Effect of the Type of Aggregate on the Properties of Alumina 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 some aggregates 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. The cast samples were left in their moulds for 24 hours in a 100% relative humidity cabinet. The de–molded specimens were left in an open air until their moisture content reaches 3–6%, then kept 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.
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Key Words: Refractory concrete – Alumina – Grog – Sodium citrate – Bauxite

1. Introduction:

The analysis of the composition, the structure, and the properties of 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 or aggregates) 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 minimum possible extent or to produce them without introducing any conventional 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. However 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 to 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 ($Al_2O_3 . H_2O$), corundum (Al_2O_3), magnesia (MgO), zirconia (ZrO_2), etc…

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

2. Experimental

2.1 Raw Materials:

The raw materials used are:

1- Refractory cement having 50% alumina was obtained from Lafarge Cement Company.

2- Calcined bauxite was obtained from the Alexandria Company for Refractories with an alumina content exceeding 80%.
3- Grog was obtained from previously fired defective bricks that were crushed, ground and screened.

2.2 X–Ray Fluorescence Analysis (XRF):

X–ray fluorescence spectrometry (XRF) was employed for the elemental analysis of the starting materials. The analysis was run on a AXIOS, panalytical 2005, Wave length Dispersive (WD–XRF) Sequential Spectrometer.

2.3 X–Ray Diffraction (XRD):

X–Ray diffraction was used for the qualitative analysis of the phases present in the tested samples.

For X–Ray diffraction study of bauxite and grog analysis, the aliquots for bulk mineral analysis were finely ground (–200 mesh), and analyzed by a BRUKUR D8 ADVANCE COMPUTERIZED X–Ray Diffractometer apparatus with mono – chromatized Cu Kα radiation, operated at 40 kV and 40 mA.

2.4 Particle Size Distribution of Aggregate:

In order to determine the grain size distribution, the procedure described by ASTM D 422/2007[5] was used. The standard sieves method was applied using screen apertures ranging from 6.68 mm (3 mesh) down to 74 µm (200 mesh).

2.5 Preparation of Specimens:

Forty dry mixtures of different formulations for both bauxite and grog at various 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 [6]". The mixed batches were then cast into cubes of 50 mm side length using a vibrating table at a frequency of 50 Hz for 4 minutes. The cast samples were left in their moulds for 24 hours in a 100% relative humidity cabinet. The hydrated samples were then demolded. The specimens were left in an open air until their moisture content reaches 3–6%, then put in the drying oven at (110 ± 5) ºC until reached a constant weight. They were then tested for water absorption, bulk density, apparent porosity and cold crushing strength.

2.6 Apparent Porosity, Water Absorption, and Bulk Density:

These properties were determined according to the ASTM Standards C 20/2007 [7]. For each test, the average measurements of five specimens at least were considered.

The five specimens for each test were weighed for their dry weight (D). The specimens were then immersed in water and boiled for 2 h without contact with the heated bottom of the container. They were cooled down to the room temperature while still completely immersed in water. The weight (S) of each specimen was determined after boiling and while suspended in water. The saturated weight (W) was also determined.

Apparent porosity (P) was calculated from the following formula:

$$P,\% = \frac{W-D}{V} \times 100 \quad (1)$$

Water absorption (A) was calculated from the following formula:

$$A,\% = \frac{W-D}{D} \times 100 \quad (2)$$

While bulk density (ρ_B) was calculated from the following formula**:**

$$\rho_B = \frac{D}{V} \times 100 \quad (3)$$

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 = volume of specimen = W –S, (cm^3);
A = water absorption, (%);
ρ_B = bulk density, (g/cm^3).

2.7 Cold Crushing Strength:

This was carried out on three specimens representing each mix composition after curing for 28 days. 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. The strength was calculated according to BS EN Standards 993–5/2000 [8]:

$$C.C.S(\sigma_c) = \frac{W}{a} \quad (4)$$

Where:
σ_c = cold crushing strength, (MPa);
W = total maximum load at visible failure, (N);
a = average of gross area of the two faces, (mm^2).

3. Results and Discussion:

3.1 Chemical Analysis of Raw Materials:

Table (1) shows the XRF results related to the elemental chemical analysis of the employed refractory cement, bauxite and grog samples.

