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PHYSICAL, THERMAL, AND MECHANICAL PROPERTIES OF CALCIUM ALUMINATE CEMENT-BASED REFRACTORY CONCRETE AT ELEVATED TEMPERATURE

Manh Tien Tran[1*], Xuan Hong Vu[2], Emmanuel Ferrier[2]

[1]Department of Mechanisms of Materials, Hanoi University of Mining and Geology (HUMG), no.18, Pho Vien Street, Ha Noi, Vietnam

[2]Université de LYON, Université Claude Bernard LYON 1; Laboratoire des Matériaux Composites pour la Construction LMC2, 82 bd Niels Bohr, 69622 Villeurbanne Cedex, France

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[*] *Corresponding author*
Email: tranmanhtien@humg.edu.vn; Tel: +84 973094737

Abstract. In the past decade, calcium aluminate cement is widely used to manufacture refractory concrete for infrastructure works which frequently were subjected to elevated temperature thanks to the thermal stability by the high content of aluminum. This paper presents experimental results of the physical, thermal, and mechanical properties of calcium aluminate cement-based refractory concrete specimens. As experimental results, with a calcium aluminate content of about 50%, the refractory concrete provides remarkable physical, thermal, and mechanical properties. The high density and low water content were characterized for this concrete. The thermal diffusivity coefficient of refractory concrete is lower from 3 to 4 times than that of normal concrete while the conductivity is around of 1.05 (W/m.K). Furthermore, from the thermomechanical tests, the direct tensile strength and Young's modulus of refractory concrete were identified at different temperature levels. The effect of elevated temperature on the performance of this refractory concrete was analyzed and highlighted.

Keywords: Physical property, tensile strength, compressive strength, refractory concrete, elevated temperature.

1. INTRODUCTION

Fire or high temperature is one of the dangerous factors that can occur in infrastructure works. According to a report by the International Fire and Rescue Services, there have been more than 1.600.000 fires reported worldwide [1]. Fires in 2017 resulted in 3763 deaths and $10.7 billion in property damage [2]. Fire is one of the causes to reduce the durability and service life of reinforced concrete (RC) members. In the case of fire, the physical and mechanical properties of the concrete and steel in RC members could be degraded under the elevated temperature action. This deterioration can lead to the failure of the structure element, which in turn leads to the collapse of the structure.

In the last decades, the refractory concretes with good thermal properties contributed an important role in reducing the impact of fire or high temperature on the durability of the building. Nowadays, calcium aluminate cement (CAC) is widely used for that thanks to the thermal stability of the respective concrete by the high content of aluminium [3] [4].

In the literature, several studies aim to identify the physical, thermal, and mechanical properties of refractory concrete based on calcium aluminate cement [5] [6]. Most mechanical results showed a strain-softening behaviour in compression and splitting tensile behaviour of CAC concrete and cement-based composite specimens. Bareiro *et al.* [6], have studied the influence of alumina content (51%, 71% and 90%) on the chemical and mechanical behaviour of refractory concretes. According to [6], when increasing the heating temperature, the mechanical behaviour showed a significant drop in compressive strength and Young's modulus of refractory concretes with alumina contents ranging from 51% and 71%. However, the refractory concrete containing 90% by weight of alumina exhibited a more stable mechanical behaviour, while maintaining to some extent the mechanical properties.

In standard Eurocode 2 [7], the mechanical properties of the concrete decrease almost linearly with elevated temperature up to 600°C, while its thermal property varies highly in the temperatures ranging from 115 °C to 200 °C, caused by the different physicochemical phenomena of water and CSH gel, depending on the nature of aggregates [8].

According to the works of Bareiro *et al.* [6], thermogravimetric analyses and derived thermogravimetry (TGA/DTG) technics were used to obtain the thermograms of three types of refractory concretes with different alumina contents (49.42%, 72.23%, and 90.14% respectively for the A51, A71, and A90 specimens). The results obtained show that the peaks of the TGA curve correspond to the following process: dehydration (free water and HA gel), loss of metastable hydrates (CAH_{10} and C_2AH_8), the reaction of conversion of metastable hydrates into stable carbonate (AH_3 and C_3AH_6) and sintering. A similar result was obtained from the TGA and DTG analyses for the refractory matrix based on CAC cement with the alumina content of 51.45% in the work of Rambo *et al.* [9].

Concerning the thermal properties, Vejmelková *et al.* [10] have identified the thermal conductivity of original concrete and fibre-reinforced concrete based on calcium aluminate cement (CAC) at three temperature levels (room temperature, 400 °C, and 1000 °C). The results on all the materials analyzed showed a significant dependence according to the water content. The thermal conductivity in the water-saturated state was up to twice that in the dry state. Its value also decreases from 1.2 W/m.K to 0.8 W/m.K with the increase in temperature ranging from room temperature to 1000 °C.

