Effect of Type of Aggregate on the Properties of Refractory Concrete

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ABSTRACT: Low cement refractory concrete samples were prepared by mixing cement (containing 50% alumina) in percentages ranging from 10 to 20% with aggregate and the necessary amount of water. Two types of refractory aggregate were used: Bauxite containing 81% alumina and grog containing 52% alumina. Four particle sizes of each aggregate were used each time. The cast samples were left in their moulds for 24 hours in a 100% relative humidity cabin. The de–molded specimens were left in open air until their moisture content reaches 3–6%, then put in a drying oven at (110 ± 5) ºC until reaching constant weight. They were then tested for phase constitution, water absorption, bulk density, apparent porosity and cold crushing strength (after 28 days curing). It was found that bauxite based samples gave better results than those prepared with grog. It was also found using statistical analysis that the percent cement used affects all properties much more than does the particle size of aggregate.


Key Words: Refractory concrete – Alumina – Grog – Sodium citrate

1. INTRODUCTION

An analysis of the composition, the structure, and the properties of the conventional refractory concretes shows that their refractory properties are governed by the filler contained in them. The binder component (bonding agent) of the concretes imparts the strength required during transportation and erection; this strength is attained after setting and drying. During subsequent heating up to the temperatures preceding sintering, irreversible destructive processes occur, as a rule, in the binder. In view of the fact that the binder (along with the finely milled additives) forms a continuous matrix phase in the structure of the concrete, the thermo–mechanical characteristics of the material are adversely affected. Therefore, in order to improve the existing refractory concretes and to create new concretes, it is necessary to decrease the content of the conventional binders (e.g., high–alumina cements) in them to the maximum possible extent or to produce them without introducing the conventional (common) binders (1, 2, 3, 4).

Thermally stable aggregates combined with a bonding agent are the principal ingredients of a monolithic refractory. These raw materials are available both naturally and artificially. Raw materials available in nature unavoidably vary slightly in their compositions. But it is important to take advantage of the characteristics of these natural minerals that cannot be developed artificially rather than to avoid their use due to variations of chemical composition.

Unlike natural raw materials, artificial raw materials allow adjustment of chemical composition as well as their mineral constituents, and it is possible to get a uniform quality.

One common type of aggregate is bauxite, a raw material for alumina containing about 60% alumina. When calcined, the alumina level is usually raised above 85%. Bauxite for refractories is calcined in a rotary kiln to make a stable product. Calcined bauxite contains corundum as its principal component, mullite and a small glassy phase.

On the other hand, grog is an artificial aggregate usually obtained from crushed defective refractory bricks. Its alumina content depends on that of the original bricks. It usually ranges from 40 to 80%.

Other types of aggregate include diaspore (Al₂O₃, H₂O), corundum (Al₂O₃), magnesia (MgO), zirconia (ZrO₂), etc.

In the present paper are studied the physico–mechanical properties of refractory concrete samples prepared from bauxite and grog with varying amounts of cement and varying particle size of aggregate.

2. EXPERIMENTAL

2.1 Raw Materials:
The raw materials used are:
- Refractory cement containing 50% alumina was obtained from Lafarge Cement.
- Calcined bauxite was obtained from the Alexandria Company for Refractories with an alumina content exceeding 80%.
- Grog was obtained from previously fired defective bricks that were crushed, ground and screened.

2.2 Particle Size Distribution of Aggregate:
In order to determine the grain size distribution, the procedure described by ASTM D 422/2007 was used. The standard sieves method was applied using screen apertures ranging from 6.68 mm (3 mesh) to 74 μm (200 mesh).

2.3 X–Ray Fluorescence Analysis (XRF):
X–ray fluorescence spectrometry (XRF) is a method of elemental analysis that assesses the presence and concentration of various elements by measurement of secondary X–radiation from the sample that has been excited by an X–ray source. The analysis was run on a AXIOS, panalytical 2005, Wave length Dispersive (WD–XRF) Sequential Spectrometer available at the National Research Center in Cairo.

2.4 X–Ray Diffraction (XRD):
X–Ray diffraction analysis differs from XRF in that it identifies, usually in a qualitative way, the phases present in the analyzed material rather than the elements.

For X–Ray diffraction study of bauxite and grog analysis, the aliquots for bulk mineral analysis were finely ground (–200 mesh), mounted randomly on an aluminum holder, and analyzed by a BRUKUR D8 ADVANCE COMPUTERIZED X–Ray Diffractometer apparatus (available at the Center of Metallurgical Research and Development Institute, Cairo) with mono–chromatized Cu Kα radiation, operated at 40 kV and 40 mA.

