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Characterization of Building Materials and Decay Hazards of the Discovered Archaeological Structures at Tell El-Hiṣn Site, Heliopolis, Egypt

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ABSTRACT

This paper focuses on characterizing the primary building materials and deterioration agents of the discovered archaeological structures at Tell El-Hisn, Heliopolis, Egypt. To achieve the aforementioned purpose, building materials were studied using a polarized light microscope, scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX), X-ray diffraction (XRD) as well as visual observation, and determination of some physical and mechanical properties. The results revealed that the studied limestone consists of calcite as the main component, quartz as a minor constituent and halite. The collected crust samples from degraded surfaces provide clear evidence of halite, gypsum, nitratine, and nitrocalcite as salt crystals, which fill the pores and coat the stone surface. The studied mud brick consists mainly of kaolinite, montmorillonite, albite, quartz and halite. After discovery, limestone and mud brick structures exhibited considerable damage related to influences of the burial environment and surrounding external environmental conditions. So, the author recommended two strategies: the first concentrates on prevention and protection procedures against the impact of the prevailing deterioration factors. The second strategy encompasses a range of restoration and conservation procedures for the discovered structures.

1. INTRODUCTION

The archaeological site of Tell El-Hiṣn is located north of El-Matariya, nearly 13 km northeast of Cairo Centre, at (30 °05N, 31 °20E) (Figure 1) (Saleh, 1981, 4). It is a part of the Ancient Egyptian city of Iunu (biblical) and (Coptic On), the capital of the 13th region of Lower Egypt, which was named "Heliopolis" in the Greek era. The city gained wide fame throughout ancient Egyptian history and the Greek era, given that it was a major centre for sun worship. An Italian Mission conducted the first excavation of the site from 1903 to 1906 (Dietze and Ugliano, 2022, 1-22). Further excavations took place in 1962 – 1972 by Mutawa Balboush. Subsequently, the archaeology faculty at Cairo University carried out excavations for several seasons from 1976 to 1994, led by Abd Elaziz Saleh. Following this, Alaa Shaheen and Mohamed Salah El-Kholi continued the work from 2006 to 2011. Since 2012, a mission from Ain Shams University, led by Mamdouh Eldamaty, has worked at the site (Saleh, 1983, 63-64; Rawash., 2019, 36- 57). The site is estimated to measure some 1,000 by 900 meters. Unfortunately, the site's architectural history and exact topography are unclear (Bains and Malek, 1980). Excavations at this site have revealed several archaeological structures and artworks that have detailed religious and symbolic scenes dating back to the 19th and 20th Dynasties, specifically from "Ramesses II" to "Ramesses IX". These included remnants of double adobe walls that likely enclosed the town or the main temple. Furthermore, remnants of adobe buildings, sections of the main temple's walls and columns, two stone gates of "Ramesses IX", the chapel of Neb Maat Ra, which still retains colour remnants, the remains of four stone pedestals of the colossal statues of "Ramesses II", some stone parts of royal statues, and remains of stone columns (standing and fallen, in addition, to a stone altar and stone mills dating from the Late Period (Saleh, 1981; Saleh, 1983; Rawash., 2019). Limestone and mud brick are the main building materials used in the archaeological structures discovered at Tell El Hiṣn. Mud brick has been widely used as a construction material in ancient Egypt since the Pre-Dynastic era due to the availability of the raw materials necessary for the manufacture and its low cost (Garcia-Vera and Lanzon., 2018, 708-717). It is a composite building material consisting of clay minerals, which act as a binder and various components such as sand, rock fragments, gritting red brick, animal dung and organic fibres (chopped straw). Each of these materials possesses special attributes, deterioration problems and patterns that result from their usage (EL-Gohary, 2012, 67-78; Elert et al., 2015, 461-469; Michalopoulou et al., 2020, 221-242). Limestone has been widely used in Egyptian architecture since the Pharaonic era until the present day, possibly owing to its availability, aesthetic appearance and durability (Tawfik., 2015, 136-151; Al-Omary et al., 2018, 35-43). Most of Egypt's ancient limestone quarries are located in the hills and escarpments surrounding the Nile Valley from Esna to Cairo (Lucas and Harris., 2012, 52-55). Furthermore, quarries are situated along the northern coast of the Mediterranean and in Suez. The most common limestone quarries used in ancient Egypt were Saqqara, Giza, Abu Rawash, Tura-Ma'sara, Mokattam, Tel El Amarna, Qurna and Max (Aston et al., 2000, 40-42; Klemm, and Klemm 2001, 631-642; Hefni, 2023, 293-309). Limestone mainly consists of calcium carbonate; owing to its physical and chemical properties, it is susceptible to aggressive destruction and deterioration caused by the influence of various deterioration factors, especially in external environmental conditions (Abd-Elkareem et al. 2017, 535-552; Emine, 2018, 1-6; Papalardo et al., 2022, 2594-2614).

