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Research Article

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Laboratory Firing Simulation for the Study Effect of Fire on Monumental Limestone, North Chapel in Tihna El-Gabal, Minia, Egypt Case Study

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Abstract Although natural stones are non-combustible materials, the fire and heat effect can cause irreversible changes in their structure and mechanical properties, which influence the strength and static behavior of the stone structures. Therefore, this study aims to understand the effect of gradual increase in firing temperatures on some properties of studied monumental limestone in the selected study area. The recent research presents an experimental laboratory study of the effect of fire and high temperatures on limestone. The Roman Chapel in Tihna El-Gabal, Minia, Egypt selected carefully as a case study for achievement aim of the recent research. As firing temperature increased from 250 °C to 1000°C some changes occur in natural studied limestone. These changes vary according to the values of the temperature exposure. The obtained results showed some of studied limestone characterization are significantly but gradually have been affected by change firing temperature. It involves color, textural relation of grains, morphological and their physico-mechanical properties. Moreover, decarbonatization or calcination reaction, plays an important role in explanation the mechanism of changes in the physico- mechanical average values under high temperature. The physical explanation of the obtained results is formation of thermal micro-cracks led to weakness in the mechanical properties of the studied limestone and finally collapsed completely to the extent that they became unsuitable for measurement.

Keywords North Chapel - Akoris (Tihna El-Gabal) - Calcination - Limestone - Firing

Introduction

Stone is an essential component of most historic building. Stone is, however, far from being an immutable material, as it can undergo severe disruption due to various decay agents [1]. Particularly, the stonework with a lime basis (limestone) is very sensitive to many of deterioration and weathering factors such as soling, pollution, oxidation, air temperature and relative humidity especially in industrial environment [2]. Research into fire damage in historic buildings is often biased towards obviously sensitive materials, such as wood and textiles. However, stone is not immune to fire damage and can experience severe decay during, and as a consequence of, fire [1].

Fire is one of the most major hazards to stone buildings due to the amount of irreversible loss and changes in their main formation and consequently the mechanical and physical properties that it produces. The consequence of fire damage is greater in historic buildings where material loss is compounded by the loss of historical evidence and artistic values of cultural heritage [3-5].

Fire stands out among decay agents because it generates irreversible decay of stone, with long-lasting effects, in a very short period of time. Studying the impact of fire on stone is therefore of great importance [1,6].

This research presents an experimental laboratory study of the effect of fire and gradual increasing of firing temperatures on the different characterization of limestone. This experimental study was conducted on limestone representative samples from Tihna El-Gabal archaeological area, Minia, Egypt. This archaeological area was specifically selected as it subjected to firing occurred several years ago in one of its monuments, which is called the North Chapel.

The site of Akoris (Tihna El-Gabal) is situated 230 km south of Cairo on the east bank of the Nile, 12 km north of El-Minia [7,8]. North Chapel (or Roman Chapel) is a rock-cut chapellocated in the north part of the archaeological site in Tihna El-Gabal, north the modern Christian Cemetery [7]. Because of the proximity of modern residential buildings to this Northern temple, it was exposed several years ago to a large fire inside it, which led to great damage to its walls, ceiling, and the mural paintings on its walls, figures (1,2). As a result of the firing process weakness of the mechanical properties of forming limestone was occurred as well as chromatic changes evident in the surface of it.

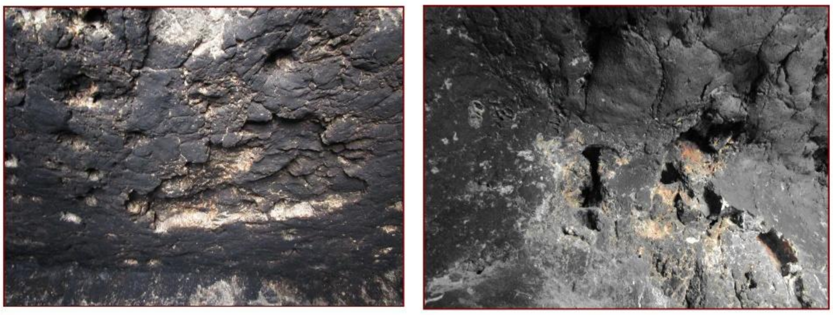


Figure 1: The fallen parts of the ceiling and the top of the walls in the inner chamber of the North Chapel

Figure 2: Change of color; one of the most common deterioration patterns of fire damage in the North Chapel

2. Materials

Studied samples

Representative limestone samples were carefully chosen from the archaeological studied area to be agreed with the next preparation processes, in addition to a sample from the North Chapel in the studied area to be examined under the Polarizing microscope. Studied experimental samples used were prepared on cubes forms of 5×5×5 cm for studying their physio-mechanial properties according to different documented standard test methods.