In conclusion, these researches have highlighted the remarkable properties of CAC concrete at elevated temperatures. They gave a better understanding of this refractory material. However, these researches just have considered some properties, not have shown the relationship between these properties at elevated temperatures. To the best of the authors' knowledge, rare existing results consider the physical, thermal, and mechanical properties of CAC concrete in an overall study. Thus, it would be interesting to solve this studying.

In order to manufacture the textile-reinforced concrete (TRC) composite for fire application, the authors' work aims to study the multi properties of refractory concrete. This paper presents the experimental results concerning the physical, thermal, and mechanical properties of refractory concrete based on calcium aluminate cement. In this paper, the aggregate and cement used were manufactured as a commercial product with high calcium aluminate content (from 40% to 50%). The concrete specimens were tested to identify their physical, thermal, and mechanical properties as density, water content, thermal conductivity, compressive strength, tensile strength, Young's modulus. The effect of elevated temperature on the mechanical properties was identified from the thermomechanical tests at different temperature levels.

In the following sections of this paper, experimental work, including the materials, test specimens, and test procedure, are presented (section 2). The experimental results and discussion will then be presented, analysed (section 3). This paper ends with a presentation of main conclusions and future works.

2. EXPERIMENTAL WORKS

2.1. Materials

2.1.1. Calcium aluminate cement

The cement used in this experimental study is a commercial product that is manufactured under a quality management system certified according to the requirements of ISO 9001 n.d. [11]. It is primarily composed of calcium aluminates, which makes it a binder of choice for refractory applications. Its high content of mono-calcium aluminate gives concrete refractory cement excellent mechanical performance. When refractory cement is used with suitable components, its low iron oxide content makes the concretes able to withstand reducing atmospheres or contain carbon monoxide. The rheological characteristics of this cement are suitable for all types of implementations and, in particular, for casting and spraying by the dry process. It is particularly recommended in cases such as speed of hardening, high mechanical performance, and fire applications. The characteristics of the cement used in this study were determined according to European standard BS EN 196-2 2005 [12]. Table 1 shows the chemical composition of this cement.

2.1.2. Aggregate

The aggregate used in this experimental study is also a commercial product. It is a synthetic siliceous-aluminous-calcium aggregate obtained by fusion and contains about 40% alumina. It is characterized by high density and exceptional hardness. This aggregate also has a remarkable chemical affinity with the refractory cement used in this study.

Table 1. Chemical composition of the cement used and of the synthetic calcium aluminate aggregate used.

Cement		**Aggregate**	
Compound	**Content (%)**	**Compound**	**Content (%)**
Al_2O_3	50.8 - 54.2	Al_2O_3	37.5 – 43.5
CaO	35.9 - 38.9	CaO	35.0 – 40.0
SiO_2	4.0 - 5.5	SiO_2	3.0 – 5.0
Fe_2O_3	1.5 - 2.5	Fe_2O_3	14.0 – 18.0
MgO	< 1.0		
TiO_2	< 4.0		
Na_2O+ K_2O	< 0.5		

It is manufactured under a quality management system certified according to the requirements in [11]. Table 1 shows the chemical composition of the aggregate used in this study (chemical analysis according to [12] and chemical composition determined on the material before crushing and classification). For a thin thickness application, the maximum overall diameter was less than 1.25mm. A particle size study was performed based on Andreasen's sieve (0.08, 0.16, 0.315, 0.63, 1.0, 1.25 mm) for this aggregate. Fig. 1 shows the granulometric curve of the synthetic calcium aluminate aggregate.

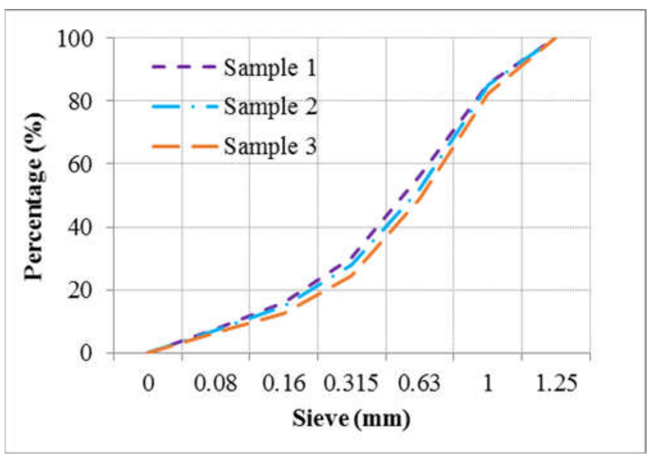


Figure 1. Granulometric curves of synthetic siliceous-aluminous-calcium aggregates.

2.1.3. Concrete composition

The refractory concrete used in this experimental study was designed following the compressible packaging model (CPM) routine [13][14] for an application in textile reinforced-concrete (TRC) composite. It is composed of two main components as presented above: siliceous-aluminous-calcium synthetic aggregate and calcium aluminate cement. With the high content of aluminium, this concrete is remarkable choice for fire applications. For low application thickness, a small amount of superplasticizer and viscosity modifier was added into the concrete composition. The water/cement ratio was 0.35. Table 2 presents the composition of refractory concrete in this experimental study.