2.5 Preparation of Specimens:
Forty pastes of different size formulations for both bauxite and grog at different percentages cement (20%, 17.5%, 15%, 12.5%, 10%) by weight were kneaded with an adequate amount of water, which was determined for each batch according to the standard "good ball in hand test". The mixed batches were then cast into cubes of 50 mm side length using a vibrating table at a frequency of 50 Hz and 4 minutes. The cast samples were then put into an open air until their moisture content reaches 3–6%, then put in the drying oven at (110 ± 5) °C until reached constant weight. They were then tested for water absorption, bulk density and apparent porosity and cold crushing strength.

2.6 Apparent Porosity, Water Absorption, and Bulk Density:
These properties are determined according to ASTM Standards C 20/2007. For each test, the average measurements for five specimens at least are calculated.

The five specimens for each test are weighed to get the dry weight (D) for each. The test specimens are then placed in water and boiled for 2 h in a boiler and kept entirely covered with water with no contact with the heated bottom of the container. They are cooled to room temperature while still completely covered with water. The weight (S) of each test specimen is determined after boiling and while suspended in water. The saturated weight (W) is also determined.

Apparent porosity is calculated from:

$$P,\% = \frac{W - D}{V} \times 100$$

Water absorption is calculated from:

$$A,\% = \frac{W - D}{D} \times 100$$

While bulk density is calculated as follow:

$$\rho_B = \frac{D}{V} \times 100$$

Where:
P = apparent porosity, (%);
W = weight of the specimen as saturated with water, (g);
D = dry weight, (g);
S = weight of the specimen as suspended in water, (g);
V = exterior volume = W – S, (cm³);
A = water absorption, (%);
\(\rho_B\) = bulk density, (g/cm³).

2.7 Cold Crushing Strength:
This was done to determine the compression stress to failure of samples consisting of three specimens cured for 28 days. It represents an
indication of its probable performance under load. Each specimen was placed between two plates of the compression strength tester. This was followed by the application of an axial uniform load. The load at which a crack appears on the sample was noted, and it is calculated according to BS EN Standards 993–5/2000 (8):

\[ C.C.S \ (\sigma_c) = \frac{W}{a} \] (4)

Where:
\( \sigma_c \) = cold crushing strength, (MPa);
\( W \) = total maximum load at 3% deformation or at visible failure, (N);
\( a \) = average of gross areas of the two faces, (mm²).

3. RESULTS AND DISCUSSION
3.1 Particle Size Distribution of Grog and Bauxite:
Grog and Bauxite were screened to different size fractions onto a set of standard sieves ranging form 3 mesh (Opening = 6.680 mm) down to 200 mesh (opening = 0.074 mm). The mean particle size of a fraction passing through a certain sieve and retained over the next was taken as the arithmetic average of the two openings. This way, the following mean sizes were used: 4.699 mm, 2.794 mm, 1.651 mm, 1.168 mm, 0.991 mm, 0.295 mm, 0.175 mm, 0.147 mm, and 0.074 mm.

Figure (1) shows the cumulative screen analyses for grog and bauxite used in the present investigation.

![Fig. (1): Particle Size Distribution of Aggregates](image)

3.2 Chemical Analysis of Raw Materials:
Table (1) shows the XRF results related to the chemical analysis of the refractory cement used, bauxite and grog.
Table (1): Chemical Analysis of Materials Used