3. Methods and techniques

Different analytical and investigation methods were used to study the experimental samples:

3.1. Polarizing microscope

Petrographical investigation conducted for the studied samples according to [9] using the polarizing microscope (Olympus BX50, Japan) associated with computer software imaging system called (analysis) in the geology and petrographical investigation laboratory of National Housing and Building Research Centre (HBRC), Cairo. It aims to differentiate between the micro- texture of standard and fired limestone.

3.2. XRD (Mineralogical analysis)

X-ray diffraction used for identification of the mineralogical composition of the different studiedreference and fired samples, using X-ray model X' Pert Pro Phillips MPD PW 3050/60 X-ray diffractometer in the XRD lab of the HBRC.

3.3. Scanning electron microscope (SEM)

Scanning electron microscope, (JEOL JSM-5400LV) at Assiut University, was used to determine the morphology of the particles, voids and to follow the effect of different firing temperatures on morphology of studied samples.

Moreover, the physico-mechanical properties (bulk density "B", water absorption "WA", apparent porosity "AP", and compressive strength "C") were measured for the studied samples based on documented standard typical methods for both of standard and fired limestone samples at varied firing temperatures [10-12].

3.4. Artificial Firing process

For the simulation of the effect of fire,the experimental samples were exposed to different degrees of temperature were selected. The highest temperature used in the study was determined as 1000°C which is the highest temperature expected in a fire incident and for the alteration of natural stones. Samples were exposed to the heat in an electric oven separately starting from 250°C, gradually 350°C, 450°C, 550°C, 650°C, 750°C, and 1000°C for 2 hours for each temperature and then compared with reference sample at room temperature according to the degree of temperature. The specific temperature was gradually attained after placing the samples in the electric oven. After attaining the desired temperature,samples were stored in the oven for 2 h in order to let the physical and mechanical changes take place at the required temperatures. Studied samples exposed to the high temperatures were cooled down to room temperature inside the oven to avoid thermal shock.An example of placing the samples in the electric oven is given in figure (3).

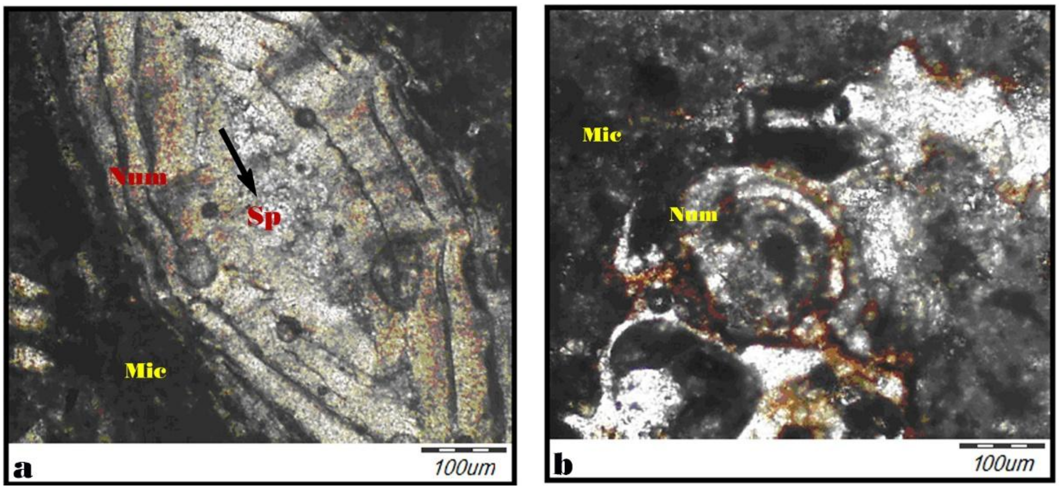
Figure 3: Some of the studied samples inside the electric oven.