Table (1): Chemical Analysis of Materials Used

Constituents (wt. %)	Cement	Bauxite Sample	Grog Sample
SiO_2	5.5	9.264	26.640
TiO_2	——	1.451	3.740
Al_2O_3	52.95	81.291	51.929
$Fe_2O_3^{tot.}$	2.5	1.816	2.994
MgO	Traces	0.372	0.510
CaO	38.05	0.435	1.215
Na_2O	< 0.1%	0.066	1.418
K_2O	< 0.1%	0.174	4.901
P_2O_5	——	0.542	1.056
SO_3	——	0.020	0.875
Cr_2O_3	——	0.120	0.069
Co_3O_4	——	0.084	0.039
Ga_2O_3	——	0.023	0.014
SrO	Traces	0.16	0.106
Y_2O_3	——	0.021	0.020
ZrO_2	——	0.272	0.190
Nb_2O_5, La_2O_3 ,CeO_2, Nd_2O_3, ThO_2	——	< 0.1%	< 0.1%
WO_3	——	0.237	——
PbO	Traces	1.6	0.011
Cl	——	0.022	4.183
L.O.I	——	1.811	——
Total	**≈100**	**≈100**	**≈100**

3.2 XRD of Raw Materials:

Figures (1) and (2) show the XRD patterns of bauxite and grog, respectively. Calcined bauxite consisted exclusively of corundum (Al_2O_3) and mullite ($3Al_2O_3.2SiO_2$). This is expected from the phase equilibrium diagram: Al_2O_3 – SiO_2 for compositions containing > 80% alumina [(9)]. On the other hand, the XRD pattern of grog (Fig. 3) shows beside the expected phases of mullite and quartz, some non–equilibrium phases of corundum and cristobalite. Halite is also present as an impurity.

Grog and Bauxite were screened to the required size fractions using standard sieves ranging from 3 mesh (6.680 mm) down to 200 mesh (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, 1.168mm, 0.991mm, 0.295 mm, 0.175 mm, 0.147 mm, and 0.074 mm.

Figure (3) shows the cumulative screen analyses for grog and bauxite used in the present investigation.

