

AGGREGATE TYPE INFLUENCE ON MICROSTRUCTURAL BEHAVIOR OF CONCRETE EXPOSED TO ELEVATED TEMPERATURE

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A b s t r a c t

Exposure of concrete to high temperatures affects its mechanical properties by reducing the compressive strength, bending... etc. Factors reducing these properties have been focused on by several studies over the years, producing conflicting results. This article interested an important factor, that is the type of aggregates. For this, an experimental study on the behavior of concrete based on different types of aggregates: calcareous, siliceous and silico-calcareous subjected to high temperatures. In addition, the particle size distribution of the aggregates was chosen to be almost identical so that the latter does not affect the behavior of the concrete. Aggregates and concrete samples were subjected to a heating/cooling cycle of 300, 600 and 800 °C at a speed of 1 °C/ min. The mechanical and physical properties of concrete before and after exposure to high temperatures were studied. In addition, a microstructural study using a scanning electron microscope and a mercury porosimeter was performed. Thus, a comparative study between various

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researches on the mechanical properties of concrete exposed to high temperatures containing different types of aggregates was carried out.

The compressive strength test results showed that the concrete based on siliceous aggregates (C-S) has better mechanical performance up to 300 ° C. However, above 300°C, the compressive strength decreases faster compared to calcareous-based concrete (C-C). According to the mercury porosimeter test, at 600 ° C, C-SC and C-S concretes have the highest number of pores compared to C-C concretes. The microstructure of concrete at high temperatures was influenced mainly by the aggregate's types and the paste-aggregate transition zone.

This study reinforces the importance of standardizing test procedures related to the properties of concrete in a fire situation so that all the results obtained are reproducible and applicable in other research.

Keywords: types of aggregates, high temperature, compressive strength, microstructure, mercury porosimeter

1. INTRODUCTION

Fire is one of the most severe accidents to which structures can be subjected [1, 2]. Thus, when designing concrete structures, engineers must take into consideration the consequences associated with high temperatures, such as explosive spalling [3] [2]. This phenomenon observed by many research hers often results in a serious deterioration of concrete [4], [5].

Fire resistance of concrete is mainly influenced by the temperature and fire duration, the type of aggregate and the cement used in its composition as well as porosity and moisture content of concrete [6].

The aggregates occupy 70 to 80% of concrete volume which strongly influences its thermal behavior [7]. Researchers Fares, H., et al [8] Xing, Z., et al [9] have shown that there is an accumulation of water at the paste-aggregate interface. This high amount of water creates a more porous area and initiates the cracking of concrete. In addition, the quality of the paste-aggregate bond strongly depends on the geometry of aggregates and their mineralogical nature (calcareous, siliceous, etc.). Damage in concrete subjected to temperature rise is mainly caused by cracking, which occurs due to the variation in temperature between the aggregates and the paste [10]. Consequently, the paste-aggregate bond plays a very important role in the resistance of concrete subjected to high temperatures [7]. Several studies have shown that concrete with siliceous aggregates has a low fire resistance than that with calcareous aggregates [11]. This is mainly due to the decomposition of calcareous aggregates at a higher temperature than siliceous aggregates [11]. In addition, at 573 °C, the thermal expansion of siliceous aggregates due to the transformation of α quartz to β quartz causes an expansion of concrete [10]. However, Robert, F et al. [10] showed that concretes prepared

with siliceous aggregates can have better residual mechanical behavior than those prepared with calcareous aggregates. The decomposed carbonates release carbon dioxide during heating. This carbon dioxide rehydrates during cooling. This rehydration reaction causes an increase in the volume of 44% which is the cause of spalling after cooling of calcareous concrete [12].

Hager, I et al [7] studied an ordinary concrete based on silica-calcareous aggregates heated up to 600 °C. They showed, using SEM, that the cracks pass through the cement paste and the interfacial transition zone and reach the siliceous aggregates. This phenomenon indicates that certain siliceous aggregates have low resistance at a temperature of 350 °C. Jean-Christophe Mindeguia et al. [13] have shown that concrete made with silica-calcareous aggregates exhibits considerable cracks at the cement paste/aggregate interface (especially around gravel), due to the high thermal deformation mismatch.

In addition, in their study Masood, A., et al. [14] cited that after heating to 800 °C, the calcareous aggregate concrete retained 40% of its resistance. Thus, crystallization and microstructure play an important role in the thermal stability of concrete based on the different types of aggregates subjected to heating-cooling cycles at high temperatures up to 750 °C.

Over the past 30 years, the study of concrete mechanical properties on a macroscopic scale has gradually reached maturity. Besides, the strength and durability of concrete depend strongly on the pore structures [15]. So, microscopic studies seem necessary to deepen our knowledge of the behavior of concrete subjected to high temperatures [16].

In addition, a recent study in 2021 by Sollero et al. [17] analyzed studies on the evaluation of the mechanical properties of concrete exposed to high temperatures. This analysis reveals discrepancies in the results obtained by several authors due to insufficient characterization of the type of concrete used. Thus, they have shown that the granite aggregates used in brésilite are different from the siliceous aggregates used in Europe. For this, it is necessary to take into consideration the country, the test methodology and the composition of the concrete, more particularly the mineralogical, chemical and physical characteristics of the aggregates.

The objective of this work is to provide a comparison of a mechanical property of ordinary concrete and its evolution with temperature (300, 600 and 800 °C) in the case where the composition of the cement pastes and the mortar, as well as their volumes remained the same in all samples. The parameter that will change was the mineralogical nature of the aggregate.

For this, ordinary concretes containing different types of aggregates (calcareous, siliceous and silico- calcareous) were made. Compressive strength was measured for all concretes, and a microstructural analysis using SEM and mercury intrusion porosimetry was performed. Mercury intrusion porosimetry (MIP) is widely

applied to study the porous structures in concrete materials. It is an essential means of the microstructural analysis of concrete [15, 18].

An analysis of the various research works on the effect of the type of aggregates on the mechanical behavior of ordinary concretes subjected to high temperature was carried out.

2. EXPERIMENTAL STUDY

2.1. Materials

This three series of concrete specimens were prepared. The first one was made with siliceous aggregates (C-S), the second series with silico-calcareous aggregates (C-SC) while the third series was made with calcareous aggregates (C-C). Their characteristics chemical nature is given in Table 1. The physical and mechanical characteristics of aggregates are presented in Table 2. The maximum nominal size and the grading curve were similar for all aggregates (see Fig.1) Portland Cement CEM II/B-L-42.5 N was used for all concrete mixtures. Its chemical composition is given in Table 3.

