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Abstract - *The objective of this study is to characterize a lightweight aggregate expanded in laboratory. Identified by physical, chemical, mineralogical and heat treatment, raw clay verified a set of conditions for the manufacture of lightweight aggregates. Smectite, illite and kaolonite were present. Expanded clay aggregates were then produced in the laboratory following a specific manufacturing process. The effect of temperature on the expansion properties of aggregates was studied. The properties obtained are comparable to those available in the commercial sector. The pellets were used in a concrete mix. The compressive strengths were determined at 3, 28 and 90 days with a cement water ratio 0.5 and a cement content of 350 kg/m^3. The results of the mechanical behavior study seem to be very similar to those required by the relevant standards for lightweight concrete. The study aimed to add value to the Tunisian clay which is not generally considered a natural resource for industrial development to meet the important needs of the construction industry and public works.*

Key words*: Expanded Tunisian clay, lightweight aggregate, lightweight concrete*.

I. Introduction

In Tunisia, the building art has undergone a revolution. In the twentieth century, the invention of concrete has replaced the stone with a compound based aggregates (sand, crushed aggregate) interconnected by a hydraulic binder. Modern requirements now require aggregates producers to offer products that meet specific criteria to develop the necessary composite material to our civilizations. The exploitation of the deposits has become an important activity in the industrial development of a country. The minerals resources are not inexhaustible. It is important, therefore, for the general interest, to use as rationally as possible in order to best ensure their sustainability in the context of the provisions related to sustainable development [1].
In the last decade, there has been an increase in the use of concrete and then the production rate of aggregates is increased. It is well known that according to concrete strength is composed about 60% to 70 % of aggregate [2]. In Tunisia, crushed aggregates are widely used because their low cost and high availability. The trend of Tunisian consumption aggregates was constant annual growth of 6 % between 2002 and 2008. A strong demand increase will lead to a global increase in the market price and will impact the global economy.

Lightweight aggregates (LWA) are different from crushed aggregates with regard to their physical and mechanical properties. They are natural ones with relatively low densities or stabilized elements obtained from thermal and chemical treatments. For their production, various materials have been used such as Perlites, wood chips, stabilized polymers, slate, shale and other ([3], [4] and [5]). It should be round, shaped with a relatively impermeable outer surface and provide a good adhesion to the cement paste. The obtained lightweight aggregates must meet certain requirements. They are chemically inert and stable beings in concrete [6]. The LWA properties differ according to raw material and production process. Their bulk density is less than 1200 kg/m^3. This lightness is due to high porosity of the aggregates. They contain different pores sizes. Some are large enough to have no capillary effect and they cannot be completely filled with water by capillarity. Generally, the quality of LWA is estimated by their densities because it is difficult to measure their mechanical properties.
To make a lightweight concrete (LWC), two aspects must be considered namely the formulation and the components. Making criteria should be established to meet the desired mechanical properties. Several researchers noted the influence of global aggregates characteristics on the quality of concrete. The mechanical

performance of concrete is influenced by lightweight aggregates characteristics such as density, size and aggregates strength. The researchers showed that the compressive strength increases with increasing the aggregates density. It depends on the aggregates size at low density ([7], [8], [9], [10] and [11]). Notably, clay is an abundant material in Tunisia. There are many deposits of clay and it has about a thousand careers spread throughout the country. We have very contrasting reserves with varied chemical compositions. It has large deposits of useful minerals that can be profitable industrial operations in order to meet the important needs of the construction industry and the public works. There is a growing interest in exploiting them in order to obtain a lightweight concrete. It reduces CO_2 emissions and the crushed aggregate consumption. For their wide availability and their physiochemical characteristics, the objective of this study is using Tunisian clay to make lightweight aggregates. Expanded clay was so produced in the laboratory following a specific manufacturing process. Then, it characterized and valorized to product a lightweight concrete.