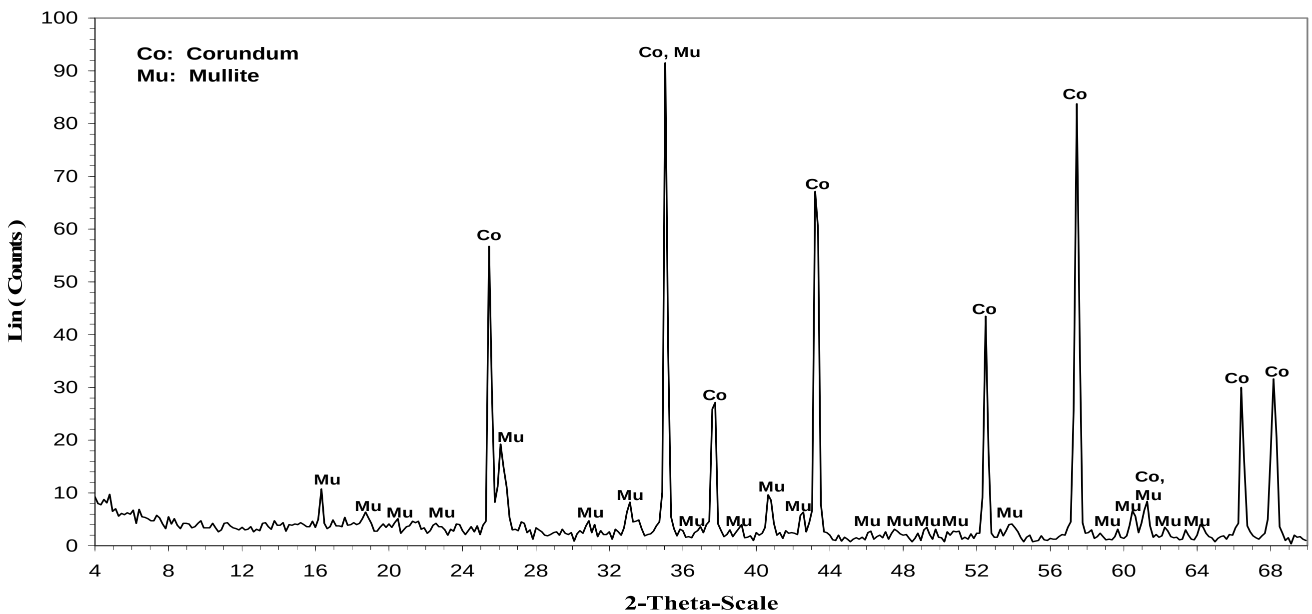

Fig. (1): XRD Pattern of Calcined Bauxite

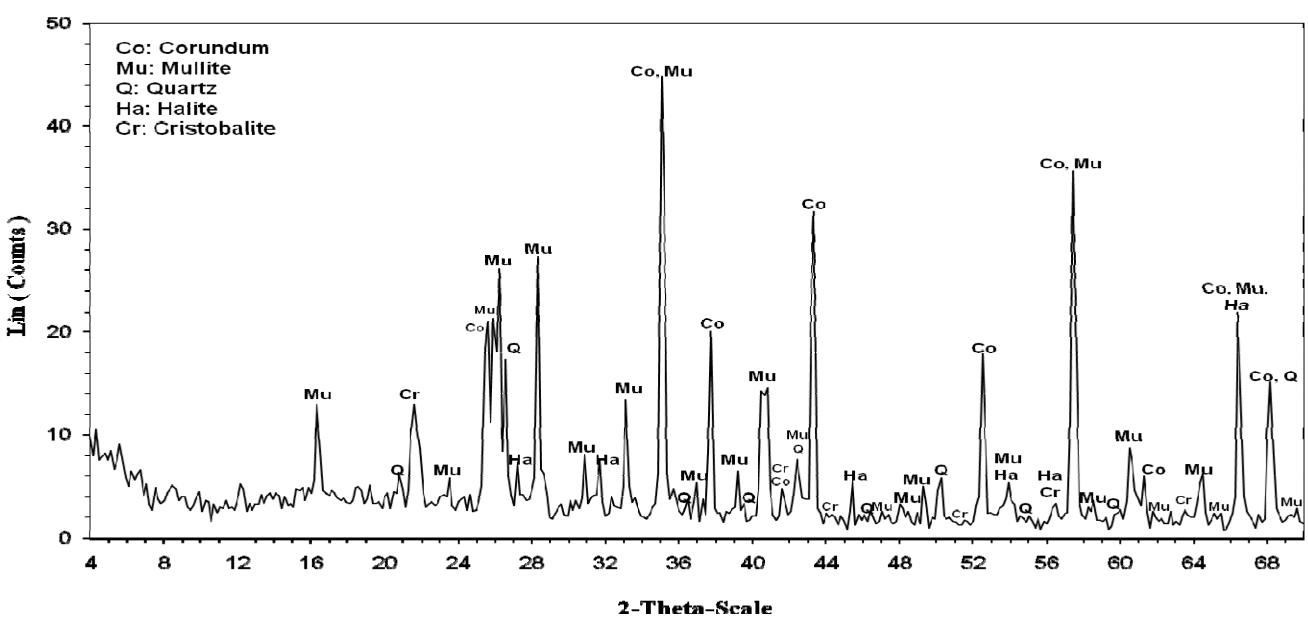

Fig. (2): XRD Pattern of Grog

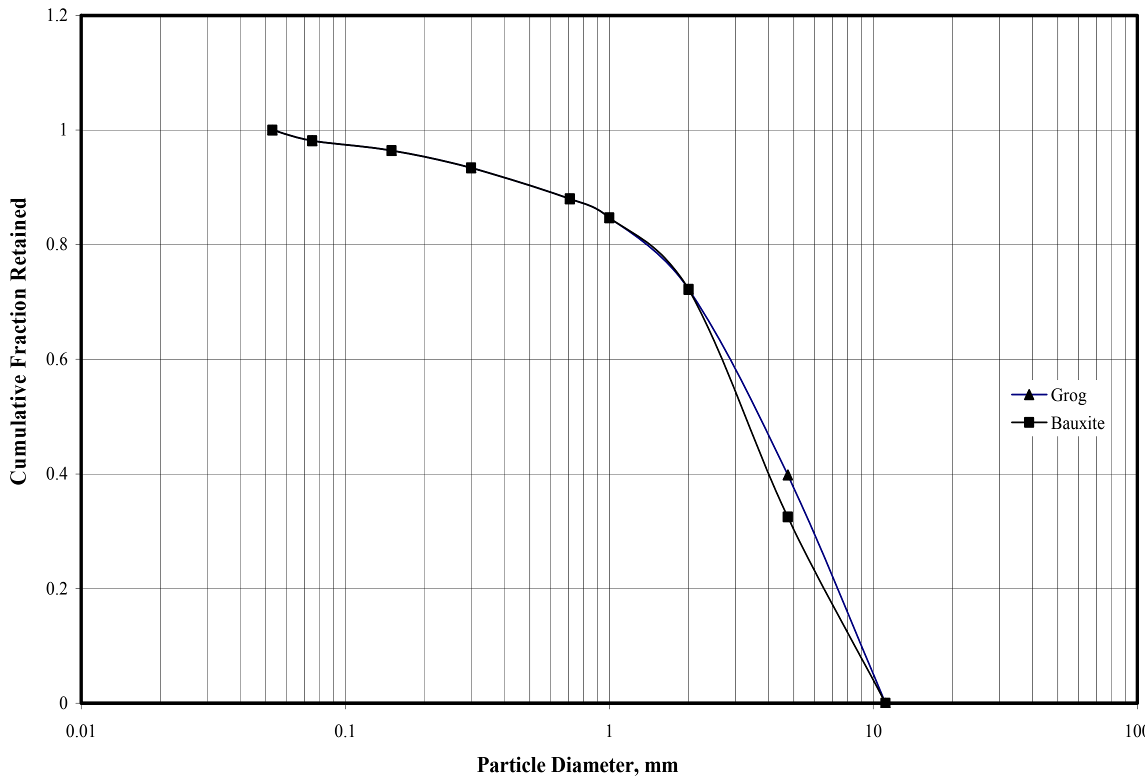

Fig. (3): Particle Size Distribution of Aggregates

3.4 Mean Particle Size of Aggregates:

In order to assess the effect of particle size of the aggregates employed (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 different particle size ranges in order to maximize compactness of samples. Table (2) shows the four mixes together with the average particle diameter of each as calculated using the method suggested by McCabe et al [10].