<table>
<thead>
<tr>
<th>Constituents (wt. %)</th>
<th>Cement</th>
<th>Bauxite Sample</th>
<th>Grog Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>5.5</td>
<td>9.264</td>
<td>26.640</td>
</tr>
<tr>
<td>TiO₂</td>
<td>—</td>
<td>1.451</td>
<td>3.740</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>52.95</td>
<td>81.291</td>
<td>51.929</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>2.5</td>
<td>1.816</td>
<td>2.994</td>
</tr>
<tr>
<td>MgO</td>
<td>Traces</td>
<td>0.372</td>
<td>0.510</td>
</tr>
<tr>
<td>CaO</td>
<td>38.05</td>
<td>0.435</td>
<td>1.215</td>
</tr>
<tr>
<td>Na₂O</td>
<td>&lt; 0.1%</td>
<td>0.066</td>
<td>1.418</td>
</tr>
<tr>
<td>K₂O</td>
<td>&lt; 0.1%</td>
<td>0.174</td>
<td>4.901</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>—</td>
<td>0.542</td>
<td>1.056</td>
</tr>
<tr>
<td>SO₃</td>
<td>—</td>
<td>0.020</td>
<td>0.875</td>
</tr>
<tr>
<td>Cr₂O₃</td>
<td>—</td>
<td>0.120</td>
<td>0.069</td>
</tr>
<tr>
<td>Co₃O₄</td>
<td>—</td>
<td>0.084</td>
<td>0.039</td>
</tr>
<tr>
<td>Ga₂O₃</td>
<td>—</td>
<td>0.023</td>
<td>0.014</td>
</tr>
<tr>
<td>SrO</td>
<td>Traces</td>
<td>0.16</td>
<td>0.106</td>
</tr>
<tr>
<td>Y₂O₃</td>
<td>—</td>
<td>0.021</td>
<td>0.020</td>
</tr>
<tr>
<td>ZrO₂</td>
<td>—</td>
<td>0.272</td>
<td>0.190</td>
</tr>
<tr>
<td>Nb₂O₅, La₂O₃, CeO₂, Nd₂O₃, ThO₂</td>
<td>&lt; 0.1%</td>
<td>&lt; 0.1%</td>
<td>&lt; 0.1%</td>
</tr>
<tr>
<td>WO₃</td>
<td>—</td>
<td>0.237</td>
<td>—</td>
</tr>
<tr>
<td>PbO</td>
<td>Traces</td>
<td>1.6</td>
<td>0.011</td>
</tr>
<tr>
<td>Cl</td>
<td>—</td>
<td>0.022</td>
<td>4.183</td>
</tr>
<tr>
<td>L.O.I</td>
<td>—</td>
<td>1.811</td>
<td>—</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>≈100</td>
<td>≈100</td>
<td>≈100</td>
</tr>
</tbody>
</table>

3.3 XRD of Raw Materials:

The following figures (2 and 3) show the XRD pattern obtained on investigating bauxite and grog. As can be seen from the figures, calcined bauxite consists exclusively of corundum (Al₂O₃) and mullite (3Al₂O₃.2SiO₂). This is expected from the phase equilibrium diagram Al₂O₃ – SiO₂ for compositions containing > 80% alumina (9). On the other hand, the XRD pattern shows besides the expected phases of mullite and quartz some non-equilibrium phases of corundum and cristobalite, are also present lines of halite present as impurity.
In order to assess the effect of particle size of the aggregate used, bauxite and grog, on the workability, physical and mechanical properties of refractory concrete paste, four different particle size mixes were used. Each is a combination of three particle size ranges as is usually the case when wanting to maximize compactness. Table (2) shows the four mixes together with the average particle diameter of each as calculated using the method suggested by McCabe et al\textsuperscript{(10)}.

### 3.4 Mean Particle Size of Aggregates:

![Fig. (2): XRD Pattern of Calcined Bauxite](image1)

![Fig. (3): XRD Pattern of Grog](image2)
Table (2): Mean Particle Size of Aggregate Formulation Used

<table>
<thead>
<tr>
<th>% Weight</th>
<th>0–1 mm</th>
<th>1–3 mm</th>
<th>3–5 mm</th>
<th>Mean Particle Size (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>10</td>
<td>75</td>
<td>15</td>
<td>1.63</td>
</tr>
<tr>
<td>B</td>
<td>25</td>
<td>60</td>
<td>15</td>
<td>1.19</td>
</tr>
<tr>
<td>C</td>
<td>40</td>
<td>45</td>
<td>15</td>
<td>0.94</td>
</tr>
<tr>
<td>D</td>
<td>55</td>
<td>30</td>
<td>15</td>
<td>0.78</td>
</tr>
</tbody>
</table>

3.5 Effect of Mean Particle Size on Water Consumption:

During mixing, the addition ratio of water is directly affects the final product considerably (2). When the amount of coarse particles in the particle size distributions used in this investigation increases, therefore the specific surface area of the particles decreases and less water is consumed because of this. The amount of consumed water as a function of the mean particle size for both bauxite and grog are shown in figures (4 and 5).