Table 1. Chemical characteristics of aggregates

| Composition (%) | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | CaO | MgO | SO ₃ | K ₂ O ₃ | Na ₂ O | Cl ⁻ |
|-----------------|------------------|--------------------------------|--------------------------------|-------|------|-----------------|-------------------------------|-------------------|-----------------|
| C | 1.86 | 0.69 | 0.38 | 93.27 | 3.69 | 0.09 | 0.16 | 0.09 | 0.06 |
| SC | 13.42 | 2.72 | 2.85 | 74.73 | 4.83 | 0.093 | 0.51 | 0.06 | 0.05 |
| S | 93.09 | 1.10 | 1.53 | 5.17 | 0.46 | 0.1 | 0.13 | 0.44 | 0.01 |

Table 2. Physical and mechanical characteristics of aggregates

| Aggregate's type | C | | SC | | S | |
|--|------|-------|------|-------|-------|-------|
| | 0-3 | 3-8 | 0-3 | 3-8 | 0-3 | 3-8 |
| ρ_{abs} Absolute density (g/cm ³) | 2.55 | 2.66 | 2.66 | 2.62 | 2.67 | 2.5 |
| γ_{app} bulk density (g/cm ³) | 1.57 | 1.54 | 1.54 | 1.39 | 1.57 | 1.43 |
| Los Angeles (%) | | 16.67 | | 17.71 | | 21.37 |
| Porosity (%) ^a | 38.4 | 49.06 | 42.1 | 46.9 | 38.43 | 46.07 |
| Fineness modulus of sand 0 / 3 | 2.53 | | | | | |

^a Present the intergranular porosity corresponds to the percentage of intergranular voids of the different fractions of aggregates (coarse gravel and sand) in the container [19].

Table 3. Chemicals composition of cement

| Composition | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | CaO | MgO | SO ₃ | K ₂ O | Na ₂ O | CaO | Cl ⁻ |
|-------------|------------------|--------------------------------|--------------------------------|-------|-----|-----------------|------------------|-------------------|------|-----------------|
| Content (%) | 23.07 | 4.94 | 4.88 | 58.95 | / | 1.92 | 0.35 | 0.13 | 0.73 | 0.002 |

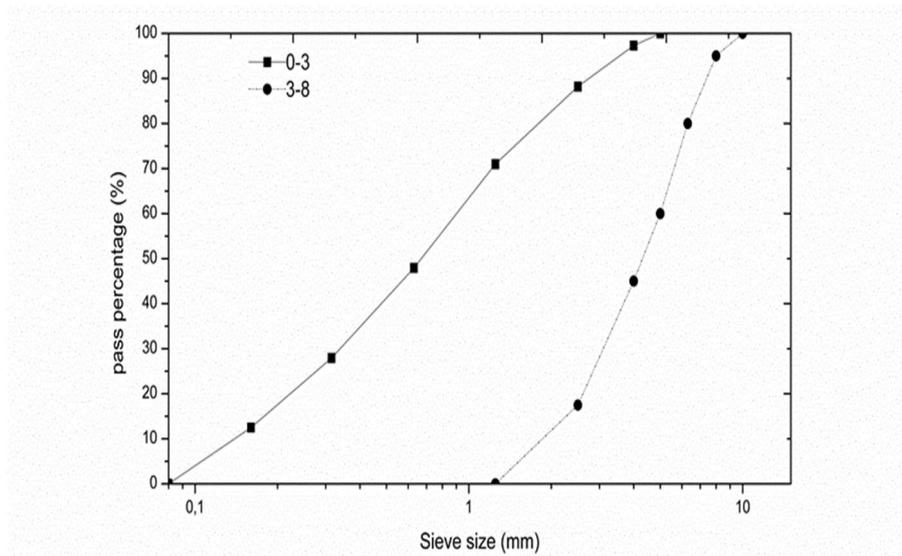


Fig.1. Sieving test results of aggregates: Particle Size Distribution

2.2. Mixture proportions

The mixture design was made according to DREUX-GORISSE formulation method [20]. The same cement content (340 kg/m³) was used in all mixtures with a water to cement ratio (W/C) of 0.6. Mixtures proportions are given in Table 4. Cubic specimens 100 x 100 x 100 mm³ were prepared to use in all tests. Specimens were cured in moisture for 28 days (20 ± 2°C, RH>95%) before testing.

Table 4. Composition of the studied concretes

| Mixtures | Cement (kg/m ³) | Water (L) | Aggregate size range | | Compressive strength at 28 days (MPa) |
|----------|--------------------------------|--------------|--------------------------|---------------------------|---|
| | | | 0-3(kg/ m ³) | 3-8 (kg/ m ³) | |
| C-C | 340 | 224 | 688.28 | 1037.06 | 34.58 |
| C-SC | 340 | 224 | 717.97 | 1017.64 | 38.24 |
| C-S | 340 | 224 | 702.78 | 1103.78 | 33.61 |

2.3. Heating and cooling cycle

After curing in normal conditions all specimens were undergone a pre-drying (at 100 °C) until mass constant to evaporate the maximum of free water in the cementitious structure and avoid spalling risks of specimens in the furnace during heating.

Using an electronic furnace, specimens were subjected to temperature rise with a constant rate of 1°C/min up to the target temperatures of 300, 600 and 800 °C. After reaching the target temperatures specimens were left in the oven for 1h at each temperature. Then, the specimens were remained inside until the furnace cooled down to 25°C. Applying a slower speed, it allows chemical reactions to proceed; and avoid spalling of concrete [7].

2.4. Instrumentation and testing (microstructural)

Using an electronic furnace, specimens were subjected to temperature rise with a constant rate of 1°C/min Mercury porosimetry (Autopore IV, Micromeritics) was used to characterize the pores size in the concrete before and after exposure to high temperatures (Fig. 2b). The pore size distribution is determined by applying various levels of pressure to a sample immersed in mercury. Scanning electron microscopy (SEM) (Hitachi S-3400N) was also used to observe the microstructural modifications (Fig. 2a).



(a) (b)
Fig.2. (a) Scanning electron microscope (SEM) (b) Mercury porosimetry (MIP)

3. RESULTS AND DISCUSSION

3.1. Compressive strength

Fig. 3 and the table 5 have shown the reduction factors of the residual compressive strength of ordinary concrete ($F_c(T) / F_c(20)$) from international studies published between 2011 and 2021, compared to the values proposed by Eurocode2 part 1-2 [17] [21] [22] [23] [24] [25] [26] [27] [6].