Table 2. Mixture composition of the refractory concrete.

Density (estimation): □ = 2584.4 kg/m3	
Aggregate (kg/m3)	1676
Cement (kg/m3)	669
Superplasticizer (kg/m3)	4.34
Viscosity modifier agent - VMA (kg/m3)	0.51
Water (kg/m3)	234.2
Water/cement ratio	0.35

2.2. Specimens

2.2.1. Specimens for tensile tests at different temperatures

For the direct tensile tests, the concrete specimens were moulded with the hand lay-up technique in the rectangular shape of cross-section. The dimensions of this specimen type were 600 mm in length, 51 mm in width, 20 mm in thickness. The average cross-sectional area of a refractory matrix specimen was determined by three measurements (width and thickness) at three different points along with the specimen.

2.2.2. Specimens for compressive tests

The compressive strength of concrete was identified according to the standard [15]. So, concrete specimens were rectangular with the dimensions of 160 mm x 40 mm x 40 mm (length x width x thickness). These specimens were moulded and cured in laboratory conditions. At the age of 28 days, these specimens were labelled before testing.

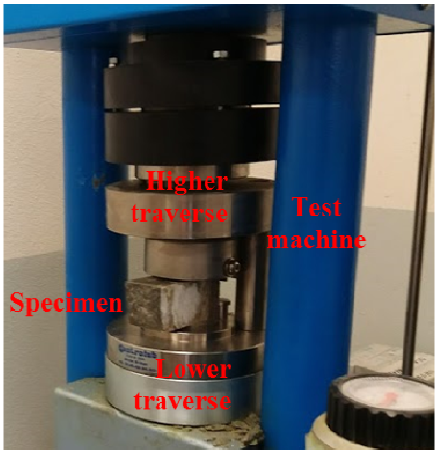
2.2.3. Specimens for other tests

For the identification of the physical properties of concrete (density, water content, and air mass loss), nine cylindrical specimens (d = 27.5 mm and h = 53.3 mm) of the concrete were manufactured and stored under two different conditions: in the air (3 ones) for the observation of the mass loss as a function of time; and in water (6 remaining ones) for the observation of the saturation kinetics. Furthermore, three other cylindrical specimens (d = 24 mm and h = 7.94 mm) were used to identify the thermal diffusivity coefficient of this refractory concrete.

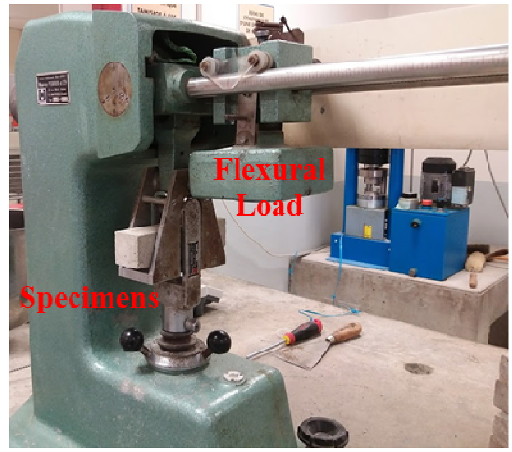
2.3. Procedure of tests

2.3.1. Compressive and flexural tests

The concrete specimens with the dimensions of 40mm x 40mm x 160mm were first tested in three point bending according to European standard BS EN 196-1 [19]. Fig. 2b presents the configuration of the flexural test in this experiment. After cracking and splitting into two parts, the compressive strength of concrete was identified from these parts, as presented in Fig. 2a. The test procedure consists of four steps as follows: placing the sample on the lower traverse of the compressor, moving the higher traverse into contact with the specimen, compressing concrete samples, and recording the result after crushing the specimen. The number of tests is 6 for the flexural test and 12 for the compressive one.



(a) Compressive tests



(b) Flexural tests

Figure 2. Configuration of compressive and flexural tests for concrete specimens.

2.3.2. Direct tensile tests

The direct tensile tests were performed on the concrete specimens with the thermomechanical machine at different temperatures. This test machine can generate simultaneously the mechanical loading (by the traverse) and action of elevated temperature (by the furnace) as presented in Fig. 3. The temperatures in the furnace were measured by integrated thermocouples and controlled by the control system. The longitudinal deformation of concrete specimens was measured by the laser sensor equipped on the machine. The test procedure consists of three steps as following: increasing the temperature around the specimens to the desired temperature level, maintaining the desired temperature for a period of 1 hour, applying the mechanical quasi-static load monotonically to the specimen until failure.