The archaeological structures (limestone and mud brick) in the studied site were highly affected prior to their discovery by chemical and mechanical weathering processes associated with the ground water-saturated burial environment. Moreover, aggressive deterioration factors related to the surrounding environment affected them after their discovery. The burial environment and its major damage factors create a new microclimate, which may differ significantly from the conditions prior to burial. Assuming these conditions are relatively constant, the archaeological structures will undergo a process of adaptation and modification to establish a stable relationship or equilibrium with the new environment. Consequently, the rate of change will diminish and ultimately cease once equilibrium is attained. This stability will persist as long as the structures remain embedded in the ground. Unfortunately, the process of deterioration and change will resume as soon as the structures are discovered and suddenly exposed to novel environmental conditions. These processes are related to burial environment characteristics, building materials' physical and chemical properties, and damage factors in the new surrounding environment. That deterioration may be perceptible within a brief span of time (Bader and Al-Gharib., 2013, 25-42). Groundwater represents the most serious threat to the studied site and their discovered archaeological structures. This is primarily due to the close association between groundwater and chemical weathering caused by atmospheric pollution gases. In addition, it contributes to salt weathering in the case of the change in water content caused by drought conditions resulting from high temperatures and winds. It is also responsible for biological and microbiological damage (Abd El-Hady and Abd El Hafez, 2012, 103-107; Yeşil, 2021, 363-387; Zaremba et al., 2021, 67-86; Germinario & Oguchi, 2021, 85-93). Unfortunately, from the normative standpoint, the archaeological studied site and their

discoveries are perceived as anomalies within their urban environs, which exist within a profoundly conflicted and segregated modern landscape. This accentuates the challenges the site faces in an aggressive and detrimental manner. Furthermore, there is a lack or unavailability of studies or reports that provide evidence of the restoration work that may have been carried out for the discovered archaeological structures at the site. It is worth noting that dealing with unfinished archaeological excavation sites and their archaeological discoveries, such as the site of Tell El-Hiṣn, are very difficult and pose significant challenges in terms of slowing down or even effectively preventing deterioration of archaeological building materials when considering various conservation approaches.
From the above, this research aims to determine the characteristics of the primary building materials used in the archaeological buildings at the site of Tell El-Hiṣn, evaluate their current state of conservation, study the causes of their decay, and provide preservation recommendations. Several analytical and examination techniques were employed to achieve these outcomes. These included visual examination, polarizing microscope, scanning electron microscope, X-ray diffraction, and an EDX (Energy Dispersive X-ray Analysis) unit attached to a scanning electron microscope (SEM). Moreover, pecific physical and mechanical properties were determined. The results derived from this paper provide essential information and details about the primary building materials and deterioration agents and evaluate the current preservation state of the discovered archaeological structures at the site of Tell El-Hiṣn. Furthermore, this information and the preservation recommendations will prove invaluable for safeguarding not only the archaeological structures at this site but also those with similar characteristics and conditions at other locations.

