

Composition design and characterization of aluminous-cement-based concrete with recycled aggregates from waste refractory bricks.

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ABSTRACT/RESUME

Abstract: This study examined the effect of elevated temperatures on the properties of refractory concrete made with recycled aggregates from wastes of alumina-silicate bricks and calcium aluminate cement as a binder. Mixture proportions were determined according to the Dreux-Gorisse design method for micro-concretes. The evolution of the concrete rheological behavior from quasi-brittle, between room and 900 °C, to viscous at higher temperatures was shown by compression tests performed from room temperature to 1200 °C on cubic (40 × 40 × 40 mm³) specimens. Physical characteristics and compressive strength of hardened samples are also investigated at the room temperature after being fired at various temperatures. Investigated physical properties included bulk density and porosity. The results indicate that the density as well as the compressive strength decrease in the temperature range of 25 °C. to 1200 °C. By contrast, porosity is inversely proportional to temperature and density.

I. Introduction

The refractory products manufacturing process generates a lot of products deemed unsuitable for sale, with negative effects on the environment. At the same time, raw materials for refractory concretes are becoming increasingly rare and costly, coming up with new substitute materials is vital.

The reuse of the refractory products manufacture scrap and the recycling of used refractory bricks seems to be a reliable alternative. Such a solution has dual benefits: economic and ecological. On one side it allows the use of available materials without resorting to rare raw materials such as bauxite and andalusite, or costly synthetic materials such as mulite or corundum. On the other, the recycling of scrap and used refractory bricks reduces their negative impact on the environment.

Recent studies have been conducted on the reuse of brick waste in concrete. Pašalić and al. studied the

use of brick waste in the mortar repair used for the restoration of old buildings. [1]

Also, Saidi and al. studied the reuse of refractory ceramic waste as a fine aggregate by partial substitution of sand to produce a heat-resistant mortar. [2]

More recently, a number of studies have been carried out in this field leading to the conclusion that refractory ceramic waste can be used as a mineral additive or aggregate in mortars and concretes [3-6].

According to this short bibliography on the using of refractory brick wastes on cement mortars and concretes, it is interesting to investigate the possibility of reusing wastes of alumina-silicate refractory bricks as fine aggregates in aluminous-cement-based concretes and see the behavior of such material through the influence of elevated temperatures, especially as the refractory industry in Algeria generate a lot of waste.

Concrete mixture is then developed using the Dreux-Gorisse concrete design method [7].

After 28 curing days, hardened concrete cubic samples are fired respectively at 150 °C, 350 °C, 700 °C, 900 °C, 1000 °C and 1200 °C for two hours.

For each firing temperature, the physical characteristics (open porosity and bulk density) and compressive strength are investigated at room temperature.

II. Materials and methods

II.1. Materials

The materials used in this study are aluminous cement acting as a binder, ground silico-alumina bricks as aggregate and a polycarboxylate based superplasticizer.

Aluminous cement is produced by the Spanish company “Cements Molins Industrial”. Its chemical composition is given on table 1 and the main mineralogical component is CaAl₂O₄ [8].

It is a quick-setting cement, as can be seen on table 2 which giving other additional physical characteristics.

Table 1 . Chemical composition of cement [8]

Component	(%)
Al ₂ O ₃	43.3
CaO	37
SiO ₂	3
Fe ₂ O ₃	12
FeO	4.5
Alcalis	0.06
SO ₃	0.1
Cl	0.01
S ²	0.03

Table 2. Some additional properties of the refractory cement [8]

Additional properties	
Setting Start (mín.)	145
Setting End (min.)	165
Bulk density (g/cm ³)	1,2
Specific gravity (g/cm ³)	3,2
Blaine specific surface (cm ² /g)	3270
Compressive strength after 6 hours (MPa)	47.7
Compressive strength after 24 hours (MPa)	65.2
Melting temperature (°C)	1360

Aggregates (fig.1) are 5 mm maximum size. They are obtained by dry crushing scraps of silico-

alumina refractory bricks collected from the Guelma Ceramic Plant in Eastern Algeria (Fig.2). The silico-aluminous aggregates here indicated were characterized before being incorporated in concrete mixture. The chemical composition of aggregate is given in table 3.

Table 3 . Chemical composition of aggregates.