II. Raw materials

II.1. Aggregates and cement

The cement used (Ce) for concrete manufacture was a Portland cement type CEM I 32.5 MPa. It has a specific gravity equal to 2950 kg/m^3 and an area equal to 3012 m^2/kg. Its chemical composition is detailed in Table 1. The gravel (Gr) used for ordinary concrete has a density equal to 2.64 g/cm^3. The sand (S) of 0/4 size has a density equal to 2.66 g/cm^3 and 2.36 of fineness modulus according to standards norms(NF P 18-598, NF P 18-554, EN 196-6) (table1).

II.2. Raw clay

II.2.1. Physical characterization

Table 2 shows the physical characteristics of the raw clay in accordance with Standards AFNOR norm (NF P 94-051, NF P94-050): Natural water content (Wn), Liquid Limit (LL), Plastic Limit (PL), Plasticity Index (PI), Consistency Index (CI) and specific gravity (Gs). The natural water content was 4.2%. According to Casagrande diagram the raw clay was highly plastic. Its Grain size distribution curve is shown in Figure 1. (NF P 18-560).

Table 1. *Physical properties of aggregates*

	S	***G***
Maximum size (mm)	*0-4*	*4-16*
Density g/cm^3	*2.66*	*2.64*
Bulk density g/cm^3	*1.6*	*1.4*
Water absorption (48h) (% by mass)	*-*	*1.61*
Sand Equivalent (%)	*96*	*-*

Table 2. Physical characteristics of raw clay

	Raw Clay
Wn(%)	4.2
LL(%)	92.3
PL(%)	41.83
PI (%)	50.48
CI (%)	1.74
G_s (g/m^3)	2.833

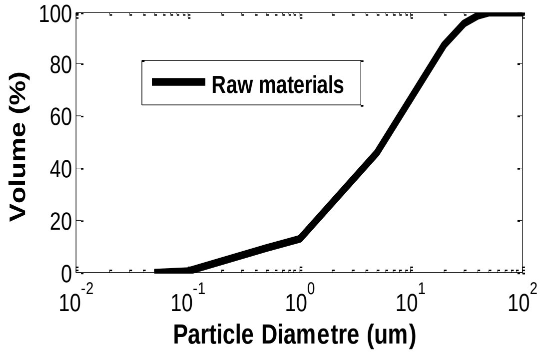


Figure 1. Grain size distribution curve of the raw clay

II.2.2. Chemical and mineralogical analysis

Figure 2. shows the results of the X-ray diffraction test. Among the minerals existing in raw clay, there are smectite as the principal mineral (S) with illite (I),

kaolinite (K), quartz (Q), geotite (G) and calcite ©. A comparison of the intensity of the diffraction lines showed that sample contains a greater proportion of smectite and illite. The chemical composition was determined by X-ray fluorescence. They contain mainly the elements Si and Al, with small amounts of Fe, Ti, K, P, Mn, Ca, Mg, as often observed in natural clay. The SiO_2 content was higher (50.44%). The sample have a higher iron content (Fe_2O_3= 14.4%) and a relatively low aluminum content (Al_2O_3=20.25%). Sample was poor in potassium (K_2O=1.06%) probably due to the relatively low illite content.

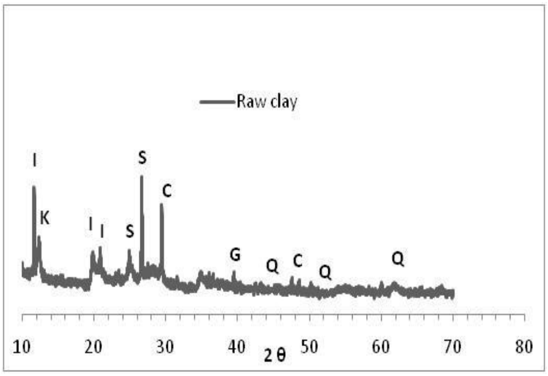


Figure 2. Mineralogical study of raw clay: I: Illite, K: Kaolinite, S: Smectite, G: Geotite, C: Calcite, Q: Quartz, M: Mullite