Table (2): Mean Particle Size of Aggregate Formulation Used

	% Weight			
	0–1 mm	**1–3 mm**	**3–5 mm**	**Mean Particle Size (mm)**
A	10	75	15	1.63
B	25	60	15	1.19
C	40	45	15	0.94
D	55	30	15	0.78

3.5 Effect of Mean Particle Size on Water Consumption:

During mixing, the ratio of water added certainly affects technological properties of the final product considerably [2]. Also, when the amount of coarse particles in the particle size distributions increases, the surface area of the particles decreases and less water is required. The amount of consumed water as function of the mean particle size of bauxite and grog are shown in figures (4) and (5), respectively.

The results show that the amount of water added increases with the decrease of both particle size in the aggregate mixture and with the amount of cement. It is also seen that water consumption for samples containing grog is higher than for those containing bauxite. These values range from 7.5 to 9.1% in case of bauxite based formulations against 9.5 to 10.9% for grog–based formulations. This is presumably due to the presence of much more open pores in grog than in bauxite particles. To assess this, the apparent porosity of samples of both types of particles was determined. And found to be 6.7% and 16.5% for bauxite and grog particles, respectively.

Using the excel DATA ANALYSIS module, it was possible to establish correlation tables in both cases to show the relative influence of both cement percent and particle size on the percent of required water. The results are given in tables (3) and (4).

The results given in tables (3) and (4) suggest 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, in the case of using bauxite, the effect of 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.

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

	% Cement	Particle Size	% Water
% Cement	1		
Particle Size	0	1	
% Water	0.642372	− 0.7489	1

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

	% Cement	Particle Size	% Water
% Cement	1		
Particle Size	0	1	
% Water	0.696767	− 0.68928	1

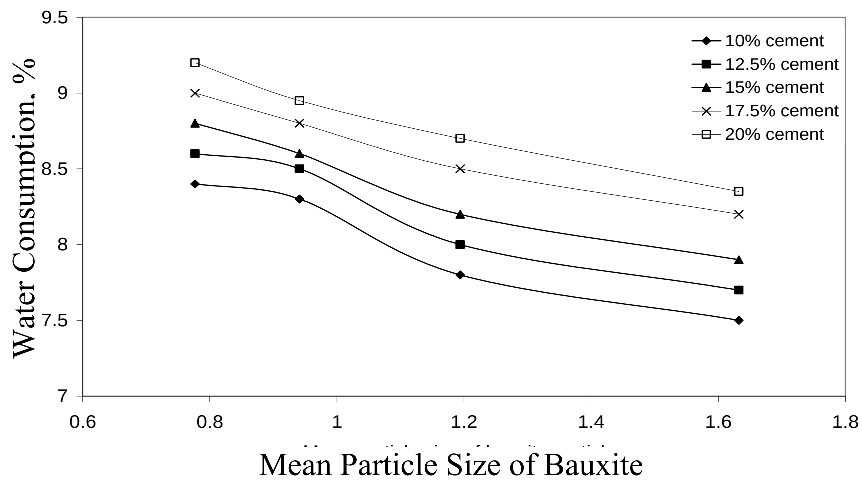

Fig. (4): Effect of Mean Particle Size of Bauxite Aggregates Containing Different Cement Percents on Water Consumption for Mixes

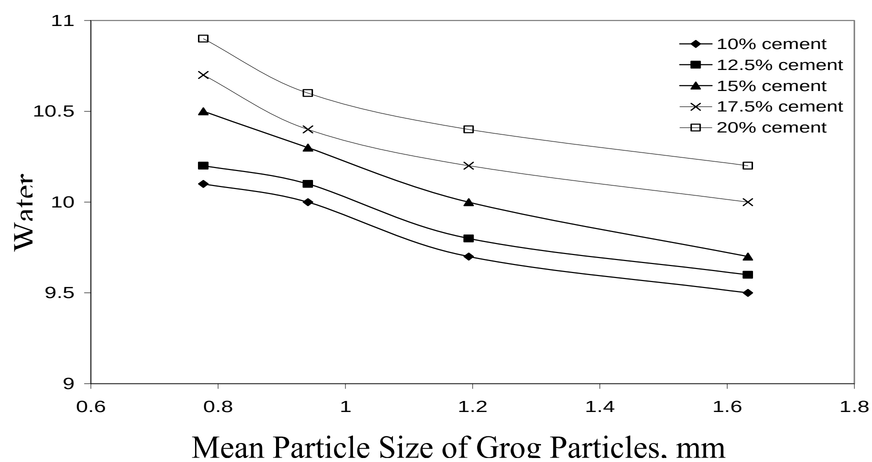

Fig. (5): Effect of Mean Particle Size of Grog Aggregates Containing Different Cement Percents on Water Consumption for Mixes

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, respectively.