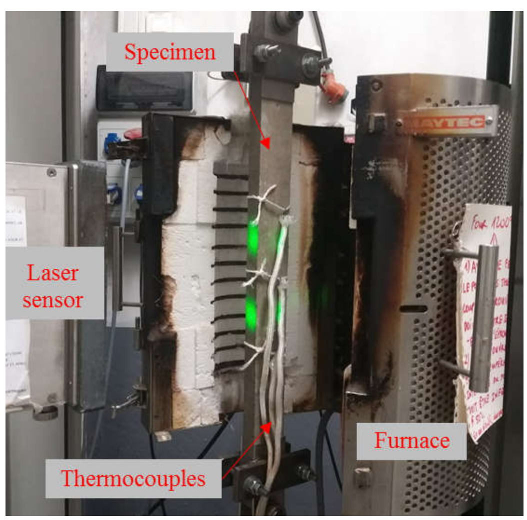


Figure 3. Configuration of direct tensile tests at elevated temperature.

2.3.3. Thermal tests

The thermal tests were performed on refractory concrete specimens to identify its thermal diffusivity coefficient at different temperatures basing on the flash method [16]. It means that the temperature variations of the back face are recorded after depositing an energy Dirac on the front face. For this method, a high-temperature diffusivimeter (HTD) apparatus was designed and used to measure apparent thermal diffusivity over temperatures ranging from 20 °C to 1000 °C under a vacuum environment. The procedure is followed for each measurement of temperature: homogeneity of temperature (at measurement temperature) inside concrete sample; acquisition of the thermo-signal, depositing a laser flash on the upper face, recording the temperature rise of the underside. After that, the thermal diffusivity coefficient was calculated from the obtained data.

3. RESULTS

3.1 Thermal and physical properties of refractory concrete

3.1.1. Physical properties

- *Density:*

The density of refractory concrete was determined from nine cylindrical specimens after removing the mould at the concrete age of one day. Table 3 below presents the obtained result concerning the density of the refractory concrete. From table 3, the average value (2.53 g/cm^3) is lower than the estimated value (2.58 g/cm^3) of concrete density. It could be explained by the evapotranspiration (in one day), the rising of porosity causing by the specimen moulding process.

Table 3. Calculation of the density on the cylindrical specimens of the refractory concrete.

Specimens	Diameter - d (mm)				Height - h (mm)				Volume V (cm3)	Mass M (g)	Density □ (g/cm3)
	d1	**d2**	**d3**	**d_{ave}**	**h1**	**h2**	**h3**	**h_{ave}**			
1	27.37	27.48	27.35	27.40	53.46	53.45	53.71	53.54	31.55	78.80	2.50
2	27.62	27.83	27.6	27.68	53.78	53.36	53.62	53.59	32.24	80.30	2.49
3	27.55	27.54	27.48	27.52	53.78	54.04	53.95	53.92	32.07	80.80	2.52
4	27.33	27.14	27.14	27.20	51.56	51.3	51.98	51.61	29.98	77.00	2.57
5	27.71	27.69	27.67	27.69	53.05	54.03	53.4	53.49	32.20	79.70	2.48
6	27.21	27.19	27.13	27.18	52.6	52.53	52.57	52.57	30.48	78.70	2.58
7	27.47	27.51	27.37	27.45	52.46	52.07	52.24	52.26	30.91	78.90	2.55
8	27.13	27.12	27.17	27.14	54.38	54.37	54.74	54.50	31.51	80.80	2.56
9	27.48	27.37	27.32	27.39	54.72	54.77	54.36	54.62	32.16	81.00	2.52
Average value											2.53

- *Mass loss:*

The mass loss of the refractory concrete was determined from three samples that were cured in the air (labelled from 1 to 3). The observation series was performed for 80 days with the timelines as presented in Fig. 4. The mass loss value in average calculated by interpolation method for the concrete age of 28 days is about 0.4 %.

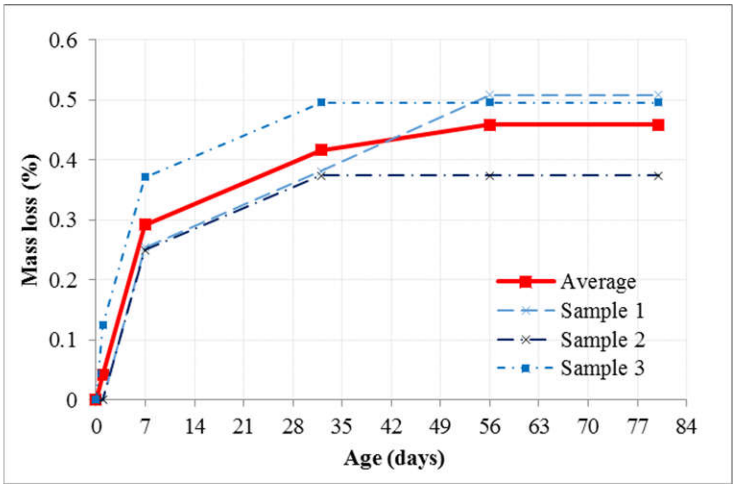


Figure 4. Mass loss of cured refractory matrix samples in the air as a function of time.