II.2.3. Thermal analysis

The differential thermal analysis (DTA) and the thermo-gravimetric analysis (DTG) were determined using a differential calorimeter Setaram. A finely ground sample was introduced into a furnace where the temperature was increased to 1000°C with a rate of 10°C/min. The thermal analysis curves for raw clay are presented in Figure 3. The loss in mass was equal to 17%. The curve shows three main endothermic peaks:

- Endothermic peak (P1) at 100°C indicates the evaporation of moisture absorbed and interlayer water.
- Endothermic peak (P2) at 470°C shows the dehydroxylation of kaolinite with traces of illite.
- Endothermic peak (P3) at 640°C corresponds to the decomposition of carbonates.

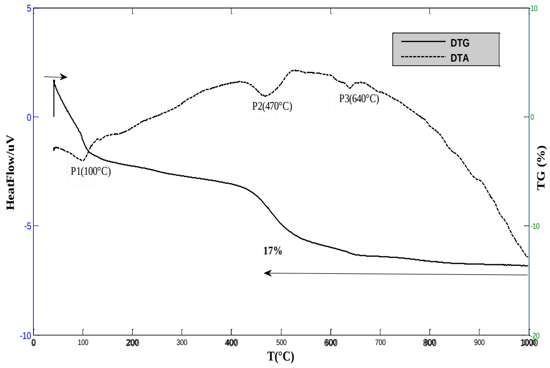


Figure 3. DTA/DTG tests of raw clay

III. Methods

III.1. LWA condition

To manufacture a LWA, we have satisfied a set condition. According to the literature, expanding clays cannot be that beyond some chemical condition which can at high temperatures (1150°C) give a suitable viscosity capable of trapping gases liable to form. The favorable graphical representation of areas to the expansion of chemical composition has been studied by several researchers [12]. They defined limits of chemical composition in a triangular or a quaternary diagram. And according to the chemical composition limits, the raw clay can be use to expanded. It must be made by clay minerals with a high percentage (60% to 80%) and other accessory minerals such as hematite (Fe_2O_3), pyrite (FeS_2), limonite ($5Fe_2O_3$ $2SiO_2$), siderite ($FeCO_3$), dolomite ($CaMg$ $(CO_3)_2$, calcite ($CaCO_3$) and gypsum ($CaSO_4$, $2H_2O$) that do not exceed 10% in total. These minerals promote expansion by helping to release the gas during the temperature raising. The presence of alkali metals and oxides is a necessary condition for the expansion [13].
The clay plasticity determines the conditions for granules preparation and drying. Plastic clay requires a high content to achieve the required state for kneading and molding. Highly plastic clays improve the rheological properties of paste and the mechanical strength of raw particles. Drying aggregates requires special precautions to avoid cracking and spalling (very slow drying time 4-5 hours) [14]. A finer grinding can be achieved [15] shows that for certain minerals such as silica, whose granularity affects the expansion. Cougny (1990) [4] proposed a favorable granulometric limits zone to expansion (Figure 4) and the minerals size affects the expansion stages process [16].