The results suggest that the percent water absorption appreciably decreases with the increase of cement content. However, as the cement content exceeds 15%, its effect on water absorption diminished. This is expected since higher cement content enhances the closure of available pores.

Of interest is the difference between the water absorption values observed in either cases. 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 such samples.

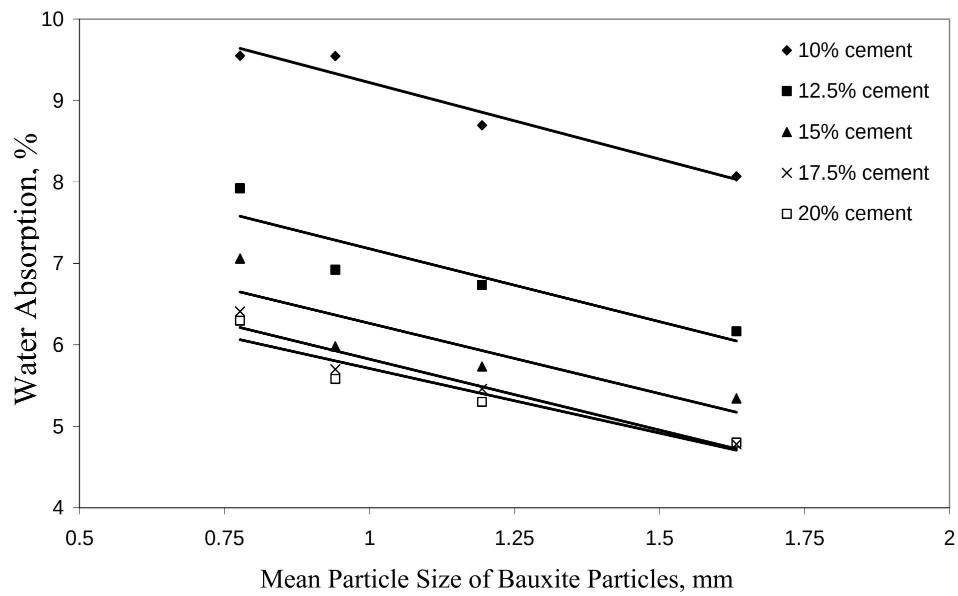

Fig. (6): Effect of Mean Particle Size of Bauxite Aggregates Containing Different Cement Percents on Percent Water Absorption for Mixes

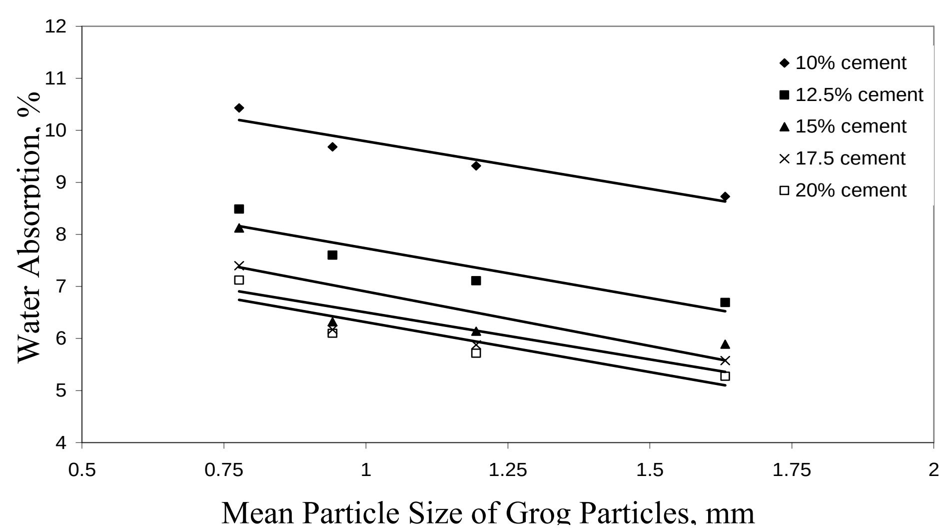

Fig. (7): Effect of Mean Particle Size of Grog Aggregates Containing Different Cement Percents on Percent Water Absorption for Mixes

Also these figures indicate that the percent water absorption is only slightly affected by the increase in particle size. To assess this, the excel DATA ANALYSIS module was used to establish correlation tables which show the relative influence of percent cement and particle size on the percent water absorption in both cases. The results are listed in tables (5) and (6).