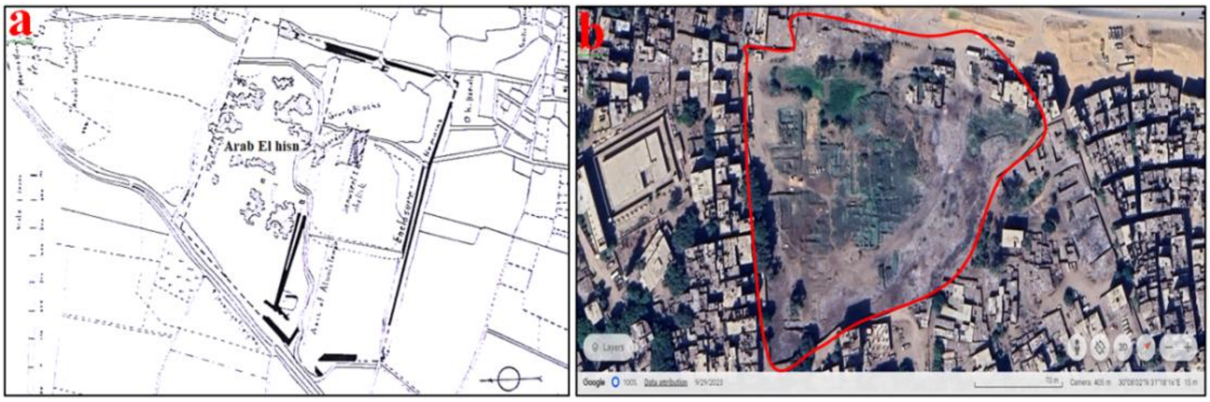

Figure 1. (a) The site of Tell El-Hiṣn at Heliopolis, Egypt (After: Saleh, 1983). (b) The site of Tell El-Hiṣn at Heliopolis, Egypt (After: Google Earth).

2. MATERIALS AND METHODS

2.1. Sampling

Several tiny samples of limestone, mud brick, salts and crusts were obtained and studied using examination and analysis methods to characterize the primary building materials used in the archaeological structures at Tell El Hiṣn. To determine the physical and mechanical properties of the archaeological limestone, cubic samples ($3cm^3$& $5cm^3$) were prepared from the fragments allocated for research and study by the excavation missions (Licciulli et al., 2011, 437-444).

2.2. Polarized light microscope

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A Nikon eclipse LV100POL polarizing microscope was used in the petrographic study of the collected archaeological limestone and mud brick samples after they were prepared as thin sections. The petrographic research aims to identify the textural features, mineralogical composition and weathering aspects.

2.3. Scanning electron microscopy (SEM) attached with EDX

The micromorphological and mineralogical studies of the archaeological collected samples were conducted using a Scanning Electron Microscopy (SEM) model Quanta 250 FEG (Field Emission Gun), connected to an EDX (Energy Dispersive X-ray Analysis) unit, with an accelerating voltage of 30 KV, with 14^x up to 100000^x magnification and resolution for the gun. The investigation was performed without covering the samples (environmental type). The micrographs were taken in backscattered electron (BSE) mode. An attached EDX (Energy Dispersive X-ray Analysis) unit equipped with a secondary electron detector was used to determine the elemental chemical composition of the collected samples.

2.4. X-ray diffraction analysis

The mineralogical study of the collected samples was conducted using the Philips Analytical X-ray V. B diffractometer. The operating conditions were - Diffractometer Type: PW1840; Tube Anode: Cu; Generator tension (KV): 40; Generator Current (mA): 25; Wavelength Alpha1 (Å): 1.54056; Wavelength Alpha2 (Å): 1.54439; Intensity ratio (Alpha2/ Alpha1): 0.500; Receiving slit: 0.2/ Monochromator used: NO. The mineralogical composition of studied samples was identified automatically by comparing the obtained XRD patterns and relative intensities with the "JCPDS cards" (JCPDS., 1967).