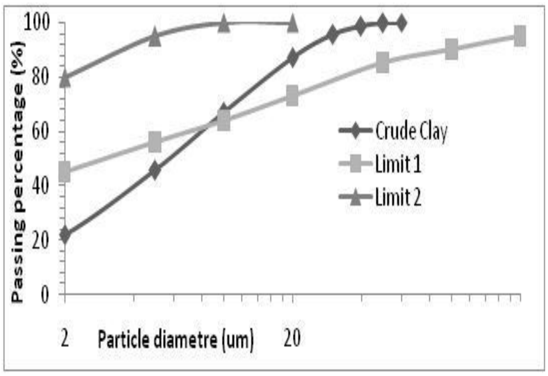


Figure 4. Granulometric limits zone to expansion

III.2. LWA manufacturing

There are three stages of heat treatment: drying, expansion and cooling. In the laboratory, we have used a static oven for heat treatment sets.
First, it is necessary to form grains, ensure water elimination forming slowly to avoid bursting aggregates and causing rapid warming at the end of burning. Precaution is necessary during drying to prevent cracking and spelling of lightweight aggregate. The particles begin to melt and to coalesce to form a stony mass characteristic. The preparation of raw material before heat treatment is to transform the raw clay calibrated and ground to less than 100 microns. Shaping aggregates were realized. The powder is prepared with a humidity of 30% to form rough spherical aggregates. Fragouhis et al used the same procedure for raw material preparation, Greek and Germany clay, with the addition of a combustible material to form spherical beads of different diameters cited by Pioro (2004) [17].
Then, the drying time of clay should be long to lose much of their water content. Drying was done in the open air for a few days. The temperature of preheating aggregate must reach 200°C for two hours and that to reduce the percentage of broken grains and to avoid splintering and cracking aggregates. The clay is very sensitive to cracks formation. So, a slow cooling is applied in order to avoid cracking. Expanded clays are obtained by burning at high temperature (1150°C) of the previously formed granules. Grains coalesce together, forming cohesive particles under the heat effect. It provides hard and brittle materials, chemically and thermally inert. Aggregates in the expansion stage are like balloons whose walls are highly deformable and subjected to injection of gases that originate inside due to various reactions [4]. The chemical reactions produce necessary gas to expansion in the clay pores. Finally the aggregates are screened and stored according to their density and size range.

III.3. LWA properties

III.3.1. Physical characterization of LWA

The porosity is estimated in accordance with ASTM C127-12 standards. The water absorption was followed (NF P 18-554). The percentage expansion (E) aggregates is estimated as the change in diameter after burning: E (%) = 100 $(d_2-d_1) / d_1$ with d_1: diameter of the dry aggregate, d_2: diameter of the aggregate after expansion [18]. The bulk density (g/cm^3) was calculated as the M/V ratio, where M is the weight of aggregates in a container of a volume V, established by the standard norm NF EN 1097-6. The fracture strength of aggregate (N/mm^2) was measured by a static press.
The results were compared with those of commercial lightweight aggregates. An application was carried out to investigate the mechanical behavior and the microstructure of lightweight concrete.

III.3.2. Microstructure study of LWA

The quantitative mineralogical analysis was carried out using information from chemical analyses and from XRD according to each sample. SEM observations were used to compare pore distribution, pore size and shape of LWA produced with more promising technical characteristics.

III.4. Mixes of LWC

The lightweight concrete manufacture was conducted in order to determine a structural lightweight concrete of laboratory expanded clay. Taking into account density and specific absorption coefficient of lightweight aggregate, the main problem was water mixing absorption. Open pores lightweight aggregates will surely adsorb water and affect strength and workability of lightweight concrete. Hence, it must predict an effective water-cement ratio and the aggregates must be pre-wetting for an hour. All aggregates were maintained in a saturated dry surface prior to mixing. Table 3 presents the ordinary (NC) and lightweight (LWC) concrete composition per cubic meter. To determine the workability of fresh concrete, a slump test was performed according to ASTM C 143 standards. For each formulation three cylindrical specimens (Φ11cm * 22 cm) were made and intended for mechanical testing.

Table 3. Concrete composition (Kg/m^3)

	NC	LWC
W/C	0.5	0.5
Ce	350	350
W	175	175
S	695.93	714.74
A or LWA	1176.05	666.37
S/A or S/LWA	0.6	0.61

The specimens were cured in water. After curing, weight and volume of the samples were then measured to determine bulk density. The hardened concrete porosity was measured on cylindrical samples and calculated according to ASTM C642-06 standards. The compressive strength was determined at 3, 28 and 90 days using a universal testing machine (3000KN) in accordance with ASTM C38-C39.

III.5. Microstructure study of LWC

The characterization of the inter-phase zone between aggregate and cement paste in LWC is studied with scanning electron microscopy (SEM). Indeed, the paste-aggregate bonding is dependent on the nature of the external shell of the lightweight aggregate. For the same grain, the external shell thickness can vary along its periphery and there are also variations from one grain to another. So, for each LWC sample, the ITZ are observed. XRD and DTA/DTG analysis are an established method to study the crystal-structural properties of materials in general, as well as to observe the crystal-structural evolution of reactive aggregate. All tests are realized to samples matured for more than 90 days.