Component	(%)
Al ₂ O ₃	35.57
CaO	1.85
SiO ₂	58.01
Fe ₂ O ₃	1.21
Na ₂ O	0.48
TiO ₂	0.19
K ₂ O	1.65
MgO	1.04



Fig 1. Silico-aluminous aggregate



Fig 2. Alumina silicate refractory brick

On figure 3 showing the particle size distribution of silicoaluminous aggregates according to the NF EN 933-1 standards [9], it is remarkable that silicoaluminous aggregates consist mainly of fine grains.

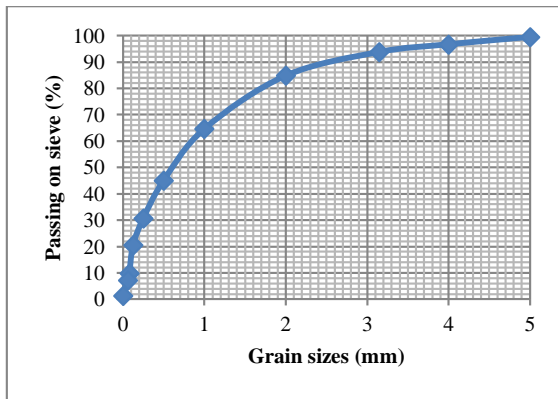


Fig 3. Particle size distribution of alumina silicate aggregate

Physical characteristics of aggregates used are measured according to the NF EN 1097-3 standards [10] and reported on table 4.

Table 4. Physical properties of aggregates

Absolute density (g/cm^3)	1.50
Bulk (g/cm^3)	3.12

II.2. Mixture Design and Mixing Protocol

To succeed aluminous cement based concrete, reference is made to the recommendations of the EN 14647 standard "Calcium aluminate cement - composition, specifications and conformity criteria" [11].

It is recommended to use a cement dosage not exceeding 400 kg per cubic meter of concrete. The water/cement ratio should be less than or equal to 0.4 (total water includes aggregate moisture). Aggregates must be clean, free of organic matter, clay, releasable alkaline elements, granite, schistous or micaceous elements.

Concrete mixture was established using the Dreux Gorrise design method for micro-concretes [12, 13].

Since refractory concretes based on aluminous cement often have a firm consistency [14], a superplasticizer admixture is added. It is presented in the form of yellowish liquid and is designed on the basis of polycarboxylates, called "MEDAFLOW 30". Its absolute density is 1.07 and its concentration in solid particles is 30 %. It is incorporated into the mixing water with a dosage of 1% of total weight of cement determined using the fluidity test by mini-cone according to the NF EN 12350-5 standard [15].

Table 5 shows the mix details concrete. The water/binder (aluminous cement) ratio used was kept constant ($W/B = 0.40$) for all samples.

Table 5. Concrete dosage according to the Dreux Gorrise design method

Water /Cement ratio	0.4
Superplasticizer (kg/m^3)	4
Cement (kg)	400
Water (l)	160
Aggregates (0 - 3,15mm) (kg/m^3)	492
Aggregates (3,15 - 5mm) (kg/m^3)	492
Slump (mm)	65

II.3 Samples preparation and conditioning

Concrete mixture was done in the laboratory with an ambient temperature of 23 ± 2 ° C. Mixing was performed using a low capacity laboratory mixer (Fig.4). Aggregates and the cement are weighed and placed respectively in the concrete mixer and then dry mixed for 1 min. The mixing is then continued for 4 to 5 minutes by gradually adding the mixing water (together with the superplasticizer).



Figure 4. Laboratory mixer

Cubic ($40 \times 40 \times 40 \text{ mm}^3$) samples were manufactured and kept in water at $22 - 25$ ° C to ensure good setting of the cement for 24 hours. Thereafter, they are dried for 48 hours at 110 ° C in a drying chamber (fig.5) and then kept at room temperature until the time of testing.



Figure 5. Drying chamber

To get a better understanding of the temperature effect on physical and mechanical of the concrete properties, tests are carried out on unheated hardened samples on the one hand, and on prefired ones on the other. Samples are so fired for two hours at desired temperature in a lab furnace (fig.6) and then tested at room temperature. The considered firing temperatures are 150, 350, 700, 900 and 1200 °C respectively.