- *Water absorption:*

The water absorption of the refractory concrete was determined from six remaining samples that were cured in water (labelled from 4 to 9). This observation series was also performed during 80 days with the timelines as presented in Fig. 5. The water absorption value in average calculated by interpolation method for the concrete age of 28 days is about 3.4 %.

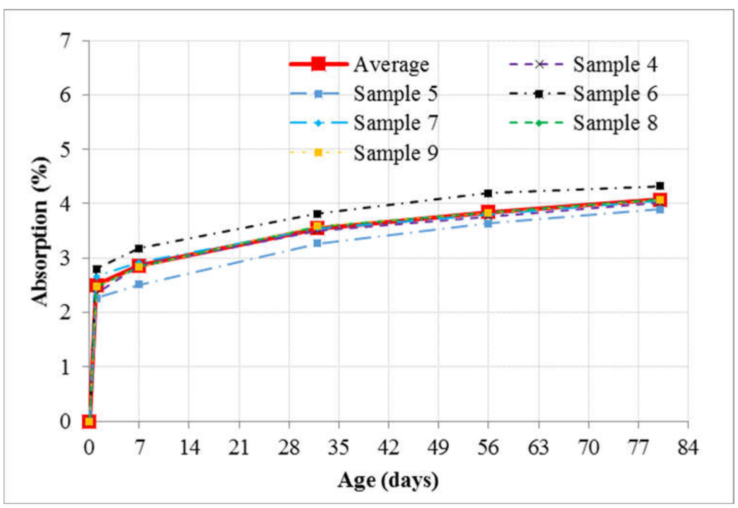


Figure 5. Water absorption as a function of time of refractory matrix samples stored in the water.

- *Water content:*

In order to determine the water content of the refractory concrete, the three first specimens were dried in the furnace at the temperature of 105 °C. From the data of the mass of the concrete specimen, the water content is calculated by the following equation:

$$W(\%) = 100.\frac{M_{28}-M_d}{M_d} \quad (1)$$

Where: M_d is the mass of the concrete specimen in the dry state;

M_{28} is the mass of the concrete specimen at the age of 28 days;

As result, the water content is 2.26% on average over 28 days as presented in Table 4

Table 4. Water content of refractory matrix samples at the age of 28 days.

Time (days)	**Mass at age of 28 days M28 (g)**	**Mass at dry status Md (g)**	**Water content W (%)**
Sample 1	78.50	77.10	2.20
Sample 2	80.00	78.60	2.16
Sample 3	80.40	78.90	2.41
Average			2.26

3.1.2. Thermal properties

- *Thermal diffusivity:*

The results of thermal diffusivity tests performed on cylindrical specimens of refractory concrete at four temperature levels (20 °C, 75 °C, 150 °C, and 600 °C) are shown in figure 6 below. At room temperature, the diffusivity coefficient was 0.46 mm^2/s on average. At higher temperatures, its value decreased slightly, reaching 0.39 mm^2/s at 150 °C. The diffusivity coefficient gradually reduces in small ranging with elevated temperature. Note that the result at 600 °C was not convergent and not presented in figure 6. So, at a temperature higher than 150 °C, this coefficient could be estimated from the extrapolation method. In comparison with the existing results on different materials in the literature [17] [18], the refractory concrete provided a remarkable thermal performance by the thermal diffusivity coefficient. Its value was similar to that of lightweight aggregate concrete specimens obtained by Nguyen et al. [17]. Furthermore, the thermal diffusivity coefficient of refractory concrete is lower by 3 to 4 times than that of TRC-PPS (aluminous mortar filled with polypropylene fibers) specimens obtained by [18].

- *Thermal conductivity:*

The thermal conductivity of refractory concrete is calculated by the following equation:

$$k\,(T) = \alpha(\mathrm{T}).\rho(\mathrm{T}).C_p(T) \quad (2)$$

Where: k(T) (W/m.K) is the thermal conductivity of refractory concrete at temperature T;

α(T) (m^2/s) is the thermal diffusivity of refractory concrete at temperature T, determined by the experiment as presented in Fig. 6 and the extrapolation values;

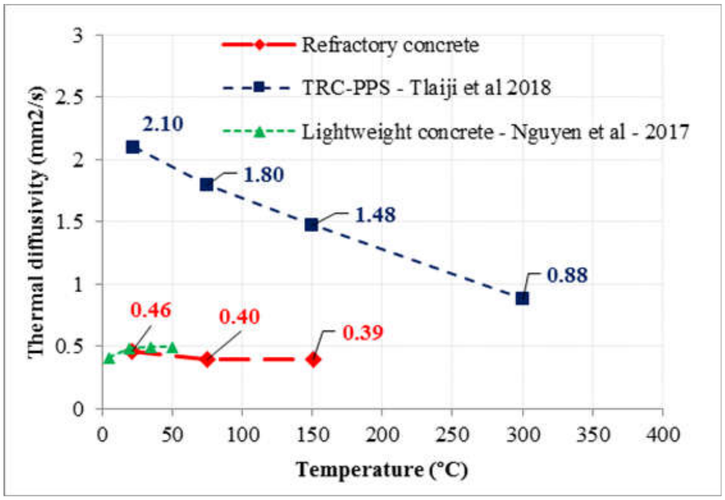


Figure 6. Comparison of thermal diffusivity of different materials [17] [18].