IV. Results and discussions

IV.1. Physical characterization of LWA

Table 4 shows the physical properties of expanded clay aggregates of fraction size equal to 4/16 expanded at 1100°C, 1150°C and 1200°C. Under the temperature effect, bulk density of aggregates decreased and they became lighter. Aggregates lost more than 30% of their weight with increasing temperature. The surface became rough and the aggregate percentage of the expansion was increasingly important with temperature. The absorption coefficient at 48 is less than 8% for any temperature which agrees with ACI 318-63. It decreases with increasing temperature due to the pore volume accessible. But observing the aggregates surface state, we notes that at a temperature of 1200°C some aggregates reached the melting temperature. Indeed, their surface states show that they are cracked and broken. Beyond a maximum firing temperature (1150°C), there is no longer any possible improvement of lightweight aggregate properties. We can thus save the energy burnt. Their density decreases with increasing time and burning temperature [15]. We determined the size of different lightweight aggregates and followed the evolution of water absorption coefficient which depends on the network of pores and the aggregates surface. The expansion is due to water and vitrification of mineral results in the formation of more or less spherical granules. They consist of a cover containing gas bubbles. The absorption kinetics is relatively fast in the early hours. The lightweight aggregates absorption is higher than that of conventional aggregates [11].

Table 4. Physical properties of expanded clay aggregates for different temperatures

	1100°C	**1150°C**	**1200°C**
Bulk density (kg/m^3)	1093	840	597
Density (kg/m^3)	2075	1516	1096
Total porosity (%)	42	48	*
Broken aggregates (%)	5.9	24	*
Water absorption W 1hr (%)	1.43	2.6	5.5
Water absorption W 48hr (%)	2.7	3.7	7.9

The physical properties of all size aggregates expanded at 1150°C were determined. Aggregates were studied according to the following granular size fractions in mm: 12.5/16, 10/12.5, 4/10 and 4/16. Table 5 summarizes the different characteristics of expanded clay aggregates: densities, water absorption coefficients w (%), water porosity (total and inter-granular) and expansion percentage (E). According to ACI 318-63 and NF P 18-309, the aggregates must have a bulk density less than 880 kg/m^3. They also require the volumetric coefficient of water absorption at 48 h to be less than 8%. The properties shown in Table 6 suggest that the aggregates prepared in the laboratory at 1150°C are suitable for the production of lightweight concrete. Table 7 shows the results of particle crushing strength for different pellets size. The crushing strength increased as the particle size decreased. During the heat treatment process, the gas was formed inside the aggregates as a result of various decompositions during the burning process. When the gas emerged to the outside and formed a line, the structure became porous [19]. The expansion decreased the grain size being influenced by the voids between the particles [20]. Water absorption increases with the aggregates size thus the accessible pore volume is higher. The size and quantity of the pores has determined the water absorption speed. The porosity is the reason for decreasing the compressive strength of the aggregates. It decreases with increasing aggregate size.

Table 5. Physical properties of all size aggregates expanded at 1150°C

	10/12.5	**4/16**
T (°C)	1150	1150
Bulk Density (g/cm^3)	0.87	0.84
Particle Density (g/cm^3)	1.55	1.516
Particle Density 1 hr immersion (g/cm^3)	1.55	1.518
Density of milled particles (g/cm^3)	1.45	*
W 1 hr (%)	2.80	2.62

W 48 hr (%)	3.76	3.68
Volumetric Water Content (48 hr)	5.83	5.58
inter granular porosity	43.87	44.59
Total Porosity	40.38	41.69
Expansion, E(%)	13.5	15

Table 6. Particle crushing strength for different pellets size

Pellet Diameter (mm)	8	12.5	16
Pellet crushing strength (kN/m^2)	8.34	3.99	3.48

IV.2. Mineralogical and chemical characterization of LWA

The thermal stresses change the composition and cracking of aggregates thereby increasing their internal defects cited by Wasserman (1997) [11]. Chemical and mineralogical (figure 5.) tests on the expanded clay aggregates were performed. Compared the mineralogy curves, it can deduce that changes in amorphous crystalline minerals (kaolinite, illite) occurred during the transformation process.