Figure 6 . Experimental lab furnace

Heating and cooling rates are respectively 50 °C/h and 90 °C/h. For temperatures over than 350 °C, the temperature is kept constant for 2 h at 150 °C (evacuation of the free water) and 350 °C (evacuation of the combined water).

Figure 7 shows the firing curve of concrete samples at 900 °C.

To investigate the concrete thermo-mechanical behavior, compression tests are undertaken at desired temperatures on 28 days aged samples in a high-temperature mechanical equipment.

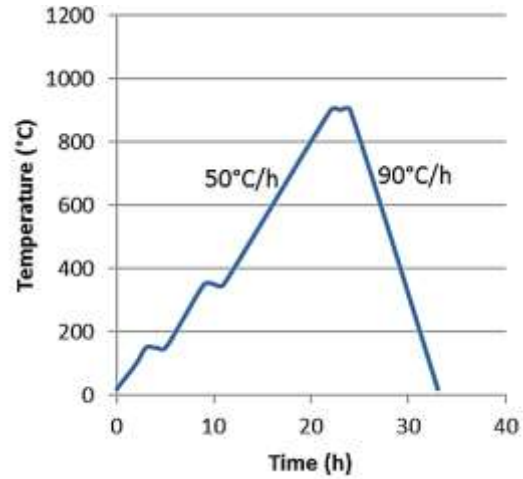


Figure 7. The firing curve of concrete samples at 900 °C

II.3. Test methods

II.3.1.Characterization at room temperature

Both physical and mechanical tests at room temperature were carried out on hardened unheated and pre-fired concrete cubic samples (40 x 40 x 40 mm³) (Figure 8). The retained values are the average of 5 experimental results per test.



Figure 8. Cubic specimens

Bulk density and open porosity are measured according to the ISO 1927-7:2012 standards [16].

Compression tests are carried out using a compressive test machine (figures 9a, 9b) according to the ISO 1927-8:2012 method [17].

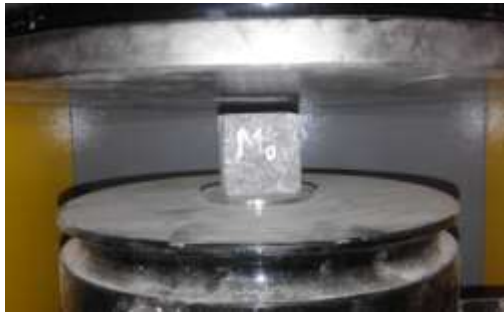


Figure 9a. Compression test on fired concrete cubic specimen at room temperature



Figure 9b. Brittle failure of fired concrete cubic specimen at room temperature

II.3.2. Refractoriness

Refractoriness was evaluated according to the DIN 51 730 standard [18] by measuring the melting point using macroscopic examination during heating.

II.3.3 Thermo-mechanical tests

Modulus of rupture mechanical testing was undertaken on high-temperature mechanical equipment. This set-up consisted of a ZWICK Z400E electromechanical testing machine equipped with a PYROX 1600 °C electrical furnace and an exterior differential displacement measuring device. This set-up could be configured to perform uniaxial compression or three-point bending tests at various temperatures. Compression tests were carried out on cubic specimens. The specimens were calibrated to an initial height of 40 mm. Figure 10 presents a thermo mechanical test progress. It consists of four phases: heating, the testing temperature maintenance phase, the compression test stage and the cooling phase.

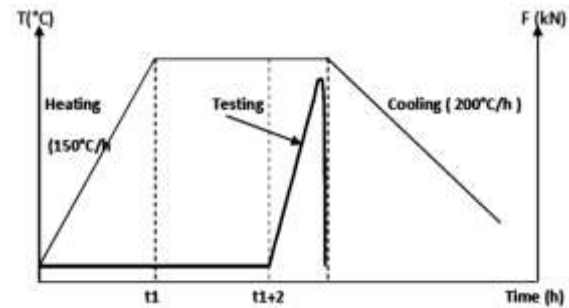


Figure 10. Typical evolution of temperature and load in a thermo mechanical test.