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

	% Cement	Particle Size	% W.A.
% Cement	1		
Particle Size	0	1	
% W.A.	− 0.82455	− 0.39599	1

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

	% Cement	Particle Size	% W.A.
% Cement	1		
Particle Size	0	1	
% W.A.	− 0.79392	− 0.42302	1

Tables (5) and (6) show that both variables affect negatively the percent water absorption whereas 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:

Figures (8) and (9) show the relations between apparent porosity and the mean particle size for formulations containing bauxite and grog. It can be seen that the apparent porosity decreases with the increase of cement content. This is expected since higher cement content enhances the closure of available pores. Also these figures show that the apparent porosity is negatively affected by the increase of particle size.

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

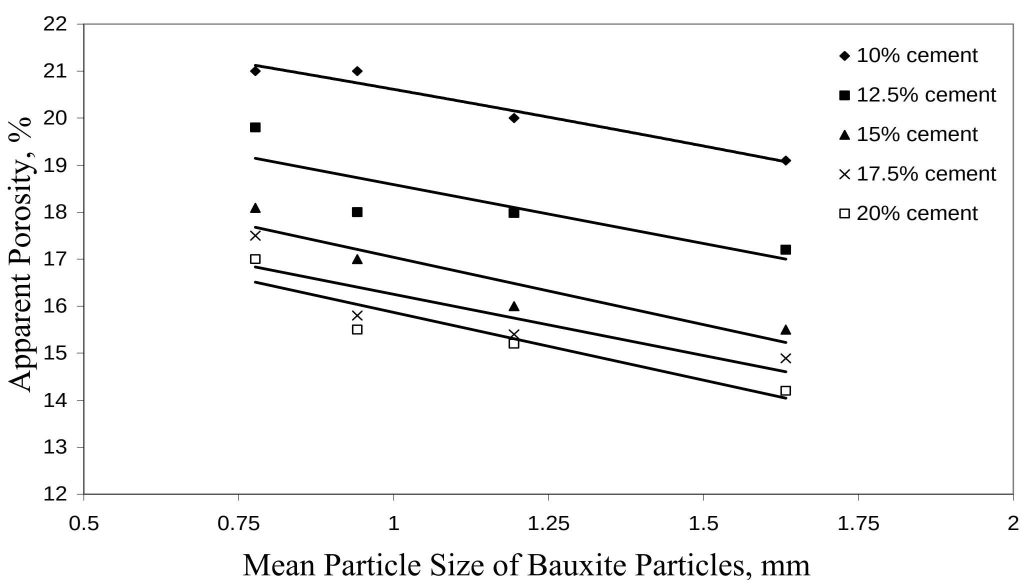

Fig. (8): Effect Mean Particle Size of Bauxite Aggregates Containing Different Cement Percents on Percent Apparent Porosity for Mixes

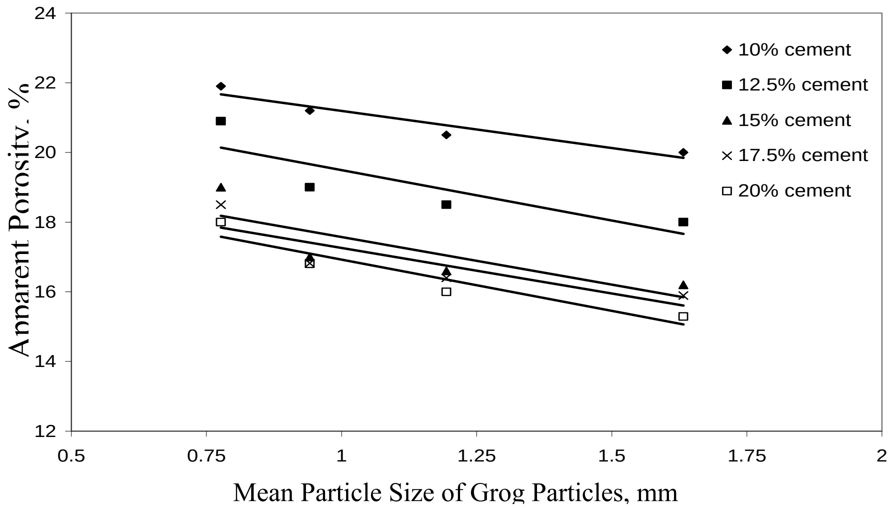

Fig. (9): Effect Mean Particle Size of Grog Aggregates Containing Different Cement Percents on Percent Apparent Porosity for Mixes

3.8 Effect of Particle Size Distribution on Bulk Density:

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

The results indicate that the bulk density increases with the increase in cement content. This is expected since higher cement contents decrease the porosity. Also these figures show that the bulk density is slightly increased with the increase of particle size.