The study strategy in this paper is based on the assessment of changes in the properties of the studied limestone samples after exposure to fire at different temperatures compared to the standard sample. It includes an assessment of the physico-mechanical parameters and petrographic investigation. Furthermore, it includes color change observations and weight loss of the studied samples after firing at different firing studied temperatures. It should be noted that weight loss ΔM% in the current study calculated with the same manner that used in salt crystallization test [13].

4. Results

4.1. Polarizing microscope investigations of the studied samples

Petrographical investigation of the studied samplesfigure (4a and b) revealed that, the effect of firing reflected on the studied micro-texture. As it can be noticed the presence of deformation in whole fired studied limestone microscopic texture evidenced by change in the main color of their fabric to be more reddish brown than usual, figure (4b). Moreover, loosing appreciate of microsparitic cement content that plays important role as binder between forming grains with its matrix. Deformation extends to involve bioclasts as most detected fossils have been deformed such as *Nummlites* which lost its sparitic core regardless presence of its original outlines.



Mic: Micritic calite , Num: Nummlites, Sp:Sparite

Figure 4: Photomicrographs of the studied limestone: a) standard sample; b) fired sample

4.2. Mineralogical Analysis by (XRD) of the studied samples

The (XRD)analysis indicated that all of studied samples both of standard sample and fired are consist mainly of calcite ($CaCO_3$), as shown in figure (5). On the other hand, the analysis exhibited presence of appreciate lime CaO in fired samples at 1000°C, and also traces of dolomite ferroan$Ca(Mg,Fe)(CO_3)_2$ in samples fired at 350°C, figure (5).

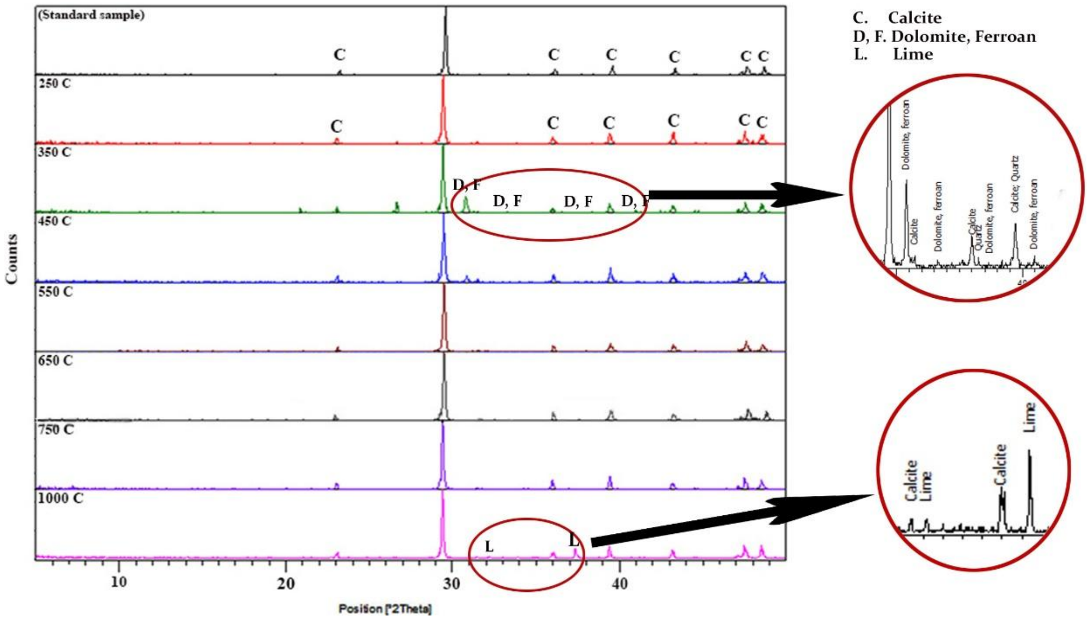


Figure 5: X-ray diffraction pattern of the reference sample and studied samples at different firing temperatures

4.3. Visual observations and weight change of the studied samples after firing process).

It is known that color change is considered to be non-reversible and therefore damage can be significant [15, 16]**.** Usually the color change is used as a guide for determination the firing temperature that fired limestone have been subjected to it.

