle furnace, which has been maintained at 1000^{o}C. This temperature was maintained for 10mins. The specimens were removed with a pair of tongs from the furnace one after the other and then cooled for 10 minutes. This process was continued until the test piece cracked and shatters into pieces. The number of heating and cooling cycles for each specimen was noted and recorded [14].

Refractoriness

Refractoriness of a material is the temperature at which the material softens and hence the measure of the fusibility of the material [15]. Test piece was placed on refractory plaque along with standard cones whose melting point was known. The plaque was then placed inside the furnace and the temperature was raised at a rate of 100^{o}C per hour. The test piece was then removed, cooled to room temperature and observed under a microscope for any crack, each time a cone is bent over. Refractoriness of the tested sample is the number of the standard cone that has bent [14].

Number of standard cones that has bent and the temperature equivalent of the cone number are determined from the Orton series shown on table 1.

Table 1: Standard Pyrometric Cone Equivalent

Cone No.	Value of Orton Cone, ^{0}C (British Standard)	Value of Segar Cone, ^{0}C (German standard)
12	1337	1375
13	1349	1395
14	1398	1410
15	1430	1440
16	1491	1470
17	1512	1490
18	1522	1520
19	1541	1530
20	1564	1540
23	1605	1560
26	1621	1585
27	1640	1605
28	1646	1635
29	1659	1655

30	1665	1680
31	1683	1695
31½	1699	-
32	1717	1710
32½	1724	-
33	1743	1730
34	1763	1755

[15]

Cold crushing strength

Cold crushing strength determines the ability of a material to withstand handling and shipping.

Three test pieces of calcined Dolomite prepared to specification shown in fig 1 were fired in a furnace at 1100°C and maintained at this temperature for 6 hours. Samples were then cooled to room temperature and 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 tensometer reading was then noted and recorded [14].

Cold crushing strength was then calculated from equation 3.

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

Where, CCS = cold crushing strength (KN/cm^2)
P_{max} = maximum load (KN), and
A = Area (cm^2)

Linear shrinkage

The test pieces rammed into standard cylindrical forms of the specification given above were dried in an oven for 24 hours at 110°C. The test pieces then removed and measured. Equation 4 was then used to calculate linear shrinkage [14].

$$Ls = \frac{LD - LF}{LD}\ x\ 100\% \quad (4)$$

Where, LS = Linear shrinkage
LD = Dried length, and
LF = Fired length

Bulk density

This is a property of refractories which defines the materials present in a given volume of it. An increase in the bulk density of a given refractory increases its volume stability, its heat capacity as well as resistance to slag penetration. Samples of calcined dolomite rammed into standard specification were air dried for 24 hours and then oven-dried at 110°C, cooled in a desicator and then weighed. The specimen was then transferred to a beaker containing water and heated for 30 minutes to expel the trapped air.

The specimen was then cooled and soaked weight taken. The specimen was then suspended in water contained in beaker placed on a balance and the suspended weight taken.

Equation 5 was then used to calculate the bulk density [14].

$$BD = D\ x \frac{P_w}{W - S} \quad (5)$$

Where, BD = Bulk desnity (g/cm^3)
D = dried weight (g)
W = soaked weight (g)
P_w = density of water (g/cm^3)
S = suspended weight (g)

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 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°C, cooled and then transferred to a desicator and weighed. The specimen was then transferred into a 250 ml beaker in an empty vacuum desicator. Water was then poured into the beaker until the test piece was 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 desicator to cool. Excess weight (soaked) was then determined. The specimen was then weighed suspended in water using beaker placed on balance. Hence, suspended weight was obtained.

The apparent porosity was calculated using equation 6 below [14].

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

Where A. P = Apparent porousity
W = Soaked weight (g)
D = dried weight (g), and
S = Suspended weight (g)

Loss on ignition

50g each of the samples collected from each of the three locations of the Alagutan site were dried at 110°C and cooled in the desicator. 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°C for three hours. The crucible and its contents were cooled in a desicator and then weighed to an accuracy of 0.001g.