2.5. Physical and mechanical properties

The physical and mechanical properties of the collected limestone samples were determined according to the following methodology: -

Bulk Density (D) in g/cm^3 was determined according to (ASTM C 97-47), by dividing the dry weight of samples (Wg) (after drying for 24h at 110^{o}C) of the specimens by the total volume (Vcm^3), (Christaras., 1996) as follows in the equation:-

$$D = \frac{W}{V}$$

Water absorption was determined according to (UNE-EN 13755: 2008), as follows in the following equation: -

$$W.A = \frac{W_2 - W_1}{W_1} \text{ X } 100 = \%$$

W.A is the water absorption in %, W2 is the saturation weight of the sample in g after immersion in a water bath for 24h, and W1 is the dry weight of the sample in g before immersion.

Apparent porosity was determined as seen in the following equation:- (Barnoos et al., 2022, 14-63).

$$A.P = \frac{W_2 - W_1}{V} \text{ X } 100 = \%$$

AP is the apparent porosity in %, W1 is dry weight in g, W2 is wet weight in g, and V is the volume in cm^3 (Hemeda, et al., 2018, 835-846).

The compressive strength of the archaeological samples was determined using an Amsler compression-testing machine, in accordance with the ASTM C 170 standard (1976), by testing three samples and calculating the average results (ASTM C., 1976; Aldosari et al., 24-38).

Abrasion resistance (AR) of the archaeological samples was determined using Bohme abrasion wheel 1006, according to EN 14157 standard (2004) (Çobanolu, et al., 2010, 3398-3404; Mehmedi, 2014, 196-207; Helmi and Hefni., 2016, 87-96).

3. RESULTS AND DISCUSSION

3.1. Field observation

Detailed visual observation and condition survey of the discovered archaeological structures of limestone and mud brick at the site of Tell El-Hiṣn confirmed that they were highly affected by many deterioration patterns on the macro and micro scale. Where it was observed the exfoliation, cracking, gaps, bulges in the lower parts, pits, open joints, discoloration, staining, soiling, missing parts, erosion, crumbling to splitting, salt efflorescence, damp patches associated with hygroscopic salts, accumulates of dirt, granular disintegration (powdering), scaling, graffiti and scratches. In addition, the soil of the site and all the discovered archaeological structures are saturated with moisture (groundwater) (Figure 2).