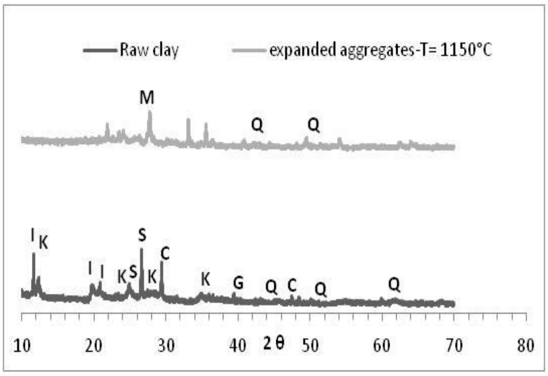


Figure 5. Mineralogy study of expanded clay aggregates: I: Illite, K: Kaolinite, S: Smectite, G: Geotite, C: Calcite, Q: Quartz, M: Mullite

IV.3. SEM analysis of lightweight aggregates (LWA)

A SEM study was conducted to show the temperature effect on the texture of aggregate's external and internal shell (figure 6.). The surface appearance varies from a more to a less smooth, dense texture and glazed to a rough and porous inside aggregates. The aggregates color varies from red brick for low temperature, dark red to 1150°C until brown at 1200°C. Some aggregates burnt to 1150°C have reddish to brown exterior and interior. The gloss in surface is due to formation of a glassy layer on the outer surface of aggregates. The temperature increase is accompanied by an increase in pore size and thinning of the walls. The average pore size at a temperature below 1100°C and between 10 and 600 microns such that the outer surface has low porosity. At a temperature exceeding 1150°C, the pore size varies from 2 to 30 microns for small pore and to 100-300 microns for the largest pores. The size of largest pores was observed for aggregates expanded to 1200°C. The internal porosity of expanded aggregates shows a heterogeneous uniform pore distribution. The number of pores has a distinct influence on the concrete strength. The pores increase will induce a decrease in the concrete strength [21].

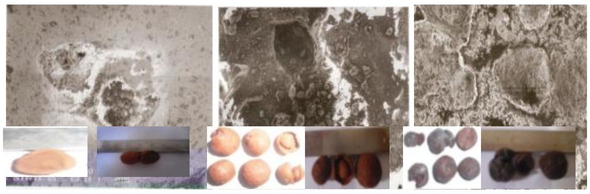


Figure.6. SEM results on expanded clay aggregates (x262) at 1100°C (left), 1150°C (center) and 1200°C(right)

IV.4. Comparison of the LWA laboratory produced and commercials

Industry aggregates such as Argex, Terreal, Granulex, Leca and Agral are compared to those manufactured in laboratory. The data collected from literature was summarized in Table 7. which includes a set of physical properties. This laboratory manufactured aggregates properties seem to be comparable with others. Their properties are slightly different from those obtained in this study due to the change in grain size and the manufacturing method. Then, manufactured lightweight aggregates may be used for different applications.

Table 7. Comparison LWA of the laboratory produced and commercials

Name	**Bulk density (kg/m^3)**	**Dens------ity (kg/m^3)**	**Inter-granular Porosity (%)**
Argex	560	921	39.20
Terréal	455	737	38.26
Granulex	729	1434	49.16
Laboratory Aggregates	840	1516	44.59