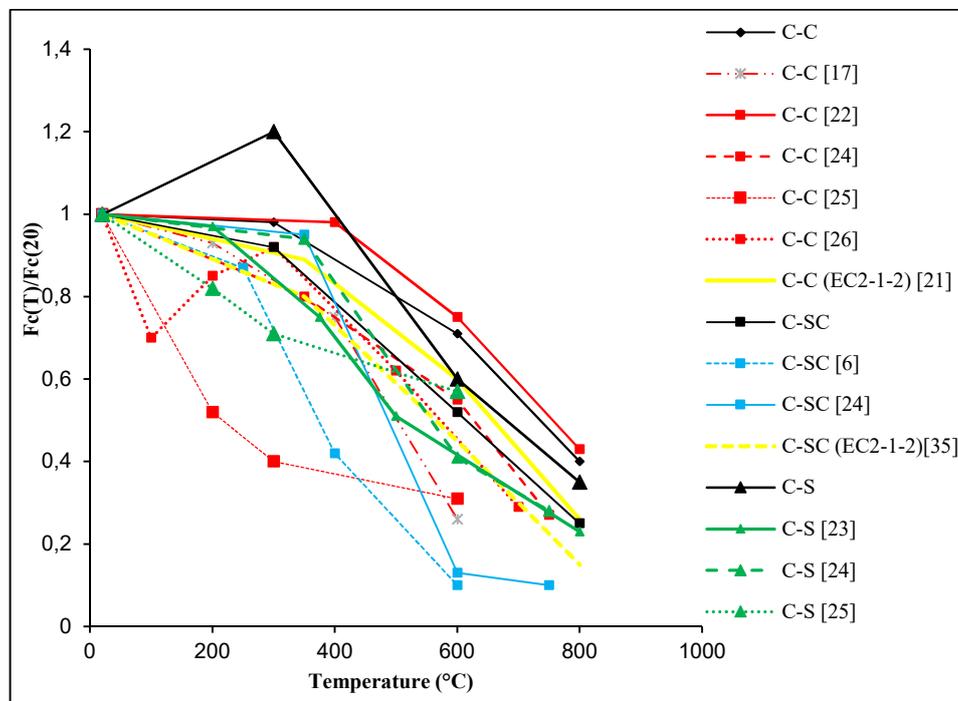


Fig.3. Evolution of the residual compressive strength of concrete as a function of temperature

Table 5. Summary of the researches carried on the concrete subjected to high temperature

| Ref | Year | Sample size | Agregate type | W/b | Test temperature | Poudre (kg/m ³) | Cure | Heating and cooling cycle |
|------|------|--|---|------|---|------------------------------------|---|---|
| [17] | 2021 | Cylindrical Ø 10x30 cm ² | Calcaire | 0.45 | 200, 400 or 600°C | 360 cement | The preheated cores were exposed to the temperature of 100 °C for 1 h to reduce humidity, and were kept in plastic film until being heated in the oven. | The heating and cooling rate was 1 °C/min. The maximum temperature was kept during 60 min. |
| [22] | 2015 | Cubes of 100 mm | Calcaire | 0.4 | 400, 600, 800 or 1000°C | 400 cement | The specimens were tested in compression at the age of 28 days without being exposed to elevated temperatures; those exposed to elevated temperatures were transferred to a dryer for drying at 105°C for 24 h. | The heating at a rate of 6.67°C/min and held at each peak temperature for 2 h |
| [23] | 2017 | Cylindrical Ø 100x200 mm | Siliceous (Granit) | 0.4 | 200, 375, 500, 700 or 800°C | 385 cement | At 28 days, the concrete is cured under water. After 28 days of age, the specimens were kept in the controlled environment of 25° C and 70% RH till the completion of tests. | The heating rate of 12 °C/min were used till the target furnace temperature is reached and then the target temperature is maintained till the completion of the test. |
| [24] | 2011 | Cylindrical specimens (Ø16×32 cm and Ø11×22 cm) | - Calcareous -Siliceous -Silico-calcareous | 0.6 | 150, 300, 450, 600 or 750 °C | 362 cement | After seven days, the specimens are demoulded and placed into sealed plastic bags at ambient temperature for 90 days | at a rate of 1°C/min stabilization at constant temperature for one hour and finally cooling to the ambient temperature. |
| [25] | 2013 | Cylindrical specimens (Ø 25 x75 mm) | Siliceous -Silico-calcareous | 0.32 | 100, 200, 300, 500, 750, or 1000 °C. | 580 cement +63.8 Silica fume | All specimen aged for at least 285 days | Heating /cooling rate 1 °C/min. Holding the temperature constant for 60 min, and then cooling at the same rate. |
| [26] | 2011 | cylinder specimens (Ø100×200m m) | Calcareous | 0.35 | 100, 200, 300, 500 and 700°C | 471 cement | The specimens were cured in water at 20±2°C until the age of 7days and then conditioned in a chamber at a temperature of 20±1°C and a relative humidity of 50±5% for another 90days | The heating at a rate of 1°C /min. stabilization at constant temperature for one hour and finally cooling to the ambient temperature |
| [6] | 2013 | cylinder specimens (Ø 100/40 mm) | - Calcareous -Siliceous | 0.5 | 200, 300 and 600 °C | 350 and 355 cement | After 28 days of curing period in the water, specimens were dried in an oven at 100 ± 5 °C for 48 h to provide similar moisture conditions | 6±2°C/min heating rate and exposed at the target temperature for 2 h in a furnace |

From Fig.3 we see that, up to 300 °C, the compressive strength decreases slightly by 0.28% and 0.91% for concrete with silico-calcareous aggregates and concrete with calcareous aggregates respectively. This can be explained by an accumulation of water at the paste-aggregate interface. This high amount of water creates a more porous area in which cracking will be initiated in concrete. In addition, the quality of the paste-aggregate bond strongly depends on the geometry of the aggregates and their mineralogical nature (calcareous, siliceous ... etc.) [17] [27] [24] [23] [6]. Hager et al [28] explained the phenomenon of decreased resistance through dehydration that occurs in CSH gel reduces its volume, which in turn increases the porosity of the cement matrix.

On the other hand, for concretes based on siliceous aggregates (C-S), there is a slight increase in resistance of 10% at 300 °C. This increase according to Fares, H. et al [8, 29] is due to the additional hydration of the anhydrous cement due to the large water movements which occur during heating between 100 and 300 °C. Consequently, the grains of anhydrous cement were rehydrated. As for Behnood et al [30] and Ghandehari et al [31], they attributed this increase in resistance to water departure between particles of CSH (Van Der Waals forces) [32].

At 600 °C, all the samples showed a reduction in compressive strength of 47%, 62% and 71% for C-C, C-S and C-SC respectively. At this point, perhaps the microstructure is rapidly deteriorated by a chemical transformation of the crystalline brucite and the decomposition of hydration products such as the evaporation of bound water in the CSH [28, 33]. These intrinsic chemical transformations produce more cracks leading to an increase in porosity of around 15% (Fig.4).