The main deterioration factors of the studied archaeological structures are groundwater, salt weathering, air pollution, human encroachments, microbiological attacks, and biological attacks by plants and animals. The main reasons for the rise in the groundwater level in the studied site's soil are the sewage water leaking from the Al-Tawfiqia Canal near the site, in addition to the random urban expansion surrounding the site, which lacks infrastructure (Coppola et al., 2020, 1-8). Most buildings in this area have underground tanks for sewage water storage, which are regularly emptied and pumped out. Unfortunately, the water from these tanks seep into the soil of the archaeological site, which is situated at a lower level compared to the surrounding areas. The presence of saline subsurface water (sewage) poses a threat to limestone through two processes. First, it leads to physical weathering, which occurs mainly due to salt weathering, causing a large amount of disintegration and destruction of archaeological stone buildings in a wide range of environments. This is due to the crystallization pressure or hydration pressure exerted by the salts (EL.Gohary, 2013, 447-458; Benkmil et al., 2020, 1-6; Alfanoa et al., 2023, 150-159). Secondly, the chemical weathering when the water is acidic ($pH < 7$) will attack limestone and dissolve it (El-Metwally and Ramadan, 2004, 363–370; Yeşil, 2021, 363-387). Groundwater poses a serious threat to mud bricks, as their constituents (clay materials) have the ability to absorb water or moisture, which then spreads through the porous structure, resulting in swelling. Conversely, during periods of drought, the mud bricks lose their water content and shrink, leading to the formation of cracks. The cumulative effects of swelling and shrinkage ultimately result in the breakdown and fragmentation of the mud brick structure (Abd-Elkareem & Fouad., 2022, 565-585). The limestone and mud brick structures under study experience salt weathering because of direct contact with saturated soil containing saline water (sewage water). This water migrates from the soil to stone structures through a capillary system. As a result of evaporation, salt crystallization frequently takes place within the stones, either near the surface (sub-efflorescence) or on the surface (efflioresce), leading to diverse forms of deterioration (Chiaki & Swe Yu., 2021, 8-32; Martínez et al., 2021, 123–132; Manci., 2023, 27-46). The deterioration of archaeological buildings due to crystallization pressure depends on several factors. These include the composition of the solution, its saturation ratio, the supply rate of the solution, capillary action, evaporation rates, the energy difference between the salt crystal and the pore wall, environmental conditions (the wind speed, changes in temperatures and direct/indirect sunlight radiation), substrate properties (physical and mechanical), location of crystallization and salt crystallization growth patterns (Steiger., 2005, 455-469; El-Gohary., 2010, 61-79; Martínez et al., 2017, 262-276; Bala'awi et al 2018, 49-66; Benavente et al., 2021, 1-16; Oguchi, and Yu 2021, 8-32). The growth of salt crystals within limestone and mud brick pores significantly contributes to its deterioration, resulting in serious surface loss and potential structural collapse. Furthermore, the crystallization of salt on the stone surfaces not only leads to discolouration but also blocks the wall inscriptions (Ruiz-Agudo et al., 2012, 40–51; Ruffolo et al., 2014, 753-758; Benavente et al., 2021, 1-16; Zhang et al., 2023, 1-13).

