



# Thermoelastic properties evolution and damping phenomena of Cameroonian calcined bauxite stabilized with calcium dialuminate refractory cement

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## Abstract

Two castable refractory concretes were made respectively from samples of high alumina calcined bauxite and calcium dialuminate ( $\text{CaO} \cdot 2\text{Al}_2\text{O}_3$ ) refractory cement. Each castable refractory concrete was made up of 8% wt refractory cement and  $w/c=0.40$  and then stabilized at 105 °C for 48 h. The refractory concretes were characterized by determination of Young's modulus according to infinite mode, dilatometric behavior, apparent density and apparent porosity. Measurement of Resonance Frequency and Damping Analysis (RFDA) to determine the Young's modulus and damping phenomena up to 1620 °C were done as well. Young's modulus of castable refractory concrete which was initially 28.3 GPa and apparent porosity of 38.2% at 105 °C led respectively to 90.4 GPa and 24.2% after treatment at 1500 °C. The study of Young's modulus versus temperature showed significant decrease around 300 °C (dehydration of cement) followed by an increase between 800 and 1620 °C (sintering of concrete). Important damping phenomena were noticeable respectively around 880 and 1280 °C which resulted in certain imperfections in castable refractory concrete, expressed as crystallization of calcium aluminate (CA) and calcium dialuminate ( $\text{CA}_2$ ). Young's modulus of 9 GPa at 1620 °C showed that elaborated refractory concrete could be suitable for the construction of kiln in steel or ceramic industries.

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## 1. Introduction

Refractory concretes are increasingly been used in steel industry and ceramic applications. Actually, their high temperature behavior (1500–1600 °C) is the object of numerous studies. Refractory concrete brings flexibility of use, as time and energy saving. In certain cases concrete made with cal-

cium dialuminate as binder also develops properties superior to those sintered using conventional calcium aluminate cement refractory. On the other hand, their behavior in intermediate temperatures (between dehydration and sintering) is not well known [1–3].

The heterogeneity of castable refractory concrete results from multi-phase compositions involving aggregates of different sizes and binding phases (cement and various additives). Grain arrangement, shape of aggregates and microstructure of binding phases have an impact on initial microstructure and thermo-mechanical properties [1–3]. Hence, the presence of calcium dialuminate as binder can offer better properties at high temperature due to its refractoriness and thermo-mechanical

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behavior [1,3]. Elsewhere, important number of studies have been made on the reactivity of calcium dialuminate used as binder [2–9].

Up to now, few studies have been done on both temperature dependence of Young's modulus and thermal properties of calcium dialuminate cement [2–6]. However, because of their coarse grains, heterogeneous and multiscaled structure, refractory concretes behavior exhibit a hysteresis loop shape for the study of variation of Young's modulus versus temperature.

The determination of dynamic elastic properties from ambient to high temperature of refractory concrete can therefore be computed if the geometry, the mass and the mechanical resonance frequencies of suitable material test specimen can be determined. Dynamic Young's modulus can be determined by using resonance frequency in flexural mode vibration. Elsewhere, ultrasonic resonance spectroscopy is used to inspect the quality of a wide range of industrial components [10–12]. One of the advantages of pulse-echo examination technique is the ability to inspect the whole component using one test. Virtually all other inspection methods require scanning of the surface which is relatively slow and expensive [10–12].

The aim of this work is to study the evolution of thermo-elastic properties and damping phenomena of Cameroonian refractory bauxite sample stabilized with calcium dialuminate cement. Characteristics such as bulk density, porosity and linear shrinkage were also determined. Young's modulus achieved by damping or resonance frequency measurements were also applied to these heterogeneous materials. Measurement of damping phenomenon on refractory concrete in regard to sensitiveness was a focus, to envisage the mechanism which affects thermo-mechanical properties of castable refractory material.