On the other hand, for the same particle size formulation, the values of bulk density of the samples containing bauxite are higher than of those containing grog. For example, at mean particle size = 1.63 mm, the bulk density of samples containing bauxite ranged from 2.37 to 2.96 g/cm^3, depending on the amount of cement added compared with 2.3 to 2.9 g/cm^3 for grog containing samples.

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

Correlation tables were established to show the relative effect of variations in cement content and particle size for the samples containing either bauxite or grog. These tables show that both cement content and higher particle size favour higher bulk density although the effect of cement variation on bulk density is more pronounced than that of particle size.

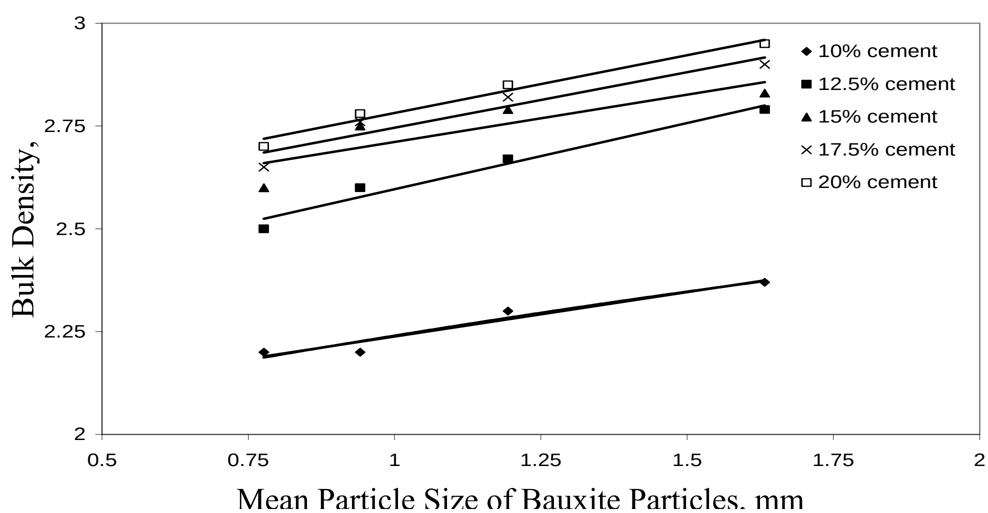

Fig. (10): Effect of Mean Particle Size of Bauxite Aggregates Containing Different Cement Percents on the Bulk Density for Mixes

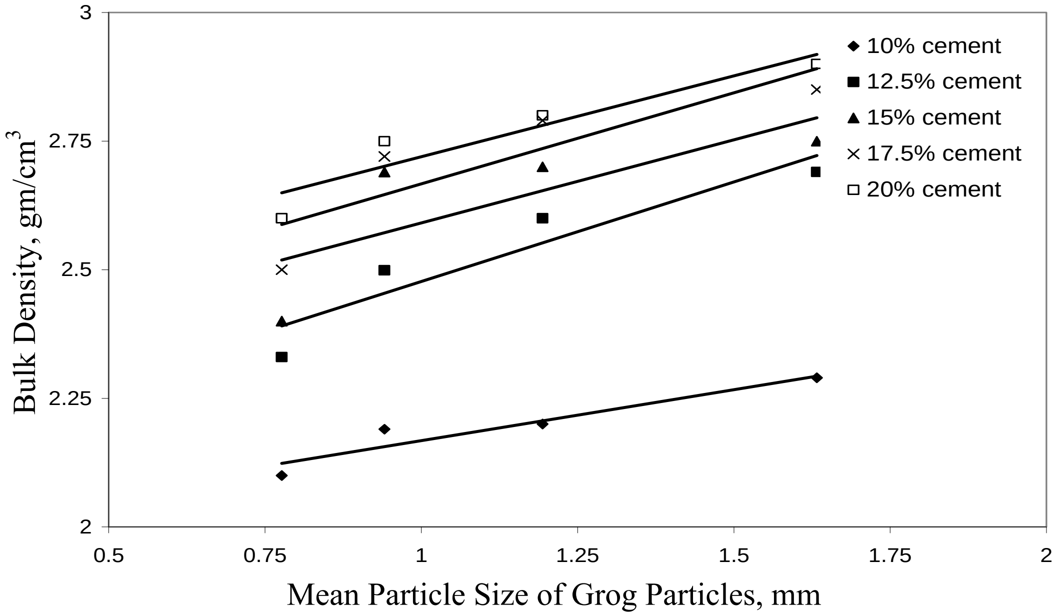

Fig. (11): Effect of Mean Particle Size of Grog Aggregates Containing Different Cement Percents on the Bulk Density for Mixes

3.9 Effect of Particle Size Distribution on Cold Crushing Strength:

Figures (12) and (13) show the relations between the 28 days – cold crushing strength and the mean particle used for different cement contents (bauxite and grog).