Concrete with calcareous aggregates (C-C) had higher residual strength compared to that with siliceous aggregates. This can be explained by the improved matrix-aggregate transition zone mainly due to the reaction of calcareous with calcium aluminates in the cement paste, thus forming the calcium carboaluminates [9, 24]. In addition, the decrease in the resistance of C-S and C-SC is due to the bursting of silica and its transformation from α quartz to β quartz at 573°C accompanied by a change in volume from 1% to 5.7 % [7] [34]. The researchers [9], [28, 33] explained this loss by micro-cracking at the paste-aggregate interface [35]. The better performance of C-C compared to C-S, in residual compression, is probably due to a better bond between the calcareous aggregates and the paste. Quartzite aggregates are round with a smooth surface unlike calcareous aggregates which are crushed with a rough texture. Other researchers, Onundi, L.O et al [33], showed that calcareous aggregates provide concrete with higher fire resistance and better spalling resistance than siliceous aggregates. This is mainly because the calcareous aggregates have a higher thermal capacity (specific heat), which is beneficial in reducing spalling and increasing fire resistance.

Above 600 °C, the compressive strength loss of C-C concrete becomes more important due to the decarbonation of calcium carbonate. As for concrete based on siliceous aggregates, it has greater thermal stability [9].

The analysis of studies conducted on the evaluation of the mechanical property of concrete exposed to high temperatures reveals discrepancies in the results obtained by several authors due to insufficient characterization of the type of concrete used. The lack of standardization of tests and specimens was identified. Table 5 shows that factors such as: cement dosage, type of aggregates, cure before the heating cycle, size of specimens (cylindrical or cube) and rate of heating-cooling are contributing to the divergence of results residual mechanical performance results [17]. From Fig 3 we note that four works of [17] [24] [26] [6] (concrete based on calcareous aggregates) show low values compared to EC2 part 1-2 [21]. By cons, our results and [22] reveal residual high resistance compared to EC2-2 part 1-2. This difference is due to the lack of application of the factors for reducing the residual strength of concrete in EC2-2 [21]. Moreover, this distinction according to Razafinjato et al. [34] is due to the different properties of thermal expansion, conductivity, thermal capacity and mechanical resistance of aggregates which represent 80% of the total composition of concrete. In addition, G. Khoury [36] have shown that the type and properties of the coarse aggregate is probably the most important parameter in the behavior of concrete subjected to high temperature. Therefore, it is possible that two types of concrete with similar mechanical properties have different behavior in fire due to changes in the type of aggregate. Sellori et al [17] have shown that the granite aggregates used in Brésille are different from the siliceous aggregates used in Europe in their thermal behavior. For this, it is necessary to take into consideration the country and the mineralogical, chemical, thermal and physical characteristics of the aggregates to know the behavior of concrete subjected to high temperature.

3.2. Weight loss of concrete

The average value of the weight loss for silico-calcareous concretes, siliceous concretes, and calcareous concretes are presented in fig 4.

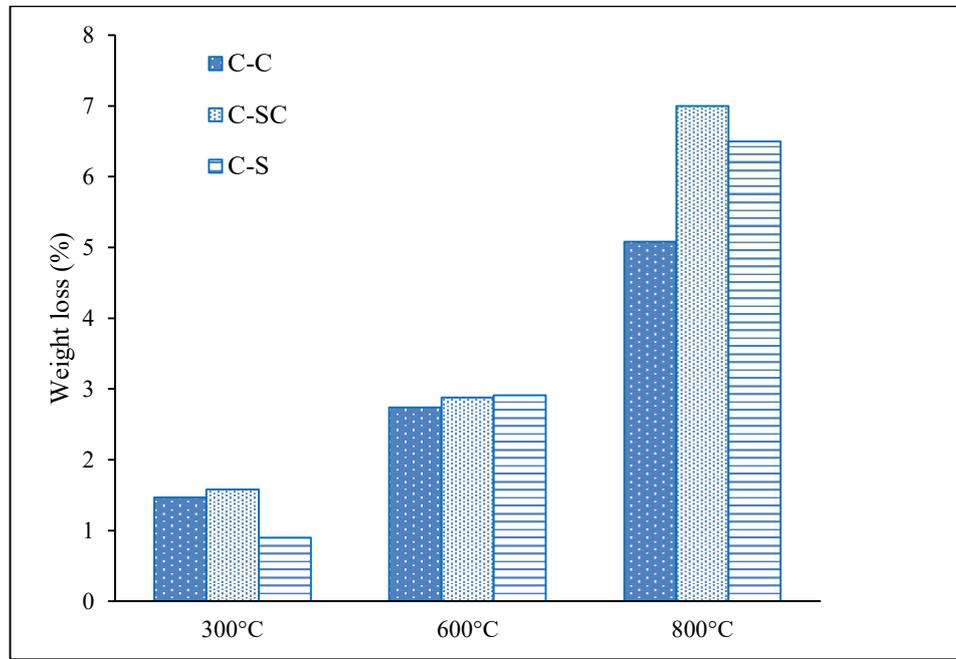


Fig. 4. Mass loss of concrete

The composition of the cement pastes and the mortar as well as their volumes remained the same in all the samples. Therefore, the mass loss varies depending on the mineralogical nature of the aggregate only. In the figure, there is at 300 °C a small difference in the loss of mass between the three concretes. This is due to the free water brought by the aggregates. The values of the mass loss of concrete are consistent with the results of the mass loss of aggregates (Fig 8).

At 300 °C C-SC represents a slight increase in mass loss compared to other concretes. These results corroborate those reported by Mind-éguia [37] and [24] who found that the mass of silico-calcareous concretes decreases more rapidly than calcareous concretes. This is due to the higher water uptake of porous carbonates thus leaving bound water [24]. At 800 °C, the mass loss of C-C is due to the decarbonation of the calcareous aggregates. On the other hand, the cause of the high mass loss of C-SC is the thermal instability of the silico-limestone aggregates compared to the siliceous and calcareous aggregates.

3.3. Visual observation of aggregates

Fig. 5 shows the macroscopic observations of aggregates subjected to heating cycles (300 °C, 600 °C and 800 °C). Observations were taken after each heating cycle.

| | Calcareous aggregates (C) | Silico-calcareous aggregates (SC) | Siliceous aggregates (S) |
|--------|---|---|--|
| 300 °C |  |  |  |
| 600 °C |  |  |  |
| 800 °C |  |  |  |

Fig.5. Visual observation of aggregates subjected to high temperature

At 300 °C, all types of aggregates remain intact. However, at 600 °C, a color change was observed for calcareous and silico-calcareous aggregates. Regarding siliceous aggregates, radial cracks perpendicular to the surfaces were observed see Fig.6.