Experimentally, in the current study limestone exhibited the following color changes and weight loss by subject to different studied firing temperatures minimized by 250°C and maximized by 1000°C as shown in table (1) and illustrated in figure (6).

Generally, discoloration started as slight change in color from yellowish white to light grey at 250°C passing through reddish pink at 350°C and grey from 450:750 °C reached to snow white powder for the studied samples that were burned at 1000°C.

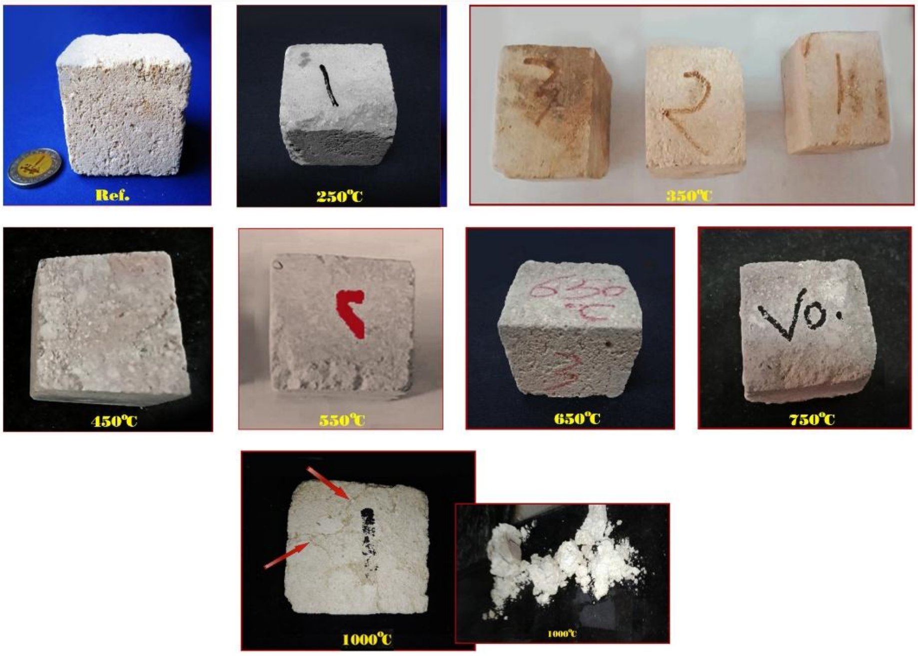


Figure 6: Effect of different firing temperatures on appearance of the studied samples compared to the standard one

On the other hand, there are notable weight change at different studied firing temperatures. The weight loss ΔM% of the samples under investigations was calculated after each firing temperatures. The recorded decreasing in weight shown in table (1). And revealed that: burned samples at 250°C, 350°C, 450°C, and 550°C (4.2%), while the rate of weight loss was significant in the burned samples at 650°C and 750°C (8.3%). On the other hand, the burned samples at 1000 became unsuitable for measurement or it called undetectable as limestone studied samples convert to Powder case.

Table 1: The Color change and weight loss of the studied samples under different firing temperature

Firing temperature °C	**250°C**	**350°C**	**450°C**	**550°C**	**650°C**	**750°C**	**1000°C**
Color change	Light gray discoloration	Pink	grey	grey	grey	grey	Snow-white
Percentage of weight loss (ΔM %)	-4.2	-4.2	-4.2	-4.2	-8.3	-8.3	Undetectable Powder case

4.4. Physico-mechanical properties of the studied samples

The changes in the physico-mechanical properties of the studied samples caused by firing processes are monitored through comparing measurements of these major properties of the studied samples before (standard) and after the firing. These studied changes in the average values of different physio-mechanical are summarized in table (2), and illustrated in figure (7).