□(T) (kg/m^3) is the density of refractory concrete at temperature T, determined by the experiment for value at room temperature and the evolution of its value with elevated temperature based on experimental results of [6] with the similar content of aluminium (51%).

C_p(T) (J/kg.K) is the specific heat of refractory concrete at temperature T, determined from [7] with a water content of 2.26%.

Fig. 7 presents the evolution of the thermal conductivity of refractory concrete as a function of temperature. As an obtained result, its value varied greatly in the temperature ranging from 100 °C to 200 °C. The maximum value was 1.73 (W/m.K) at 101 °C. Outside of this range, this thermal property gradually decreased from 1.05 (W/m.K) at room temperature to 0.87 (W/m.K) at 1000 °C.

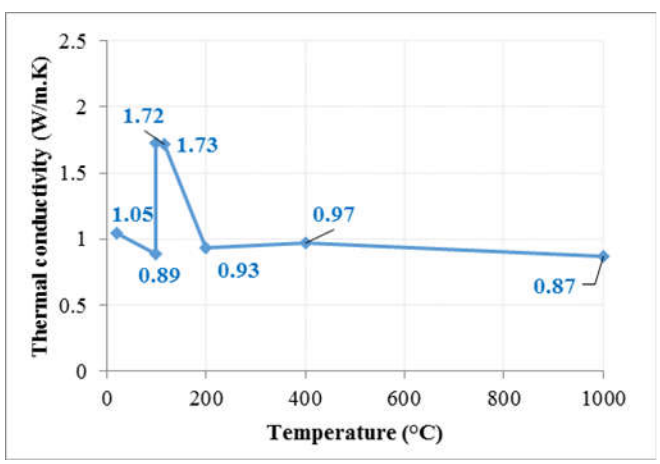


Figure 7. Evolution of thermal conductivity of refractory concrete as a function of temperature.

3.2 Mechanical properties of refractory concrete at room temperature

3.2.1. Flexural tensile strength

The concrete specimens with the dimensions of 40mm x 40mm x 160mm were firstly tested in three points bending to identify the flexural tensile strength. The obtained data were analysed according to European standard BS EN 196-1 [19]. The flexural tensile strength is calculated by the following equation:

$$\sigma_{ft} = \frac{3\,P\,x\,106.7}{b^2} \quad (3)$$

Where: σ_{ft}(MPa) is the flexural tensile strength;

P (N) is the maximum bending force of each specimen;

b (mm) is dimension of cross-section;

106.7 (mm) is the distance between two supports at two ends;

As result, the flexural tensile strength of studied concrete (at the age of 28 days) was 12.5 MPa on average with a standard deviation of 1.04 MPa.

3.2.2. Compressive strength

The compressive tests were performed on the concrete specimens (dimensions of 40mm x 40mm x 160mm) after splitting into two parts in the flexural tests. The compressive strength is calculated by the following equation:

$$\sigma_c = \frac{P}{A} \quad (4)$$

Where: σ_c (MPa) is the compressive strength of concrete specimen;

P (N) is the maximum force of each specimen, recorded from the test machine;

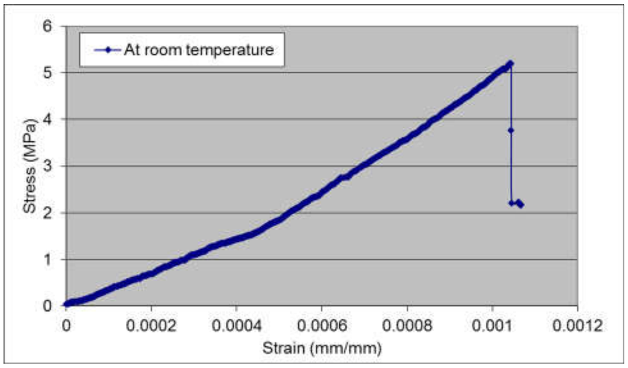
A (mm^2) is area of the compressive zone on the lower and higher traverses, A = 16 mm^2 in this case of test machine (Fig. 2);

All output data were analysed according to European standard BS EN 196-1 [19] to identify the compressive strength. As an obtained result, its average value for refractory concrete at the age of 28 days was 58.1 MPa with a standard deviation of 2.50 MPa.