The uniaxial compression tests were carried out at ambient temperature, 500 °C, 700 °C, 900 °C, 1000 °C and 1200 °C. The loading procedure consisted of applying a preliminary load (2 KN in compression at a rate of 0.5 mm/min to eliminate positioning defects of the specimens. For tests conducted at elevated temperatures, a standard thermal cycle was applied: samples were heated from room temperature to the target test temperature at the rate of 200 °C/h, followed by a 2 h hold at the test temperature before the mechanical loading was applied in isothermal conditions.

After testing, samples were cooled to room temperature at a cooling rate of 150 °C/h. The mechanical loading consisted of imposing a 0.1 mm/min displacement rate to the upper plunger. The two LVDT sensors of the differential displacement devices recorded the specimen's height variation in compression tests. It then became possible to display force–displacement curves generated from each test. The stresses and strains in compression tests were determined by the equations (1) and (2) respectively:

$$\sigma = \frac{F}{a^2} \quad (1)$$

$$\varepsilon = \frac{i}{a} \quad (2)$$

Where F is the actual measured load, a is the specimen side length and i is the average of the sensor indications.

III. Results and discussion

III.1. Characterization at room temperature

- Physical properties

The physical and mechanical properties of concretes are intimately linked to the behavior of the cementitious matrix.

The cementitious matrix is the preferred place for conversion phenomena, because it is composed of the most reactive constituents [19]. Thus, during the first heating, the rise in temperature leads to the unfolding of the conversion phenomena.

If the aggregates remain relatively chemically inert to the effect of temperature, the alumina cement matrix undergoes changes with the phase reactions occurring at certain temperature peaks and the dehydration / fusion / crystallization phenomena which accompany these transformations [20, 21]. The thermal decomposition of hydrates has been the subject of numerous works which are summarized by B. Myhre [22], J. Soro [23] and N. Gimet [24]. It appears that it depends, among other things, on the nature of the material (Hydrate formed from pure CA or cement), the physical characteristics of the samples (powder, mass, porosity), as well as experimental parameters (heating rate, pressure).

The mechanism of dehydration which can be measured by differential thermal analysis and thermogravimetry is quite complex and several studies have focused on the study of this process [25, 26]. CAH10 begins to lose some of its water of crystallization at very low temperatures compared to those indicated by thermal analysis (50 °C - 70 °C).

The C3AH6, in turn, shows partial dehydration, indicated in the ATD curves by two endothermic peaks, the first at around 300 °C. and the second near 500 °C.

The alumina gel AH3 and the gibbsite generally dehydrate between 210 °C and 300 °C but they can also undergo a conversion to bohemite (AH) which dehydrates at between 530 °C and 550 °C.

All components formed during the hydration of the cement are completely dehydrated above 550 °C. At 900 °C., the AC recrystallizes.

Between 1000 °C. and 1100 °C., the calcium monoaluminate (CA) reacts with the alumina to give the calcium dialuminate (CA 2). At 1200 °C, the crystals of CA2 become larger and more globular.

Figure 11 displays evolution of the open porosity of the concrete as a function of temperature. The phenomena of dehydration of the cement due to the increase in temperature are responsible for the increase of the open porosity.

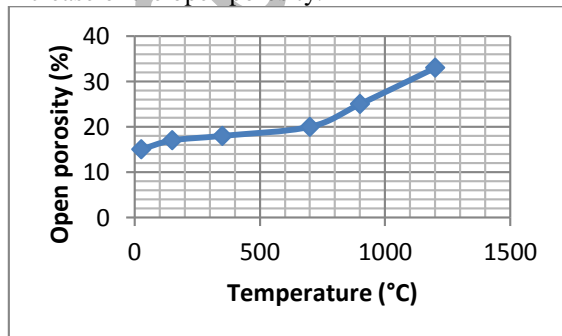


Figure 11. Open porosity of specimens as a function of the firing temperature

Mixtures containing a high proportion of cement have a greater increase in open porosity between 110 and 450 °C. This is particularly the case for Silico-alumina concretes.

On the other hand, the density decreases in the temperature range of 25 °C. to 1200 °C (fig.12). This is due to the process of massive dehydration of the cement matrix, mainly to the destruction of the network of cement hydrates.