Fig. 6. Siliceous aggregates at 600 °C

Calcareous aggregates remain intact, which confirms the results of [9]. At 800 °C, the calcareous aggregates were cracked (Fig. 7) and turned white. These changes can be explained by the decarbonation of calcareous. The calcite (CaCO_3) transforms at high temperature into quicklime (CaO) with a CO_2 departure [34]. After 5 hours of cooling, the aggregate surface is damaged due to the hydration of CaO . This free CaO reacts with relative humidity and transforms into portlandite $\text{Ca}(\text{OH})_2$ with a volume expansion of 200% [9]. This remark can explain the bursting of C-C at 800 °C.



Fig. 7. Calcareous aggregates at 800°C

3.4. Mass loss of aggregates

The mass loss values for the C, S and SC aggregates are shown in fig 8.

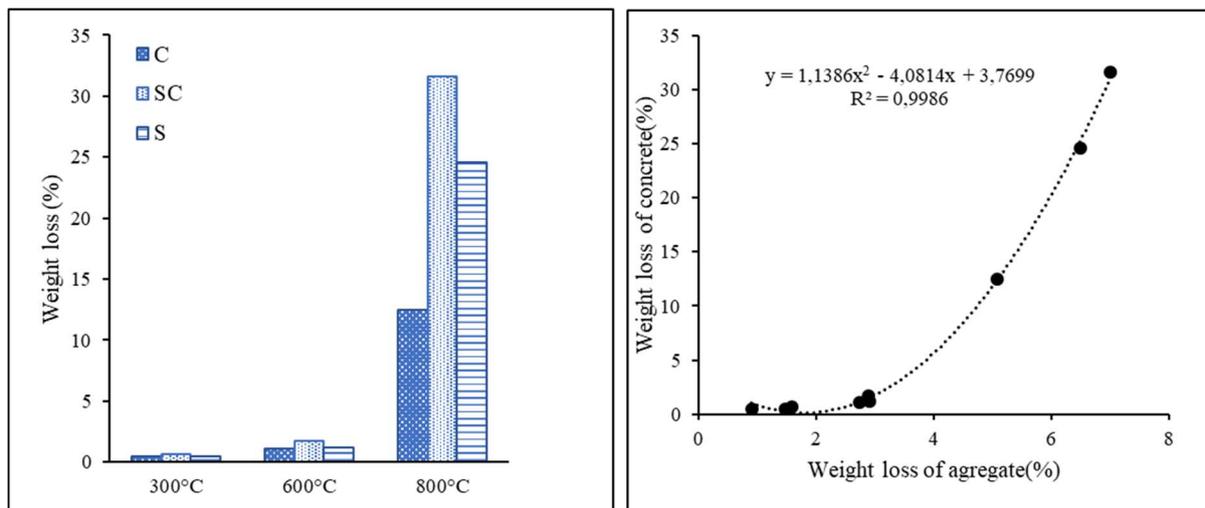


Fig. 8. (a) Average mass loss values for C-aggregates, SC-aggregates and S-aggregates. (b) Correlation between the loss of mass of concrete and the loss of mass of aggregates

The mass loss value is obtained by an average made on 10 grains for each mineralogical nature of aggregates. The mass loss measurements were only carried out on aggregates dried beforehand in the oven. From the fig 8, the aggregate mass loss values were almost similar up to 300 °C. At 600 °C, an increase in mass loss of aggregates of approximately 1.1% -1.7% compared to control samples. At 800 °C, the loss of the SC aggregates increases significantly by 31% compared to the control. [24] explained this increase in the mass loss by the high porosity of aggregates and their significant thermal instability compared to siliceous and calcareous aggregates.

Fig 8.b shows a polynomial relationship between the mass loss of concretes and aggregates with $R^2 = 0.99$. This reveals the importance of the properties of aggregates in the behavior of concrete subjected to high temperatures.

3.5. Mercury intrusion porosimetry

The high mechanical strength of concrete comes from a low cumulative porosity. The pore size distribution is the ratio between the pores volumes of different sizes and the overall pore volumes [18]. To better understand the mechanical behavior of the studied concretes, after exposure to high temperature, the pore size distribution was evaluated using mercury porosimetry. The obtained results are presented in Fig.9 (a-b).

We see from Fig.9 (a) that the total intrusion of mercury for C-C, C-SC and C-S subjected to different temperatures is 0.0872 ~ 0.0492 mL / g, 0.0811 ~ 0.0511 mL / g and 0.097 ~ 0.061 mL / g, respectively.

At 25 °C, the differential distribution of pore sizes for C-C and C-SC is almost similar with a pore diameter between 0.011 to 2.18 μm and 0.021 to 2.08 μm respectively. On the other hand, for C-S the pore diameter varies between 0.011 to 8 μm , see Fig.9 (b). According to Fig. 10, the porosity of C-C, C-CS, C-S is 11.2%, 14.3%, 14.1% respectively. This difference is mainly due to the aggregate's nature and the quality of the matrix-aggregates transition zone. C-C Concrete has the lowest pore diameter and porosity, which results in higher compressive strength. This is due to the reaction of calcareous with cement components improving the paste-aggregates adhesion [6], [38]. It can be seen, also, that the porosity obtained by the MIP increases with temperature. This can be explained by the evaporation of free water at a temperature of about 150 to 200 °C. In addition, the CSH begins to decompose [39]. As water evaporates, pore pressure develops. This build-up of pore pressure, combined with the effect of temperature and mass concentration of the constituents, causes evaporated water to transport through the pores of the material (see fig.11). In addition, this vapor pressure in the concrete generates high tensile stress causing the concrete to burst. As the temperature increases, the pore size distribution curve increases. At 600 °C, C-SC and C-S concretes have the largest number of pores compared to C-C