The obtained results exhibited that the bulk density average values for the studied limestone samples which were fired at the selected temperatures from 250°C up to 750°C, showed slight change by gradual decreasing with increasing the firing temperature from 2.01(g/cm^3) for standard unfired limestone to 1.8(g/cm^3) for studied limestone fired at 750°C. Whereas samples that were burned at 1000°C, also collapsed completely and became unsuitable for measurement, table (2) and figure (7a). It is noticed also from the obtained results that both studied hygroscopic properties (water absorption and apparent porosity) in the current study increased gradually with firing temperature increased from 10.9% for unfired standard samples to 14.5% for fired samples at 750°C, it is also associated with increase in the average values of apparent porosity from 21.9% for unfired standard samples to 26% for fired samples at 750°Cas seen in table (2) and the graphs in figure (7b,c).

Uniaxial compressive strength average values of the studied limestone are regarded with increasing firing temperature from 250 °C to 750°C as mention. This regression in average values of uniaxial compressive strength from (85 Kg/cm^2) for unfired standard samples70 Kg/cm^{2}for fired samples at 750°C as shown table (2) and figure (7d).

Table 2: The changes in the physico-mechanical properties average values of the studied limestone samples after different firing temperature compared to the standard sample

Properties / Samples	Bulk density (g/cm^3)	Water absorption (%)	Apparent porosity (%)	Uniaxial compressive strength (Kg/cm^2)
(Standard sample)	2.01	10.9	21.9	85
250ºC	2.01	12.3	24.7	85
350ºC	1.9	12.8	24.3	80
450ºC	1.9	12.8	24.3	80
550ºC	1.9	14	25.6	72
650ºC	1.8	14.5	26	70
750ºC	1.8	14.5	26	70
1000ºC	Undetectable	Undetectable	Undetectable	Undetectable

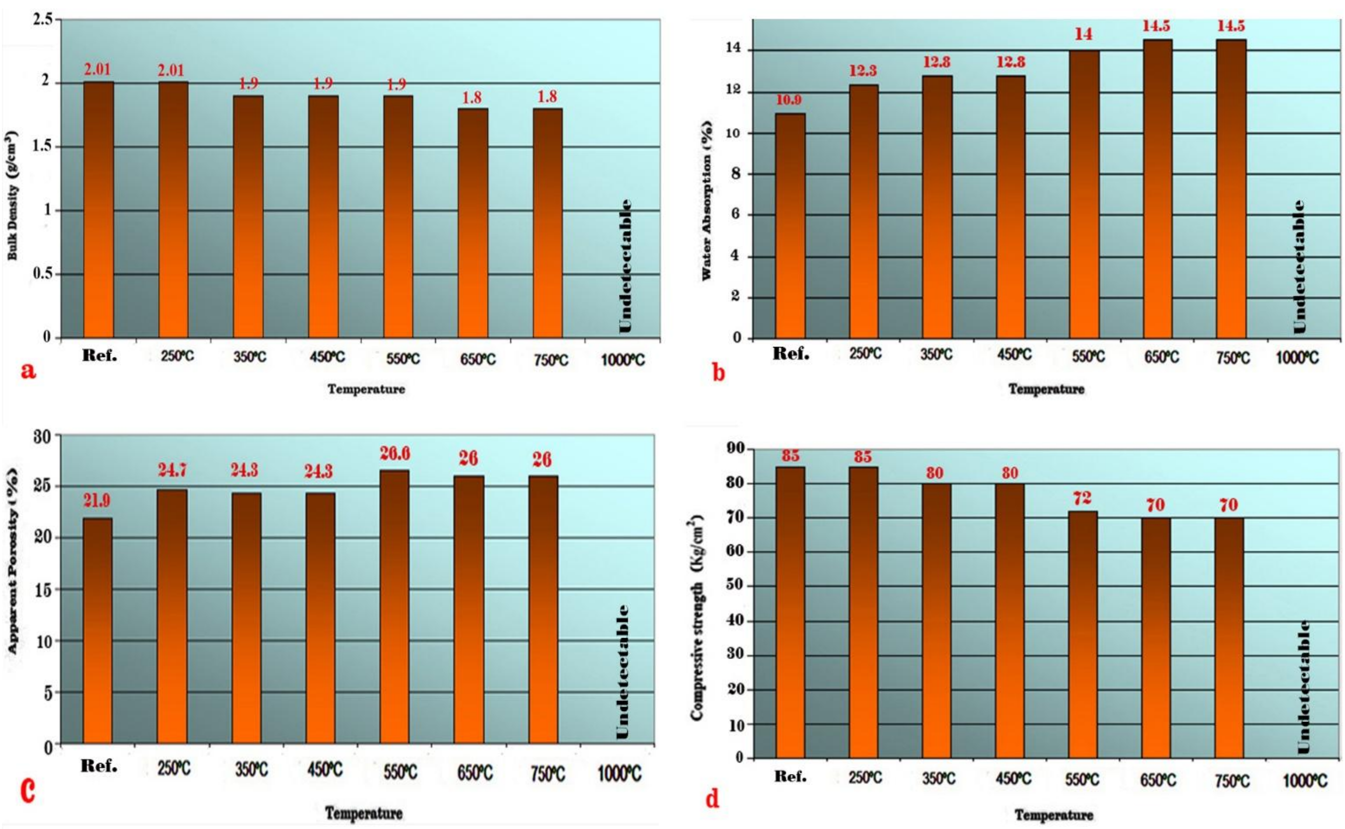
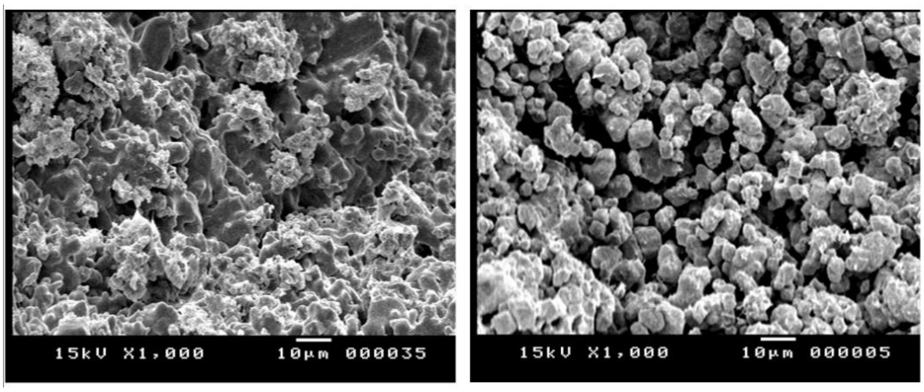