It could be seen that the compressive strength increases with the increase in cement content. The effect of mean particle size is more complicated. All curves seem to follow the same pattern: First, the cold crushing strength increases up to a mean particle size of about 0.95 mm then degreases 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 a minimum total porosity [11] and maximum compaction of the mix leading to a maximum value in C.C.S.

The values of crushing strength were higher for the mixes containing bauxite than for those containing grog: In the former the C.C.S. ranged between 34 and 39 MPa, compared with 32 to 37 MPa in the latter case.

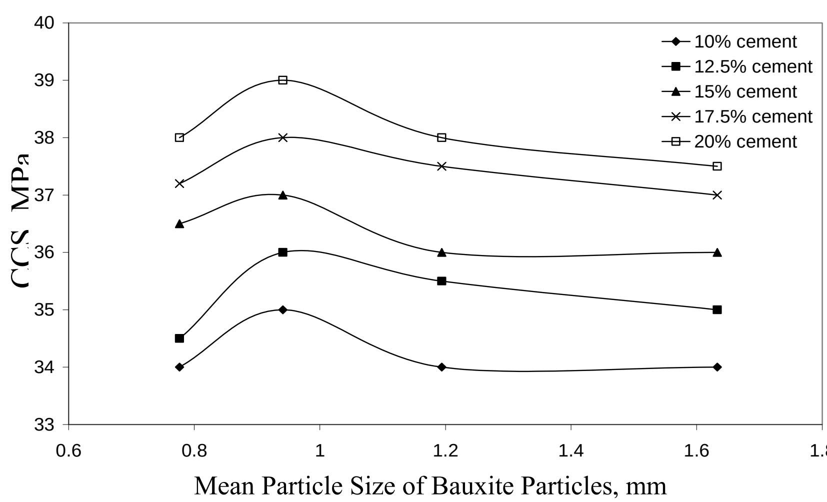

Fig. (12): Effect of Mean Particle Size of Bauxite Aggregates Containing Different Cement Percents on the Cold Crushing Strength of Mixes

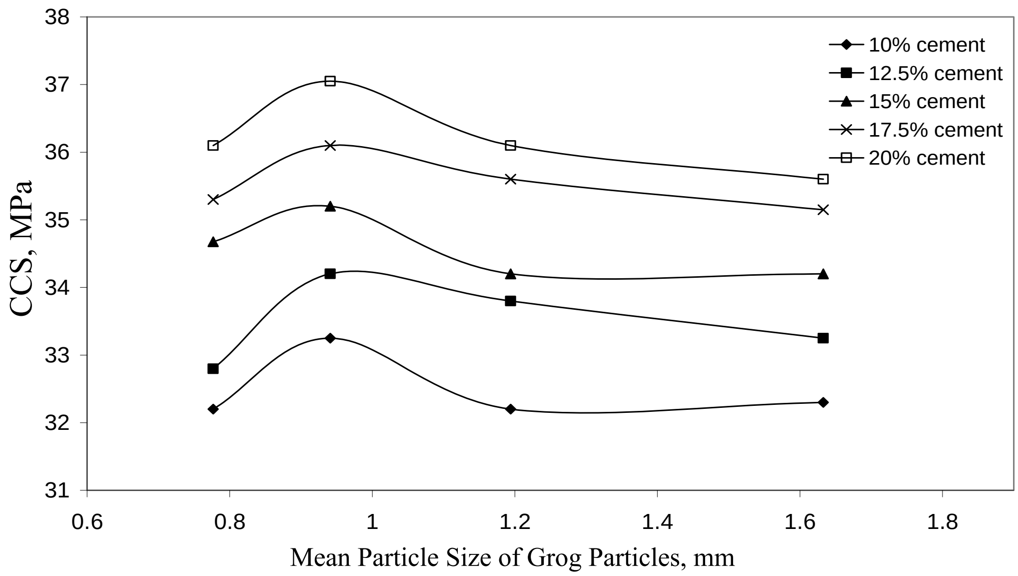

Fig. (13): Effect of Mean Particle Size of Grog Aggregates Containing Different Cement Percents on the Cold Crushing Strength of Mixes

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

	% Cement	Particle Size	CCS
% Cement	1		
Particle Size	0	1	
CCS	0.945608	− 0.12905	1

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

	% Cement	Particle Size	CCS
% Cement	1		
Particle Size	0	1	
CCS	0.943331	− 0.12633	1

4. Conclusions:

Samples of refractory concrete wereprepared using between 10 to 20% of cement containing 50% alumina and two types of aggregates: calcined bauxite (~ 81% Al_2O_3) and grog 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 / solid ratio, lower water absorption lower porosity and higher bulk density.
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- Using coarser aggregate resulted in a reduction in water used for mixing, lower water absorption, lower 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 and 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.

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