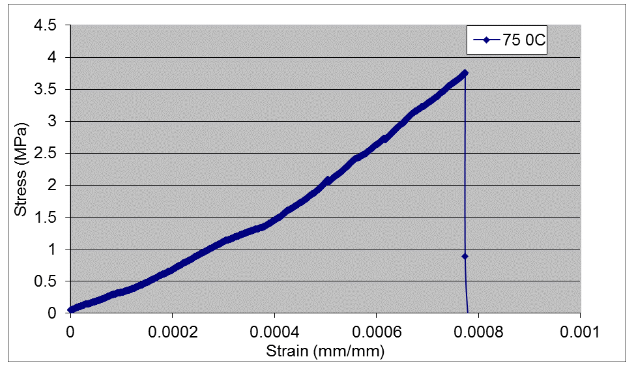
3.3 Thermomechanical tests at elevated temperature

3.3.1. Tensile behaviour of refractory concrete

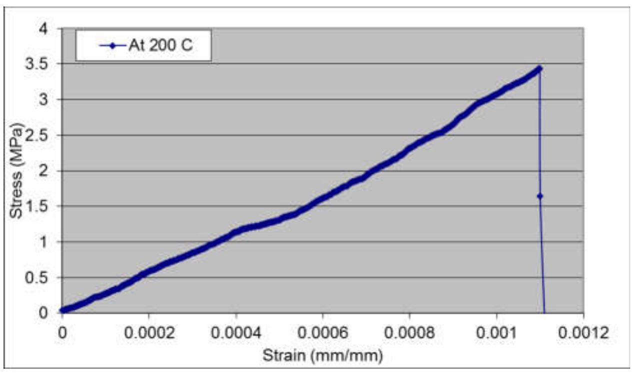
The direct tensile behaviour of refractory concrete at the elevated temperature ranging from 25 °C to 700 °C was identified from thermomechanical tests. As a result, the refractory concrete specimens provided only the brittle behaviour at different temperatures, which was noticed by an abrupt rupture when it reached a limit state. Fig. 8 presents the thermomechanical stress-strain relationship of concrete specimens which could be represented for the remaining ones at the same temperature level.



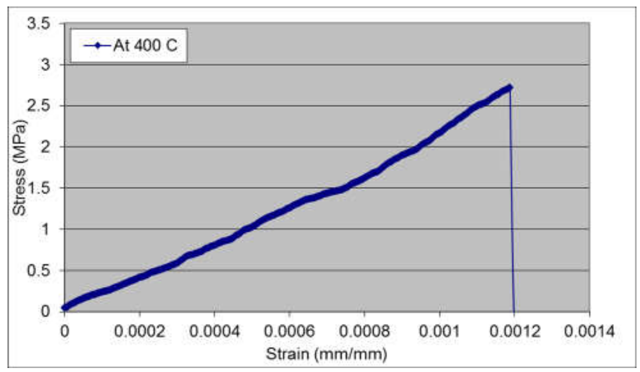
At room temperature



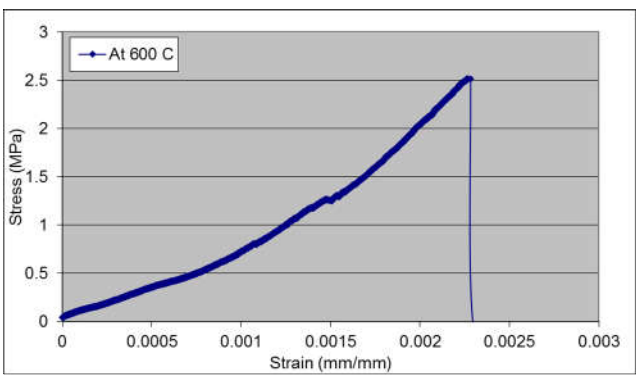
At 75 ^{o}C



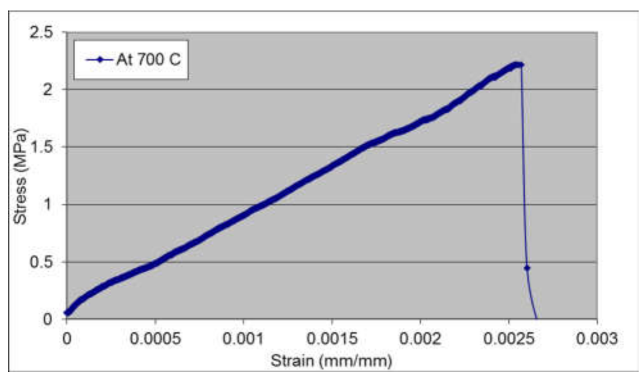
At 200 ^{o}C



At 400 ^{o}C



At 600 ^{o}C



At 700 ^{o}C

Figure 8. Thermal mechanical behaviour of refractory concrete at different temperatures.