The loss on ignition was calculated from equation 7 [14].

$$Loss\ on\ Ignition = \frac{M_2 - M_3}{M_2 - M_1}\ x\ 100\% \quad (7)$$

Where, M_1 = Mass of porcelain crucible
M_2 = Mass of sample and porcelain crucible, and
M_3 = 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.

IV. Results and Discussion

The results obtained from the tests conducted are given in tables 1 and 2.

Chemical Analysis

Chemical analysis reveals that the material contains high percentage of CaO and MgO.

Table 2: Chemical Composition of Alagutan Dolomite

Types of oxide	$SiO_{2\%}$	Al_2O_3 (%)	Fe_2O_3 (%)	T_iO_3 (%)	CaO (%)	MgO (%)	Na_2O (%)	K_2O (%)	L. O. I (%)
Composition (%)	2.12	-	-	-	33.10	21.28	-	-	43.50
Standard value	>1.0	<1.5			>30.0	>20.0			42.47

This sample of dolomite contains high percentage of CaO and MgO. This implies that refractory lining obtained from it is chemically basic i.e. at high temperature, it exhibits the properties of a base such as turning red litmus blue, reacting with acids to produce salt and water, reacting with ammonium salts to generate ammonia gas, etc. Hence, the material will produce refractory lining suitable for use in a basic environment. A standard basic refractory grade dolomite should contain minimum of 30% Ca), minimum of 20% MgO, minimum of 1% silica (S_iO_2) and maximum of 1.5% of (FeO + Al_2O) [15]. The results obtained agree with this standard.

Permeability

The samples gave an average value of 86mm/s. According to [14] and [15], permeability number range of 25 – 90mm/s is acceptable for dolomite refractories. Subsequently, refractories under the influence of gases 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 the acceptable range.

Thermal Shock Resistance

TSR value of 23 cycles was obtained. According to [14], the acceptable value for refractory grade dolomite is 25 to 30 cycles [14]. 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.

Refractoriness

A value of 1778^{o}C (PCE) was obtained for refractoriness. This value compares favourably with the range of 1540^{o}C to 2130^{o}C acceptable for refractory grade dolomite [14].

Linear Shrinkage

Value of 0.2% obtained for L.S is within the acceptable range specified for refractory grade dolomite [14].

Bulk Density

An average value of 2.76g/cm^3 was obtained for bulk density. This result lies within the acceptable range of (2.40 – 3.20) g/cm^3 for dolomite refractories [16].

Apparent Porosity

This investigation gave an average value of 21.71% for A.P for the sample collected. An A.P value of 18 – 20% range is acceptable for refractory grade dolomite [16]. The obtained result fell slightly outside this acceptable range but [15] cited an A.P of 23% for dolomite refractories.

Table 3: Refractory Properties of Alagutan Dolomite

Property	Sample 1	Sample 2	Sample 3	Mean	Standard value
Permeability to Air (mm/s)	85	87	86	86	25 – 90
Thermal shock resistance (cycles)	21	23	22	22	23 – 28
Refractoriness (^{0}C)	1785	1763	1785	1778	1540 – 2130
Cold crushing strength (KN/cm^2)	655	650	650	652	Above 400
Linear shrinkage (%)	0.2	0.2	0.2	0.2	0 – 0.2
Bulk Density (g/cm^3)	2.78	2.74	2.76	2.76	2.40 – 3.20
Apparent porosity (%)	21.68	21.74	21.70	21.71	18 – 20

V. Conclusion

It was shown in this work that on the basis of the physio-chemical characteristics, Dolomite from Alagutan is basic. This is consequent upon the fact that the composition reflects higher percentage of CaO and MgO, negligible percentage of SiO_2 and absence of Al_2O_3 and other acidic components.

The Dolomite from this site is characterised as being calcitic as it contains higher percentage of CaO [1]. The sample has a refractoriness estimated at 1778^{o}C. Since steel melts at a temperature of about 1500^{o}C, refractory produced from Alagutan Dolomite is suitable for lining steel making furnaces. It can also be suitably used in other furnaces and kilns requiring basic environment and below 1778^{o}C operating temperatures. Hence, it is recommended for use in such furnaces and kilns.

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