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Additionally, the presence of archaeological limestone structures in the open air, without shelter or protection, exposed them to chemical weathering by various factors. These factors include acid rain and atmospheric pollutants like hydrocarbons, carbon monoxide, carbon dioxide, sulphur dioxide, oxides of nitrogen (NOx), volatile organic compounds (VOCs) and particulate matter (PM). This has been substantiated through the careful observation and examination of many related deterioration forms, such as discoloration due to the deposition of soot, soiling, and the presence of recrystallized crusts (Giavarini et al., 2008, 14-22). It is worth noting that the Tell el Hiṣn area experiences significant air pollution by vehicular traffic (cars, trucks, and buses), urban industrial activities and the burning of large quantities of garbage, which frequently accumulated both inside and outside the site due to the harmful activities of the surrounding population (Rovellaa et al., 2021). The pollutants emitted by these sources settle on the surface of archaeological structures, and the extent of their reaction depends on various factors such as the nature of the building material, moisture content, atmospheric water (rain, dew or fog), type of pollutant and deposition mechanism. Over time, acid deposits form, which could increase the dissolution of calcium carbonate, the main component of limestone and the formation of the crust. The specific result of this process depends on the type of deposited acid (Saheb et al., 2016, 742-752; Comitea and Fermo., 2018, 1-10).In addition, there are clear signs of direct human impact, as evidenced by scratches and graffiti on the archaeological limestone structures. The site under study is currently experiencing biological attacks due to the dense growth of plants, including trees and high grasses. This excessive growth obstructs the visibility of various aspects and elements of the site. Moreover, it is causing the chemical deterioration of the discovered archaeological structures and stone blocks by the acidity of their root tips, the acidity of their exudates and other organic compounds they release. These compounds may also be responsible for the discoloration observed on the stone surfaces (Badr and Al-Gharieb., 2013, 25-42; Badr., 2015, 828-845; Aksoy and Çelik., 2020, 9-18). It is also a source for attracting groundwater under the foundations of the walls via its root system. Mechanical deterioration occurs when seeds or roots grow in joints and small cracks in the stones and walls. The root system expands the cracks, eventually causing mutable cracks and fragmentation of the stones and walls. They also pose the main threat to mud brick walls, where roots penetrate more than a meter deep into those walls, causing bulges in the lower parts and causing large areas of them to collapse or calve off. It is worth noting that some of these plants were exposed to dryness and caught fire, which caused thermal shock, burning, an accumulation of soot and collapse of the small gate of "Ramesses IX" (Khalil et al., 2022, 1-25; Orabi & Sallam, 2022, 217-225). The accumulation of soot leads to noticeable alterations in the appearance of decorations and obscures the details, reducing their artistic and aesthetic values. Soot contains acidic components that actively contribute to the deterioration of the limestone. It also constitutes, along with other organic compounds, an ideal medium for the absorption of SO_2 from the atmosphere, and $CaSO_4$ further intensifies these effects (Motawea et al., 2023, 75-90). Birds and dogs engage in destructive behaviour and pose another biological threat. Birds search for food or nesting spots by burrowing into the mud brick walls, while dogs accelerate the destructive process by digging dens for shelter at the base of these walls (Ismael., 2015, 5-32; El-Derby and Elyamani., 2016, 295-315). Biodeterioration was observed by the accumulation of microbiological colonization on the stone blocks, which took the forms of black, brown, green, and white stains that caused discoloration of the stone surface. Predominant deterioration mechanisms of building materials due to microbial growth are microbial oxidation and reduction, the creation of appropriate physicochemical conditions, the production of acidic organic and inorganic compounds, and the formation of biofilms (Negi& Sarethy, 2019, 1014–1029; Gaylarde., 2020, 1469–1482; Paiva et al., 2023, 1-25; Isola et al., 2023, 1-20).