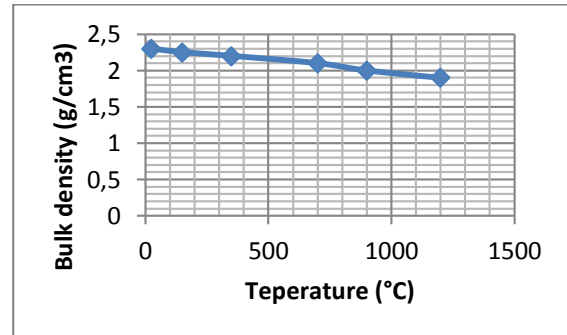


Figure 12. Bulk density of specimens as a function of the firing temperature

At 25 °C., the samples have a high density (with a fairly low porosity). The bulk density of the cured concrete samples begins to fall with the temperature rise. This fall is due, on the one hand, to the release of the mixing water, leading to an increase in the porosity and, on the other hand, to the microcracks appearing in the cementitious matrix. In addition, cracks are generated by matrix / aggregate decohesion due to the difference in their dilatometric behavior.

The same observations were made by Edwinge Y. F. [27] on an andalusite-based concrete which had undergone a heat treatment at 900 °C.

Between the temperatures of 600 °C - 1200 °C, the bulk density undergoes practically no significant change.

Overall, the values obtained for the open porosity and the density after heat treatment at various firing temperatures are very close to those obtained by F. Simonin [28] for aluminous industrial refractory concretes containing magnesium spinel using the Andreasen formulation method for refractory concretes [29].

- Mechanical properties

Figure 13 shows the evolution of the compressive strength, as a function of firing temperature. The breaking stress is relatively high at 20 °C. and then falls with increasing temperature up to 1200 °C.

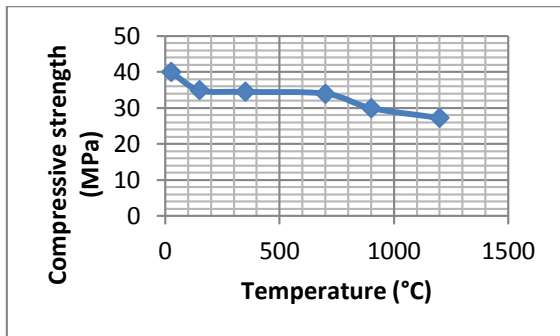


Figure 13. Compressive strength of specimens as a function of the firing temperature

The cement dehydration phenomena due to the increase in temperature are responsible for the increase in open porosity, which influences the evolution of mechanical properties. However, they do not explain alone the strong degradation of the mechanical strength of concrete.

The decrease in mechanical properties occurs at a temperature where the matrix undergoes shrinkage while the aggregates expand. These two contrary effects between the matrix and the aggregates are responsible for microcracking in concrete, as was recently observed on a similar material. Blundell [30] and Roux [31] also noticed that the differential expansion between aggregates and cement paste could lead to degradation of the interface by microcracking.

The gap between the expansions creates deformations probably sufficient to damage the material because they lead to a higher stress than the cohesion stress at the matrix - aggregate interface.

Microcracking of the interface is further accentuated when using aggregates with high thermal expansion coefficient.

The various components of the material also have very different elastic moduli. During cooling, this difference in elastic modulus generates local stresses which can also contribute to the microcracking of the interface: it is the "composite" effect.

The compressive strength of the prefired concretes varies between 30 MPa when heattreated at 1200°C and 40 MPa, for concretes without heattreatment. Very often, at very high temperatures (> 1000 °C), the process of ceramization of the concrete commonly called sintering leads to a densification of the material which results in an increase in its breaking strength. Here, it can be assumed that the expansive formations of CA2 between 1100 °C. and 1200 °C., accompanied by an increase in porosity, has thwarted the sintering process. This is why no improvement is visible for the mechanical and

physical properties of the concretes studied up to 1200 °C.

Overall, the increase in the firing temperature up to 1200 °C. is detrimental to the strength of concretes.

III.2 Thermo-mechanical behavior

Fig. 14 plots the stress/strain curves of compression tests carried out at various temperatures on unheated specimens.