Figure 7: Physico-mechanical measurements of the studied standard and fired samples: (a) bulk density, (b) water absorption, (c) apparent porosity, and (d) compressive strength.

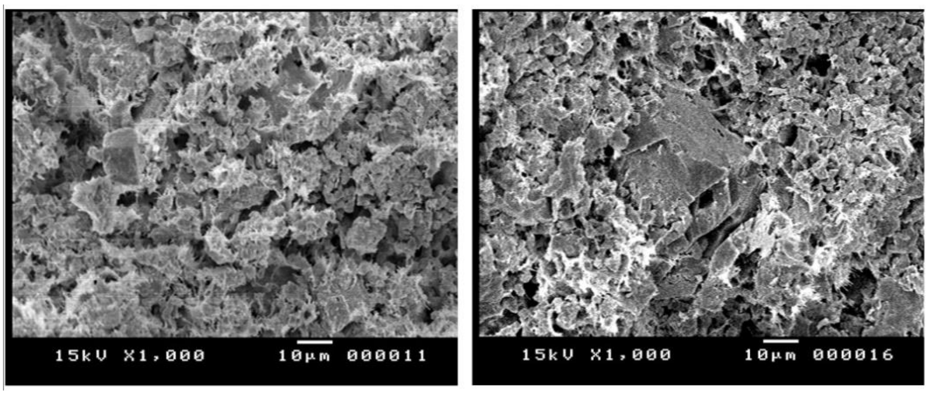
4.5. SEM investigations of the studied samples

The influence of increasing suggested firing temperature in the recent research required usage of SEM as investigation method to follow the gradual increase of firing temperature on the studied limestone as shown in figures (8-10). The obtained results showed the following:

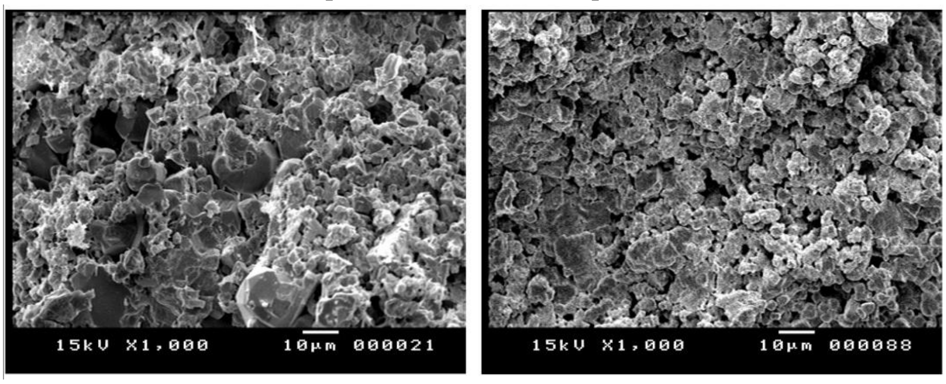
- All of the studied fired limestone samples showed increase on its grain size compared to the standard unfired limestone.
- The increase in grain size of the studied fired limestone in a direct relation with increasing of studied firing temperature from 250ºC to 750ºC.
- The studied fired limestone sample at 750ºC showed a relative increase in grain size associated with slightly removing of the binder material figure (9). On the other hand, the fired sample at 1000ºC showed leaching for cementation material which represented the binder material, figure (10).



Standard unfired sample **Fired sample at 250 ºC**



Fired sample at 350 ºC Fired sample at 450 ºC



Fired sample at 550 ºC Fired sample at 750 ºC

Figure 9: SEM photomicrographs show a slightly gradual increase in grain size of the different fired limestone samples under different suggested firing temperature beginning from 250ºC to 750ºC (referenced by standard unfired sample).

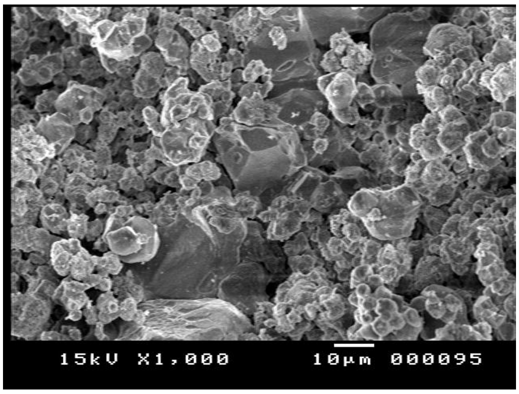


Figure 10: SEM photomicrograph of the fired limestone sample at 750ºC show a relative increase in grain size, and slightly removing of the binder material associated with increase in porosity.

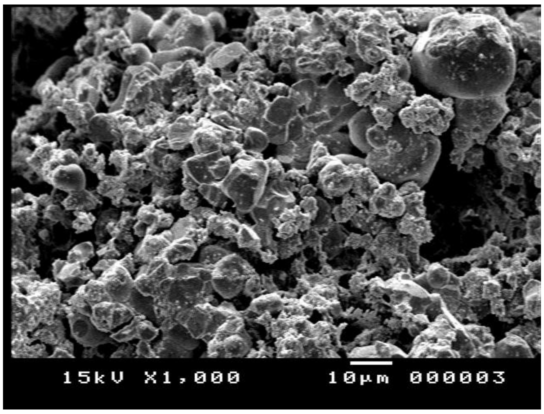


Figure 11: SEM photomicrograph of the fired sample at 1000ºC shows leaching for cementation material which represented the binder material, leads to increase the porosity, and finally converted to a powdery state.

5. Discussion

The natural color of limestone is related to the mineral composition [15]. As noticed the studied fired limestone samples at 350°Cexhibited reddish pink to reddish brown discoloration as a result of formation dolomite ferron phase according to its XRD pattern figure (5).This dolomite ferron contains relative enrichment percentage of iron and magnesium that plays role in converting the original color of the standard studied limestone to this reddish pink one. The previous discoloration is agreed with [15].

According to [16], temperature changes can induce micro-cracks in the rocks due to the mismatch in the thermal properties of the different mineral compositions (intergranular) or within grains (intragranular).Consequently, weight loss and other different physico-mechanical properties were affected by formation of such thermal micro-cracks.