## 2. Materials and experimental procedures

### 2.1. Materials

#### 2.1.1. Calcined bauxite granules

Two bauxite samples collected at the deposit of Haleo-Danielle, Minim-Martap (Adamaoua Region of Cameroon) denoted respectively as BX55 and BX8 were used in this study. The chemical composition of the bauxite samples and lime are given in Table 1 and data show that bauxite samples were of high alumina content. A solution of 4% Polyethylene Glycol (PEG) was used as binder and mixed with bauxite powder samples ( $D_{97}=200\ \mu\text{m}$ ) for preparation of pressed pastes which were fired at  $1550\ ^\circ\text{C}$  in an electric muffle furnace for 2 h. The sintered products were then ground

Table 1  
Chemical composition of raw materials.

| Sample      | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | CaO          | MgO         | Na <sub>2</sub> O | K <sub>2</sub> O | Fe <sub>2</sub> O <sub>3</sub> | TiO <sub>2</sub> | Total        |
|-------------|------------------|--------------------------------|--------------|-------------|-------------------|------------------|--------------------------------|------------------|--------------|
| <b>BX5</b>  | Nd               | 89.40                          | 0.018        | Nd          | Nd                | Nd               | 1.83                           | 1.40             | <b>92.60</b> |
| <b>BX8</b>  | 0.65             | 91.10                          | 0.00         | 0.01        | 0.000             | 0.050            | 2.14                           | 1.13             | <b>95.08</b> |
| <b>Lime</b> | Nd               | Nd                             | <b>95.58</b> | <b>1.30</b> | Nd                | Nd               | Nd                             | Nd               | <b>97.10</b> |

to  $D_{97}=2.5\ \text{mm}$  and separated in different fraction sizes ( $\Phi \leq 100\ \mu\text{m}$ ;  $100\text{--}250\ \mu\text{m}$ ;  $250\text{--}400\ \mu\text{m}$ ;  $400\text{--}630\ \mu\text{m}$ ;  $630\text{--}1250\ \mu\text{m}$ ;  $1250\text{--}2000\ \mu\text{m}$ ;  $2000\text{--}2500\ \mu\text{m}$ ) [13–16].

#### 2.1.2. Calcium dialuminate cement

The bauxite powdered samples of BX55 and BX8 ( $D_{97}=100\ \mu\text{m}$ ) with high gibbsite content [16] were mixed with lime powder ( $D_{97}=100\ \mu\text{m}$ ) as follows: 72% Al<sub>2</sub>O<sub>3</sub>, 28% CaO and water. The obtained mixtures were labeled as CBX55 and CBX8. The mixtures were molded, dried in an oven at  $105\ ^\circ\text{C}$  for 24 h and heated at  $1550\ ^\circ\text{C}$  for 2 h in an electric muffle furnace under the ambient atmosphere of the laboratory with a heating ramp of  $5\ ^\circ\text{C}/\text{min}$ . The obtained products were ground and sieved ( $\Phi \leq 100\ \mu\text{m}$ ).

#### 2.1.3. Formulation of castable refractory

Calcined bauxite granulates of BX55 and BX8 of different grain sizes were assembled into two batches according to Funk and Dinger's volume cumulative formula [1] which is expressed as:

$$V(\%) = \frac{D^q - DS^q}{DI^q - DS^q} 100$$

where  $q=0.26$ ;  $DI$  and  $DS$  are respectively the dimensions of biggest and smallest granulates. Each batch was mixed with both 8% of calcium dialuminate cement and water (Table 2) according the ratio  $w/c=0.4$  using the three stapes process (1/3 of water every 1 min 30 s) in order to get a self-flowing castable. Each concrete was cast in plastic molds with respective dimensions of  $40 \times 40 \times 10\ \text{mm}^3$  and  $101.42 \times 32.40 \times 12.70\ \text{mm}^3$  to get a series of concrete samples which were vibrated on a vibrating table at a frequency of 50 Hz for 3 min. The molded and vibrated samples were then cured for 48 h in 100% relative humidity and later on demolded and cured at  $105\ ^\circ\text{C}$  with 100% relative humidity to accelerate hydration of concrete components. The resulting concrete samples were denoted as BBX5 and BBX8.

### 2.2. Experimental procedures

The concrete samples were characterized by determining the bulk density and the apparent porosity using nitrogen

Table 2  
Concrete formulation.

| Constituents                               | Mass proportion (%)  |    |
|--|--|----|
| Calcined bauxite at $1550\ ^\circ\text{C}$ | 1.25–2.5 mm  | 18 |
|  | 0.63–1.25 mm   | 15 |
|  | 0.4–0.63 mm  | 9  |
|  | 0.25–0.4 mm  | 8  |
|  | 0.1–0.25 mm  | 13 |
| Matrice                                    | Calcined bauxite at $1550\ ^\circ\text{C} \leq 0.1\ \text{mm}$ | 15 |
|  | Calcined bauxite at $1000\ ^\circ\text{C} \leq 0.1\ \text{mm}$ | 15 |
|  | Cement $\leq 0.1\ \text{mm}$                                   | 7  |

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