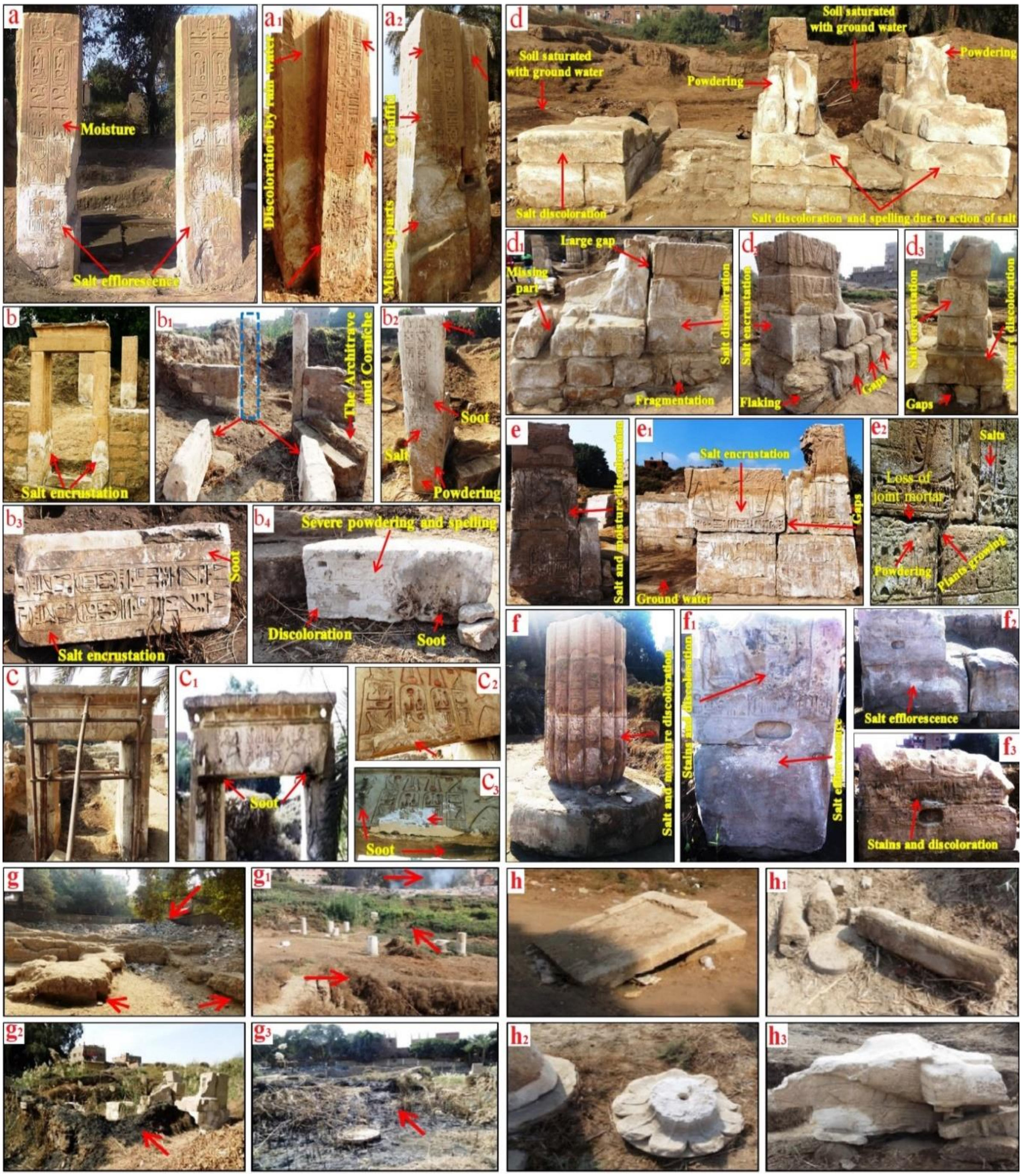

Figure 2 Deterioration aspects of the discovered archaeological structures at Tell El-Hisn. (a- a2) the great gate of "Ramesses IX; (b- b4) the small gate of "Ramesses IX". (c- c3) the gate of "Neb Maat Ra”. (d d3) the statue bases of King "Ramesses II". (e- e2) remains decorated walls of the temple of "Ramses II". (f- f3) remains of the column and decorated walls of the temple dates bake to "Ramesses IV". (g- g3) notes huge piles of garbage inside the site, very heavy vegetation at the site, the dry plants covering large areas of the sites and burning of the garbage and dry vegetation that covers the archaeological buildings; (h- h3) neglecting of the disassociated decorated blocks.

3.2. Petrographic study

The petrographic study revealed that the examined limestone samples from the studied site are composed of calcite as the primary component, along with minor amounts of quartz, iron oxides, gypsum and opaque minerals. Calcite occurs as very fine-grained micrite anhedral

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crystals that represent the matrix of the sample. The micritic matrix contains a significant number of microfossils and fossil fragments that are filled with recrystallized, coarser-grained calcite (sparite). Quartz and opaque minerals occur as very fine-grained, single crystals scattered in the rock matrix. In some parts, the samples show slight staining from traces of iron oxides (Figure 3a-d). According to the results of the petrographic study of the archaeological limestone sample, it is proposed that the discovered archaeological structures at Tell El-Hiṣn are related to the Tora and Al-Masara quarries (Barsoum and Ganguly., 2006, 3788–3796). The petrographic study revealed that the studied mud brick samples are composed mainly of quartz, organic fibres, and fragments of limestone, chert, sandy ironstone and firebrick. These components are associated with minor amounts of feldspar and rare amounts of hornblende, pyroxene, iron oxides, glauconite and opaque minerals. All these components are scattered in a matrix of clay minerals to form very fine-grained aggregates (mainly kaolinite). Quartz is present as fine to medium-grained sand and silt-sized, single anhedral crystals, subrounded to angular in shape. Fragments of limestone, chert, sandy ironstone and firebrick are medium to coarse-grained. Fine-grained, subhedral crystals of feldspar were observed in various parts of the sample. The matrix contains elongated fibres of organic material of different sizes. Very fine to fine-grained iron oxides and opaque minerals were observed and scattered in the clay matrix. (Figure 3e & f).