Physico-mechanical properties. The results proved that the studied fired limestone samples show not significant changes in the physio-mechanical properties at lower temperatures, but at higher temperatures, specific mineral reactions, such as decarbonatization or calcination, are directly evidenced by sudden jumps in thermal expansion curves [17,18]. The Density of the samples and their uniaxial compressive strength were observed to decrease with increasing temperatures exposure over the 2 hour period for each temperature separately.

Density values did not change significantly with the temperature up to 750°C. This agreed with [19] they stated that the real density does not show significant changes after heating. This hypothesis appears realistic since the rock is basically made up of pure calcium carbonate, and the heating temperatures are much lower than the carbonate dissociation temperature (900°C) [19]. As for the samples that were fired at 1000, they collapsed completely.

There was no significant reduction in strength when the samples were heated up to 350°C but the decrease in the strength of the stone began to increase after 350°C. The decrease in mechanical properties can be attributed to the opening of micro-cracks consequent to the stress induced by calcite anisotropic thermal deformation during heating. This is confirmed by the increase in porosity detected for increasing heating temperatures [20]. Also, according to [21] the increase in porosity of samples explains the decrease of compressive strength very well.

In the current study, the most catastrophic change occurred in studied limestone cores, beganto take place above 750ºC due to calcinations processes. At 1000ºC firing temperature, samples were found undestroyed at the end of the test, but the samples crumbled after some hours exposed to air as a result of the volume increment produced by the reaction of CaO with air moisture to form $Ca(OH)_2$ (portlandite) as shown in figure (6), this is what agreed with XRD analysis, and which was confirmed by other researchers in similar cases as a result of high-temperature testing of stones containing calcite [21-23].According to [21,24], calcareous stones undergo severe processes of physical destruction in zones affected by fire above 800°C due to the calcination of calcite, moreover, the vapor pressure due to temperature rise inside the specimens causes micro and visible cracks which in turn reduce the compressive strength. According to [19], limestone becomes quicklime (CaO or CaO + MgO) by calcination in 900–1000°C oven. Quicklime is very active against water. In this case, the following reactions occur:

$$CaCO_3 + HEAT \rightarrow CaO + CO_2\uparrow$$

$$CaO + H_2O \rightarrow Ca(OH)_2 + HEAT$$

When quicklime reacts with water it releases heat and the fast-rising temperature in the beginning becomes constant towards the end of the burn out. This heat causes the fragmentation of unslaked lime until it becomes dust by major thermal internal tensions in the lime particles [18].

On the other hand, all the studied samples experienced an increase in porosity and water absorption, as a consequence of the anisotropic thermal deformation of calcite crystals as shown in SEM photomicrographs and capillary cracks occurring due to heat. There are obviousporosity increases seen starting from 250°C up to 750°C. As for the samples that were burned at 1000, they had a complete collapse. In addition, according to [20], the increases in porosity lead to increases in water absorption.

After heating, the morphological features of these samples were highly affected and experienced an increase in porosity and water absorption, as a consequence of the anisotropic thermal deformation of calcite crystals, as shown in figures (8-10).

Conclusions

From the results obtained in this study, the conclusions includethe following:

- By application thermal durability by firing under some suggested firing temperature from 250 :1000 °C on selected monumental limestone of Tihna El-Gabal some of its properties and characterization are significantly but gradually affected involve color, textural relation of grains ,morphological and their physico-mechanical properties.
- High temperatures cause micro-cracks cracks and weakness in the mechanical properties of the stone.
- At higher temperatures, specific mineral reactions, such as decarbonatization or calcination, are directly evidenced by sudden jumps in thermal expansion curves play an important role in explanation the mechanism of changes in the physico-mechanical average values under high temperature.
- Calcite turns into portlandite at 1000°C. This conversion leads to changing of structural and textural properties of natural building stone.
- Increase in the temperature causes decreases in the weight of bulk density caused.
- Increase in the amount of absorbed water by increasing temperature can lead tostructural demolitions. Therefore, safety measurements should be taken while exposing to water when a fire has been detected.
- Fire and high temperatures cause changes in the inner structure and in the mineral composition of natural stones. Some mineral transformations cause a volumetric increase, or different thermal expansions initiate cracks in the stone. This effect can be responsible for the increase in porosity and the decrease in strength in some cases.

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