It appears from these figures that the amount of water used increases with a decrease in particle size and with the amount of cement used. It is also seen that water consumption for samples containing grog is higher than for samples containing bauxite; these values range from 7.5 to 9.1% in case of bauxite based formulations against 9.5 to 10.9% in grog based formulations. This is presumably due to the presence of much more open pores in grog than in bauxite particles. To assess this point, the apparent porosity of samples of both types of particles was determined. It was found to be 6.7% for bauxite particles against 16.5% for grog particles.
Using the excel DATA ANALYSIS module it was possible to establish correlation tables in both cases that show the relative influence of percent cement and particle size on the percent water added. Such tables are shown below.

**Table (3): Correlation Table for Water Added for Bauxite Based Mixes**

<table>
<thead>
<tr>
<th></th>
<th>% Cement</th>
<th>Particle Size</th>
<th>% Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Cement</td>
<td>1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Particle Size</td>
<td>0</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>% Water</td>
<td>0.642372</td>
<td>-0.7489</td>
<td>1</td>
</tr>
</tbody>
</table>

**Table (4): Correlation Table for Water Added for Grog Based Mixes**

<table>
<thead>
<tr>
<th></th>
<th>% Cement</th>
<th>Particle Size</th>
<th>% Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Cement</td>
<td>1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Particle Size</td>
<td>0</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>% Water</td>
<td>0.696767</td>
<td>-0.68928</td>
<td>1</td>
</tr>
</tbody>
</table>

The previous tables point out to the following:

- First, the relation between the percent water added and percent cement used is an increasing relation. On the other hand the negative sign associated with the effect of particle size means an inverse relation between the percent water added and particle size.

- Second, it appears that in case of using bauxite the effect varying particle size on the percent water added is higher than that of varying the cement ratio. On the other hand, on using grog, the two variables have comparable effects.
3.6 Effect of Mean Particle Size on Water Absorption:

Figures (6 and 7) show the relations between the percent water absorption of cast cubes and the mean particle size of either bauxite or grog.

From these two figures, it can be seen that the percent water absorption appreciably decreases with an increase in cement content. However, as the cement content exceeds 15%, its effect on water absorption diminishes. This is expected since higher cement content will have for effect to enhance the closure of available pores.

Of interest is the difference between the water absorption values observed in either case. It is clear from Fig. (6) that the values of water absorption in case of bauxite based formulations range from about 4.8% to 9.5% depending on the cement level and particle size. In case of using grog the range of water absorption is 5.3 – 10.5% showing that the use of grog has lead to higher water absorption presumably due to increased open porosity of samples.

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**Fig. (6): Effect of Amount of Cement Used and Mean Particle Size of Aggregate on Percent Water Absorption for Bauxite Based Mixes**

**Fig. (7): Effect of Amount of Cement Used and Mean Particle Size of Aggregate on Percent Water Absorption for Grog Based Mixes**
Also these figures show that the percent water absorption is only slightly affected by an increase in particle size. To assess this effect the excel DATA ANALYSIS module was used to establish correlation tables that show the relative influence of percent cement and particle size on the percent water absorption in both cases. Such tables are shown below.

Table (5): Correlation Table for Percent Water Absorption for Bauxite Based Mixes

<table>
<thead>
<tr>
<th>% Cement</th>
<th>Particle Size</th>
<th>% W.A.</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Cement</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>Particle Size</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>% W.A.</td>
<td>−0.82455</td>
<td>−0.39599</td>
</tr>
</tbody>
</table>

Table (6): Correlation Table for Percent Water Absorption for Grog Based Mixes

<table>
<thead>
<tr>
<th>% Cement</th>
<th>Particle Size</th>
<th>% W.A.</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Cement</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>Particle Size</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>% W.A.</td>
<td>−0.79392</td>
<td>−0.42302</td>
</tr>
</tbody>
</table>

These tables show that both variables affect negatively the percent water absorption where as the effect of the variation of percent cement is almost as twice as that of fineness.

3.7 Effect of mean particle Size on Apparent Porosity:

Fig. (8) and Fig. (9) show the relations between apparent porosity and the mean particle size for formulations containing bauxite and grog.

From these two figures, it can be seen that the apparent porosity decreases with an increase in cement content. This is expected since higher cement content will have for effect to enhance the closure of available pores. Also these figures show that the apparent porosity is negatively affected by an increase in particle size.

Using the excel DATA ANALYSIS module it was possible to establish correlation tables in both cases that show the relative influence of percent cement and particle size on the apparent porosity. Although such tables are not shown, their result indicates that is both variables affect negatively the apparent porosity where as the effect of the variation of percent cement on porosity is almost as twice as that of fineness.