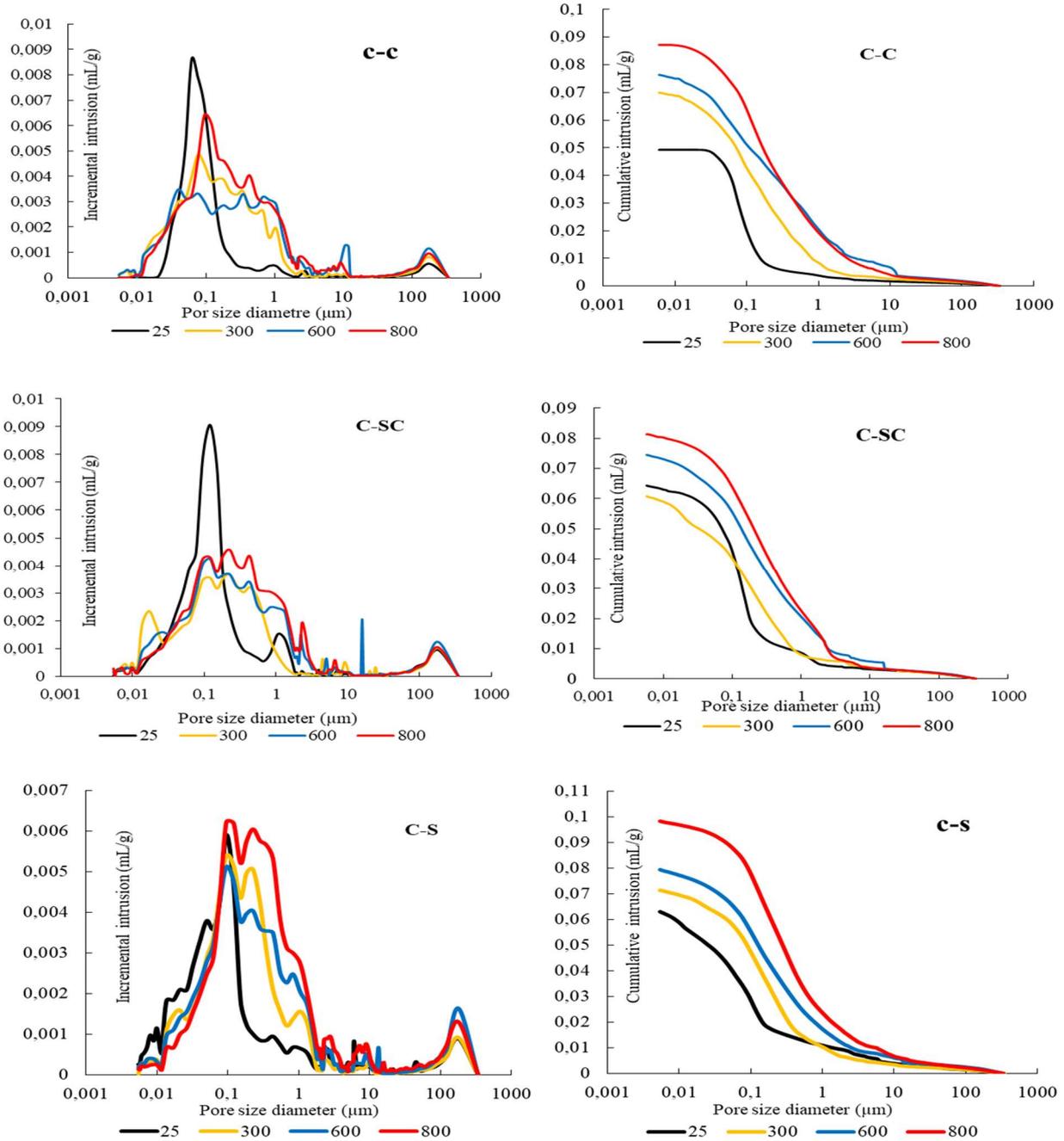


Fig. 9. Porosity size distribution with temperature for different concretes
 a) incremental intrusion volume, and b) cumulative intrusion volume

concrete (16.6%, 16.9% 15.8% respectively). This is explained by the bursting of silica and its transformation from α quartz to β quartz at 573 °C accompanied by a change in volume from 1% to 5.7%. These microstructure transformations cause voids in the paste-aggregate interface zone (see Fig. 11). At the same time, the mesopores and capillary pores pass through each other and the microstructures of old concrete loosen as the microcracks continue to develop. These remarks are in coherence with the compressive strength test results and the works of [7],[9] and [28], [18].

At 800 °C, almost complete destruction of the concrete structure creates a pore appearance of different diameters. Calcareous-based concrete shows larger pores with diameters between 0.011 and 14.48 μm and others between 36 and 341 μm . On the other hand, the distribution of pores in silico-calcareous and siliceous concrete has a peak between 0.01 and 4.71 μm and another between 45 and 341 μm .

In fact, calcareous-based concrete shows larger pores compared to silico-calcareous and siliceous concretes. Because calcareous aggregates become weaker due to the decarbonation of calcium carbonate. While siliceous aggregates have greater thermal stability [9].