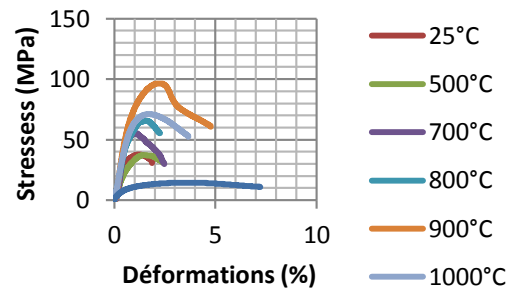


Figure 14. Stress–strain uniaxial compression diagrams for studied concrete samples at different testing temperatures: 25, 500, 700, 900, 1000, 1200 °C

The curves have bell-shape forms characteristic of quasi-brittle materials. During testing, materials endure a maximal stress (resistance), and then lose those stresses because of high temperature material softening during heating. The strain reached when the stress is a maximum (strain at peak stress) indicates the ability of a material to deform before collapsing, thus corresponding without any doubt, to ductility [32].

The maximal stress increases with increasing temperature up to a maximum value at 900 °C, then decreases at higher temperatures.

The compressive strength values obtained in this study seem to be low, compared with those obtained by Mohsen Roosefid for a commercial silica-aluminous refractory concretes [33] tested in the same conditions, but similar to values obtained by Prompt [34] for an aluminous refractory concrete.

Figure 15 shows the evolutions of the compression strength measured on samples fired at different temperatures and tested either at room temperature or at elevated temperatures (testing temperatures).

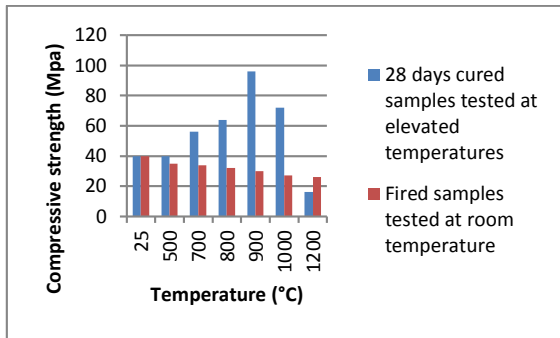


Figure 15. Compression strength of prefired samples tested at room temperature and samples tested at elevated temperatures.

III.2 Refractoriness

Refractoriness of a material is dependent on microstructural parameters such as grain size and shape, the viscosity of the vitreous phase, and sample porosity [35, 36]. Refractoriness can be evaluated by examining the evolution of the morphology of test samples with increasing temperature under their own weight (without any other load). Refractoriness in this study is defined as the temperature corresponding to the moment when a material begins to lose its shape (melting point). As can be seen from the photos in figure 16, the samples tested here maintained their shape without undergoing any deformation beyond 1250 °C.

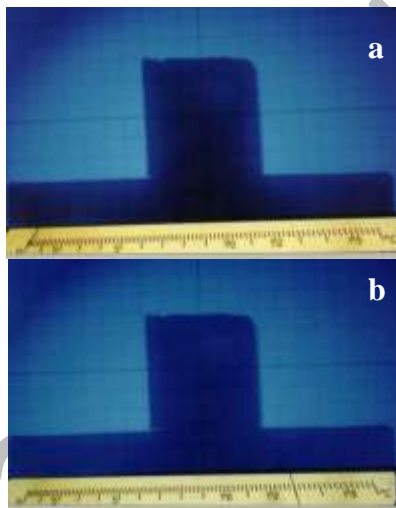


Figure 16. Refractoriness (deformation) of samples at: (a) 20 °C and (b) 1280 °C.

IV. Conclusion

The conclusions drawn from the results obtained in this study are as follows:

- The reuse of recycled refractory aggregates makes it possible to obtain refractory concretes with physical and mechanical characteristics similar to

concretes using natural or synthetic refractory aggregates.

- The Dreux-Gorriese design method for micro-concretes gives satisfying results in terms of density and resistance when used for the mix design of refractory concretes.
- Silico-aluminate refractory concretes based on recycled silico-alumina aggregates can be used at temperatures exceeding 1250 °C., as shown by the refractoriness tests.
- The chemical reactions, which take place predominantly in the binder phase (dehydration of the cement, formations of the CA, CA2, CA6 and spinel phases), induce microstructural transformations that affect the thermomechanical properties of the material during its first rise in temperature. It should be particularly attentive to some constitutive factors (choice of constituents and their weightings in concrete) or environmental (choice of temperature increase speed), if it is desired to optimize the performance of the refractory concrete.

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