3.3.2. Thermomechanical properties of refractory concrete

The mechanical properties of refractory concrete were identified from the stress-strain curves at different temperatures. The thermomechanical properties of the concrete (direct tensile strength ($\square_{C,T}$) and Young's modulus ($E_{C,T}$)) at six temperature levels are presented in Table 5 below. From Table 5, it could be found a gradual decrease of these properties with elevated temperature. The tensile strength decreased from 5.29 MPa at room temperature down to 2.36 MPa at 700 °C. Stiffness value was in a similar tendency of reduction from 8.41 GPa down to 1.44 GPa. In comparison with the property at room temperature, the concrete specimens remained their tensile strength of about 45 %, while it lost almost (than 80 %) its stiffness at 700 °C.

Table 5. Thermomechanical properties of refractory concrete at the elevated temperatures (the standard deviation values are written in parallel).

Specimens	Tensile strength (MPa)	Young's Modulus (GPa)
At 25 °C	5.29 (0.11)	8.41 (1.14)
At 75 °C	3.76 (0.04)	6.58 (0.26)
At 200 °C	3.69 (0.33)	4.39 (0.60)
At 400 °C	3.28 (0.60)	3.09 (0.23)
At 600 °C	2.54 (0.29)	1.67 (0.27)
At 700 °C	2.36 (0.21)	1.44 (0.42)

3.4 Discussion

As result, it could be found the effect of elevated temperature on the thermal and mechanical properties of CAC concrete specimens. Concerning the evolution of thermal properties, the diffusivity coefficient was in a lower value, so it decreased slightly with elevated temperature thanks to the thermal stability of the cement and aggregates. The thermal conductivity coefficient increased suddenly in the temperatures ranging from 100 °C to 200 °C caused by the phenomena of water evaporation.

Regarding the effect of elevated temperature on mechanical properties of refractory concrete specimens, figure 9 presents the evolution of normalized values depending on the temperature. From figure 9, the normalized stiffness value decreased gradually with a non-linear curve while the normalized value of ultimate tensile strength reduced with two intervals: a significant decrease at a temperature from 25 °C to 75 °C and a slight one at the higher temperatures.

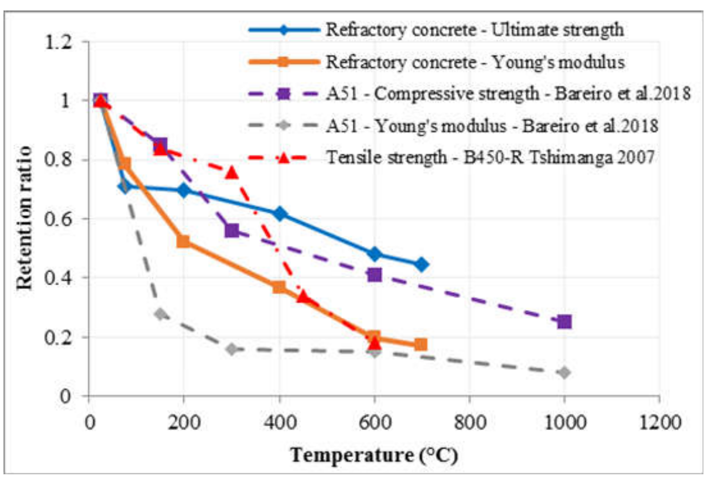


Figure 9. Evolution of normalized mechanical properties obtained for refractory concrete specimens in comparison with the available experimental results.

In comparison with the experimental results on A51 concrete specimens in Bareiro et al. [6], it could be found that the studied concrete remained normalized stiffness value at the temperature ranging from 75 °C to 600 °C. Regarding the tensile strength, non-available results were in [6] to compare. However, in comparison with the tensile strength of the B450-R specimen in Tshimanga, [20] it could be found the remarkable tensile strength of studied refractory concrete at elevated temperatures higher than 400 °C. At 700 °C, this concrete could remain its capacity of about 45 %.

4. CONCLUSION

Aim to study a cementitious matrix for an application in textile-reinforced concrete (TRC) composite in case of fire, this paper presented the results on the physical, thermal, and mechanical properties of calcium aluminate cement-based concrete specimens at elevated temperatures. The following conclusions can be drawn for this study:

The thermal and physical of refractory concrete have been identified and calculated from the experiment. The concrete provided a high density and a small value of thermal diffusivity thanks to the high content of alumina. These properties presented a remarkable capacity of refractory concrete at elevated temperatures.

The mechanical tests according to the European standard BS EN 196-1 [19] have identified the compressive and flexural tensile strength at room temperature. The tensile strength at elevated temperatures has been characterized by thermomechanical tests. From the results, the elevated temperature affected the mechanical properties of refractory concrete. In comparison with the property at room temperature, the concrete specimens remained their tensile strength of about 45 %, while it lost almost (than 80 %) its stiffness at 700 °C.

For future works, it will be interesting to study the relationship between the thermal diffusivity and the mechanical properties of concrete by a comparative study of different concretes. It also could develop a numerical model for the thermal and mechanical behaviour of refractory concrete at elevated temperatures. This model bases on the input data from the results of this study.

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