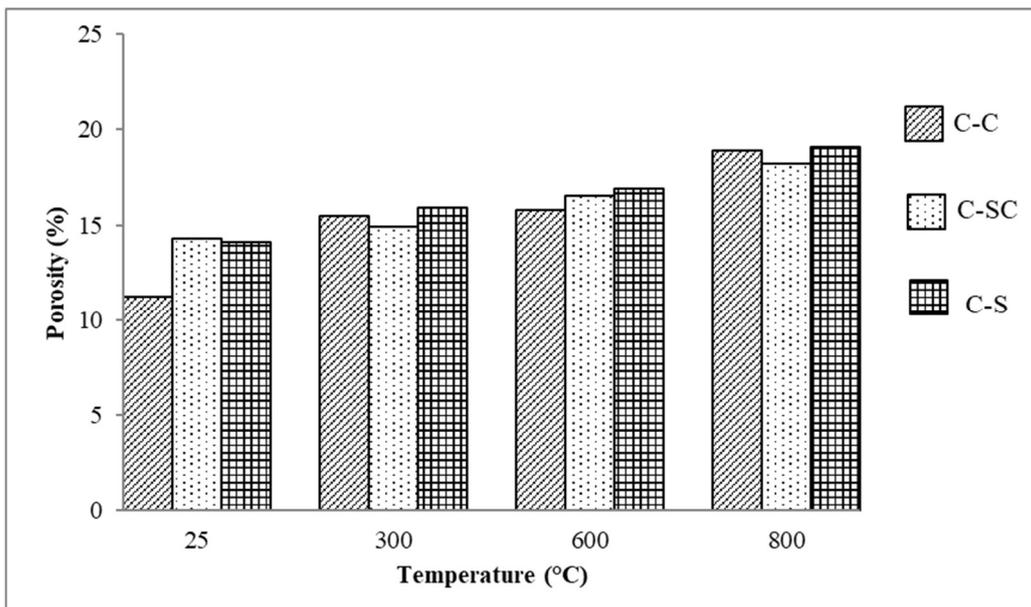


Fig.10. Total porosity of various compositions

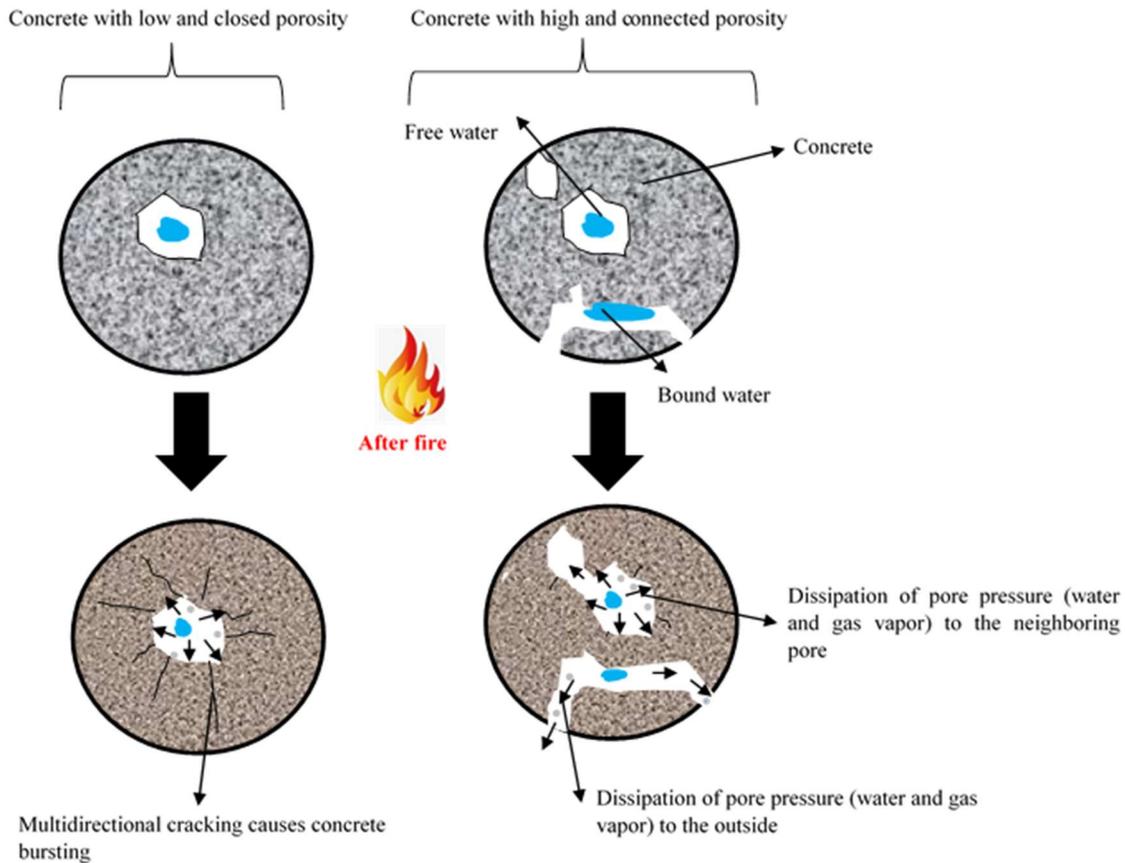


Fig. 11. Mechanism of the effect of porosity on the spalling of concrete

3.6. Description of thermal damage of concretes

Fig12 shows the cracking and crumbling of the surface of concrete subjected to temperatures of 800 °C.

The highest cracking rate is observed for C-SC concrete at 800 °C, which confirms the low mechanical resistance of this concrete at this temperature. C-C concretes heated to 800 °C will degrade after several days of cooling due to the hydration of CaO which results in Ca(OH)₂. The formation of portlandite is associated with an increase in volume and leads to deterioration of the sample.

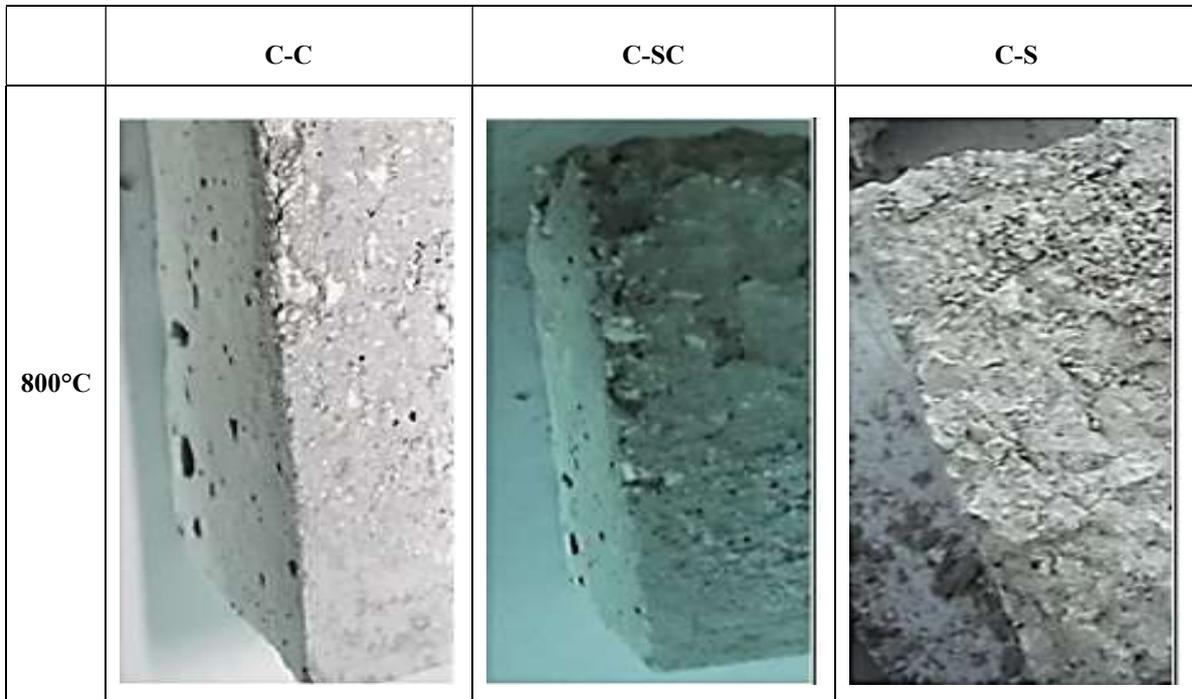


Fig.12. Specimens after heating/cooling cycles at 800 °C

3.6. SEM Observation

Heating the concrete causes the aggregates to swell and, at the same time, the cement paste around it to contract. Consequently, the paste-aggregate bond is the weakest point of heated concrete [35].