Leca	518	*	*
Agral	676	*	*

IV.5. Study of LWC

IV.5.1. Compressive strength

The concrete density decreased from 2375 Kg/m^3 for ordinary concrete (NC) to 1978 kg/m^3 for lightweight concrete (LWC) with expanded aggregates clay formed in laboratory and expanded at 1150°C which confirms lightness. The mechanical strength increases with curing age. It increased from 10.88 MPa at the age of 3 days up to 17.89 Mpa at the age of 28 days for LWC (table 8.). It can consider that the compressive strength of lightweight concrete has lightweight conforms to its required at the age of 28 days by ACI guide (318-63) and AFNOR norm (NF P 18 – 309). Many studies can be found on the mechanical properties of lightweight concrete aggregates in polystyrene [22], [23], wood chips, expanded clay aggregates [9] and [15]. Unlike regular concrete, lightweight concrete generally fails with a break inside the lightweight aggregate and the boundary between aggregates and paste [12] and [24].

Table 8. Mechanical study of lightweight (LWC) and normal concrete (NC)

		NC	LWC
Density oven dry (Kg/m^3)		2365	1978
Water Porosity (%)		13.3	20.68
Compressive strength (MPa)	**3**	11.73	10.88
	28	29.19	17.89
	90	37.23	23.45

The effect of lightweight aggregate on the mechanical properties seems to depend on the type of aggregate. In addition, Laukaitis et al have shown, in the case of polystyrene aggregates, dependent on the characteristics of both the aggregates size and shape. The high porosity and low mechanical properties of lightweight aggregates may explain the low strength of lightweight concrete [22]. It has a lower compressive strength than ordinary concrete for the large inter and intra granular porosity and the large absorption coefficient. The aggregates strength normally varies according to the production process and the hull structure. It seems that lightweight concrete strength is not directly related to the aggregates size but to the aggregates density. It was directly proportional to crushing strength and overall density [21]. According to Tommy et al, concrete strength depends on the used aggregates strength, the aggregate size and the density. The aggregates are the fragile component of lightweight concrete but not the matrix or the interaction zone. These define the mechanical behavior of concrete [19]. The compressive strength and the absorption coefficient of the paste are interdependent and their effects are not easily separable [12].

IV.5.2. Microstructure study of LWC

The concrete strength can be influenced by the aggregate strength, the paste and also the pores and the pozzolanic reactivity of the outer texture. Several authors have noted the existence of two chemical processes. The first is a pozzolanic reactivity that occurs between the aggregate and the cement which penetrated into the granules pores (CSH formation). The second is pozzolanic reactivity causing a deposition of Portlandite (CH) in surface pores of aggregates. We evaluated the pozzolanic reaction by XRD (figure 7.) where it was found that the Portlandites peaks are less intense and less wide for lightweight concrete. The water inside aggregates contributed to internal curing and cement hydration in the concrete which would probably be higher compared to that of normal concrete. The SEM samples observation (figure 8.) and the DTA/DTG test (figure 9.) are used to make out the hydrated amorphous phases. It indicates that the lightweight concrete is the least anhydrates particles. Thermogravimetry continuously measures the mass of a sample subjected to a steady increase of temperature in order to quantify reactions involving gaseous emissions.
Thermogravimetric analysis was used to determine the molar amount of Portlandites due to decomposition of chemically bound water between 510°C and 550°C and amount hydrate (CSH). The identification of the main hydrated phases and gas can be made from their dehydration or decomposition. It is interesting to note that the hydration reaction of the main cement, which is the calcium silicate hydration CSH gel and calcium hydroxide form, can be easily followed by the DTG peaks. Weight loss can be obtained from the DTA curves that in turn allow the quantification of phases. Very small peaks for CSH dehydration between 275° C showing that calcium silicate hydration have started. A small peak of the decomposition of calcium hydroxide occurring between 520°C because of CO_2 released during the decomposition of calcium carbonate [25] and [26].