![Fig. (8): Effect of Amount of Cement Used and Mean Particle Size of Aggregate on Percent Apparent Porosity for Bauxite Based Mixes](http://www.americanscience.org)
Fig. (9): Effect of Amount of Cement Used and Mean Particle Size of Aggregate on Percent Apparent Porosity for Grog Based Mixes

3.8 Effect of Particle Size Distribution on Bulk Density:

Fig. (10) and Fig. (11) show the relations between bulk density and the mean particle size for formulations containing bauxite and grog.

From these two figures, it can be seen that the bulk density increases with an increase in cement content. This is expected since higher cement content will have for effect to decrease porosity. Also these figures show that the bulk density slightly increases with an increase in particle size.

On the other hand, due to their lower porosity, the bulk density of samples containing bauxite is higher than that of samples containing grog, for the same particle size. For example, at mean particle size = 1.63 mm, the bulk density of samples containing bauxite ranged from 2.37 to 2.96 g/cm³, depending on the amount of cement added while it ranged from 2.3 to 2.9 g/cm³ for grog containing samples.

Also, due to the irregular shape of the grog particles with respect to bauxite, the packing efficiency of a body containing grog is less than that of bauxite. This assists the increased density in case of using bauxite.

Correlation tables were established to show the relative effect of variations in cement content and particle size on samples containing either bauxite or grog (Not shown). These tables show that both cement content and higher particle size favor higher bulk density although the effect of cement variation on bulk density is more pronounced than that of particle size.

Fig. (10): Effect of Amount of Cement Used and Mean Particle Size of Aggregate on the Bulk Density of Bauxite Based Mixes
3.9 Effect of Particle Size Distribution on Cold Crushing Strength:

Figures (12 and 13) show the relations between cold crushing strength and the mean particle size used for different cement contents (bauxite and grog). Three specimen samples were first prepared and air cured for 28 days before subjecting to the test.

From those figures, it could be seen that the compressive strength of specimens increases, as expected with an increase in cement content. The effect of mean particle size is however more complicated. All curves drawn seem to follow the same pattern: First, the cold crushing strength increases up to a mean particle size of about 0.95 mm then decreases with further increase in particle size. This mean particle size corresponds to formulation C in which the fine particles (D < 1 mm) constitute 40% of the mix whereas the coarse portion represents the remaining 60% (Table 2). Such recipe approaches a state of minimum total porosity \(^{(11)}\). This had for effect to maximize the compactness of the mix leading to a maximum value in C.C.S.

Here also, the levels of crushing strength are higher in case of mixes containing bauxite than in those containing grog: In the former the C.C.S. ranges from 34 to 39 MPa, while ranging from 32 to 37 MPa in the latter case.
Fig. (13): Effect of Amount of Cement Used and Mean Particle Size of Aggregate on the Cold Crushing Strength of Grog Based Mixes

Tables (7) and (8) describe the relative effect of the variation in cement content and mean particle size on the variation in CCS for both types of samples. These tables show that in both cases, the variation of cement content plays a much higher role than that of particle size in assessing variations in CCS.

Table (7): Correlation Table for CCS for Bauxite Based Mixes

<table>
<thead>
<tr>
<th>% Cement</th>
<th>Particle Size</th>
<th>CCS</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>10% cement</td>
<td>0.945608</td>
<td>−0.12905</td>
</tr>
</tbody>
</table>

Table (8): Correlation Table for CCS for Grog Based Mixes

<table>
<thead>
<tr>
<th>% Cement</th>
<th>Particle Size</th>
<th>CCS</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>10% cement</td>
<td>0.943331</td>
<td>−0.12633</td>
</tr>
</tbody>
</table>

4. CONCLUSIONS

Samples of refractory concrete cubes were prepared using from 10 to 20% cement containing 50% alumina and two types of aggregate: calcined bauxite containing about 81% alumina and gog containing 52% alumina. These were graded to yield four portions of different mean particle size.

The following results could be deduced:

- Increasing the amount of cement added lead to higher water consumption, lower water absorption and porosity and higher bulk density.
- Using coarser aggregate resulted in a reduction in water used for mixing, lower water absorption and porosity and a higher bulk density.
- The effect of variation in cement content on the aforementioned properties is generally higher than that of variation of particle size.
- A higher cement content favored higher cold crushing strength but a maximum value was obtained at a mean aggregate particle size of about 0.95 mm.
corresponding to a state of maximum compactness.

- Better results were generally obtained on using bauxite aggregate rather than grog presumably due to their lower intrinsic porosity.

REFERENCES