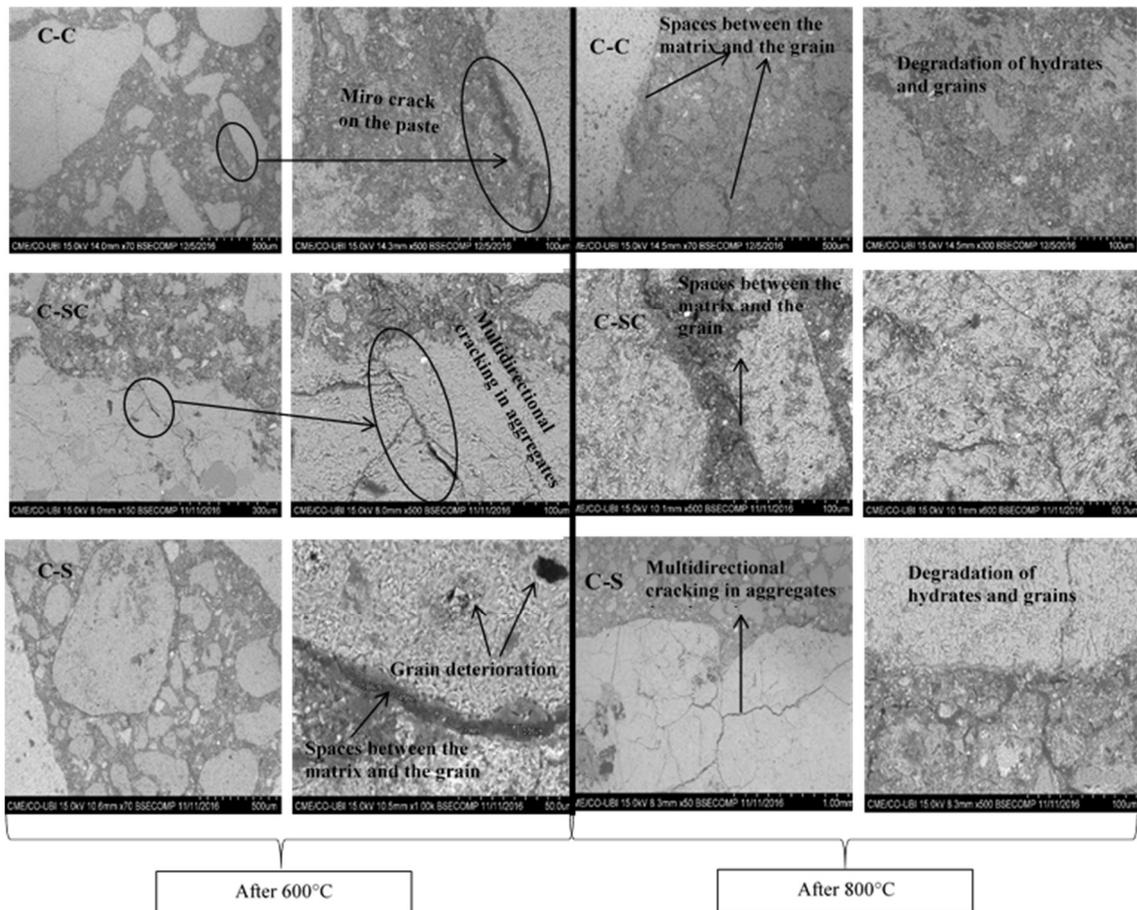


Fig.13. SEM observations of different samples under 600 and 800 °C

Fig.13 shows the morphology and microstructure of concrete after exposure to a high temperature of 600 °C and 800 °C.

At 600 °C, it can be seen that C-C has a more uniform and homogeneous transition zone compared to that with siliceous aggregates in which a discontinuity was observed. This is mainly due to the chemical reaction between the cement matrix and the calcareous aggregates. On the other hand, siliceous aggregates are chemically neutral (no chemical reaction occurs between the cement paste and the aggregate). Further, degradation inside the siliceous grain was noticed. This degradation is caused by the change from the α phase to the β phase of the siliceous aggregates accompanied by an increase in volume from 1 to 5.7% [9]. The cracks can reach an opening of 5.66 μm . Regarding C-SC, we notice that there are multidirectional cracks in the grains. This indicates the fragility of C-SC against

fire. These remarks are consistent with the MIP and the compressive strength test results at 600 °C in which C-C shows a high resistance compared to other concretes.

The SEM observation clearly shows the almost complete degradation of the internal structure of concrete after exposure to a temperature of 800 °C (Fig.13). Concretes with calcareous aggregates have more cracks around the grains. These cracks are due to the decarbonation of calcite. On the other hand, the rehydration of free lime (CaO) by ambient humidity which is accompanied by significant swelling causes the decohesion of aggregates and cement matrix.

According to Hager [7], the mineralogical composition of the aggregates determines the overall thermal deformations, since minerals are differentiated by their thermal expansion properties. That is, the minerals type controls the chemical and physical changes that occur during heating.

Carbonated stones (limestone and dolomite) are stable up to 600 °C. At high temperatures (around 600°C - 750°C), carbonates decompose into CaO and CO₂. In addition, the CaO, formed during decarbonation, can hydrate on cooling resulting in grains expansion of about 44%.

Heating the concrete increases the volume of aggregates, and at the same time, it causes the cement paste surrounding it to contract. As a result, the paste-aggregate transition zone is the weakest point during concrete heating. To a large extent, concrete damages are caused by cracking, which occurs due to the thermal disparity between aggregates and paste.

The SEM photos of different concretes subjected to 600 °C and 800 °C show cracks crossing the cement paste and passing through the interfacial transition zone. Also, cracks crossing siliceous aggregates are present indicating the tendency of certain siliceous aggregates to break at 350 °C [7].

4. CONCLUSION

Appropriate knowledge of the mechanical properties of concrete exposed to high temperatures is essential for the optimization of the design of fire-rated structures, post-fire analysis of structures and subsequent restoration or reinforcement.

This article presented an experimental study of macro and microstructural changes induced by the heating of concretes based on different types of aggregates. The following final notes summarize our main findings:

- The residual compressive strength of C-S is higher than that of C-C and C-SC at temperatures between 25 and 300 °C. However, between 300 and 600 °C, C-C has good fire resistance compared to C-S and C-SC. This is due to an improvement in the matrix-aggregates transition zone.

The MIP mercury intrusion results give a good microstructural explanation of the mechanical properties of concrete exposed to fire. The factors that affect the

porous structure of concrete are the aggregates nature and the matrix-aggregate transition zone.

- SEM observations of concretes heated to 600 °C show that C-C has a more uniform and homogeneous transition zone compared to that with siliceous aggregates in which a discontinuity was observed.
- The analysis of studies conducted on the evaluation of the mechanical property of concrete exposed to high temperatures reveal discrepancies in the results obtained by several authors due to insufficient characterization of the type of concrete used. The lack of standardization of tests and specimens was identified.

This study reinforces the importance of standardizing test procedures related to the properties of concrete in a fire situation so that all the results obtained are reproducible and applicable in other research.

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CONFLICT OF INTEREST

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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