Cement hydration was increased to reduce the capillary porosity and to increase the tortuosity of the pores in the cement paste which reduces the absorbency of lightweight concrete [27]. A porous material can be made as long as pores are not interconnected (closed pores). Although lightweight aggregate (LWA) are more porous than other. The lightweight concrete absorption with porous lightweight aggregate is actually low and comparable to that of normal concrete. This was attributed to a combination of interfacial transition zone and a more homogeneous microstructure. Lightweight aggregate with higher absorption capacity showed a very thin transition zone ITZ (figure 8.). The zone thickness appears to decrease linearly with the aggregate absorption capacity [28]. The adhesion of the lightweight aggregates to cement paste depends primarily on the characteristic pore size of the aggregate surface [29]. The interfacial zone size of lightweight concrete is smaller than normal weight concrete [21].

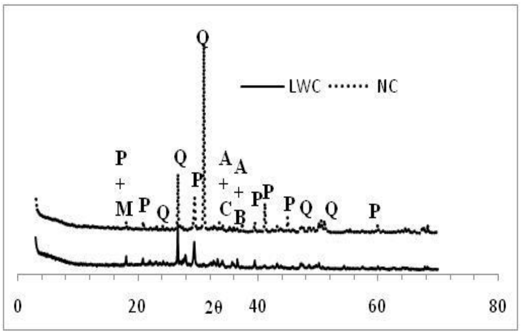


Figure 7. XRD of lightweight and normal concrete, P: Portlandite, A: Alite, B: Belite, Q: Quartz, M: AFM, C: Calcite

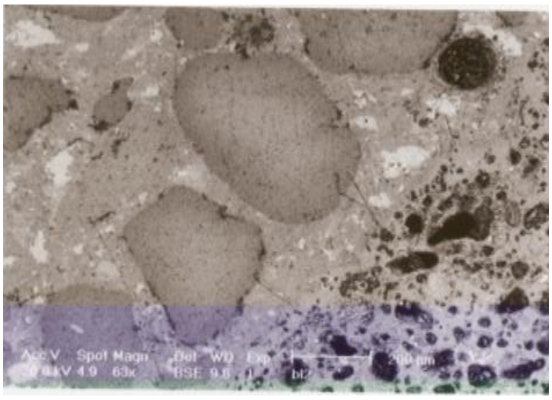
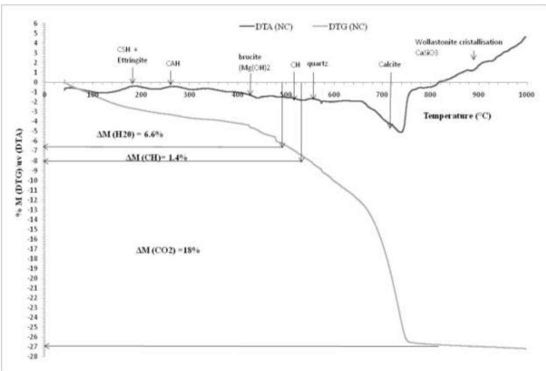
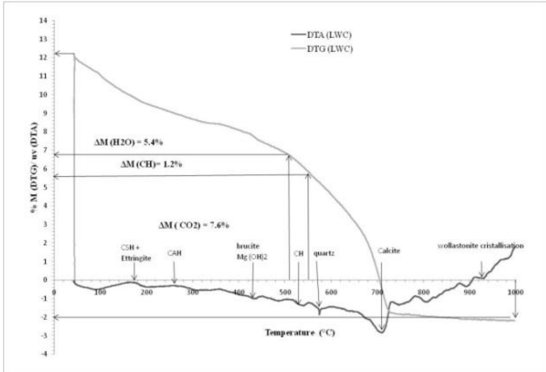


Figure 8. SEM of LWC (x63)



(a)



(b)

Figure 9. DTA / DTG of normal (a) and lightweight (b) concrete

V. Conclusion

The objective of this study was to beneficially use Tunisian clay to manufacture in laboratory an expanded clay aggregates for lightweight concrete. We characterized the physical, chemical and mineralogical properties of the raw and burnt clay. Expanded clay aggregates were produced whose properties were comparable to others commercially available ones. It has generated value for Tunisian clay by manufacturing expanded clay aggregates. These aggregates were used to produce a lightweight concrete with acceptable compressive strength in accordance with norms.

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