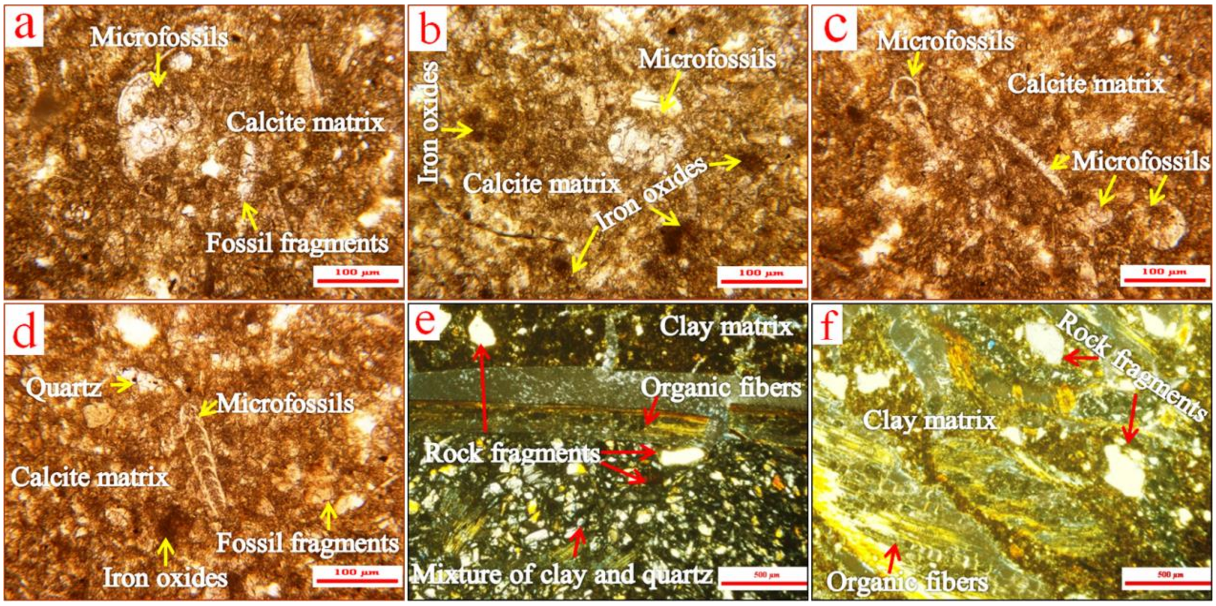

Figure 3 Petrographic microphotographs of limestone and mud brick samples. (a - d) limestone samples under plane-polarized light (PPL); (e& f) mud brick samples under plane-polarized light (PPL).

3.3. Morphological study

The morphologic studies of the archaeological limestone samples at the site under study by scanning electron microscope revealed that the studied samples suffer from many forms of degradation, such as granular disintegration, micro-exfoliation, dissolution of bonding materials, micro-cracks, voids and cavities due to the growth of salt crystals between the calcite grains inside the pores (Figure 4). SEM examination revealed the presence of a dense salt layer coating the surface of the limestone structures, which takes many crystal forms, including salt crust, large euhedral cubic aggregates, acicular, prism, elongated fibrous and needles (Figure 4a-f). These results reflect that the salt layer is composed mainly of sodium chloride (halite) and gypsum. The morphologic studies revealed that the mudbrick sample is generally weak due to the granular disintegration and irregular pores, channels, voids, cracks, cavities and crushed organic fibres elongated in the sample (Figure 4g & h). The cracks in the mud brick sample result from repeated swelling and shrinking of the clay-based materials contained in the mud brick, where swelling is associated with the absorbed water (rainfall, groundwater). In contrast, the subsequent shrinking occurs due to the drying process (the evaporation of water content by wind and thermal influences). The presence of deep fine cracks ultimately results in the disintegration and fragmentation of the overall structure of the mud brick (Abd-Elkareem, 2022, 37-53).

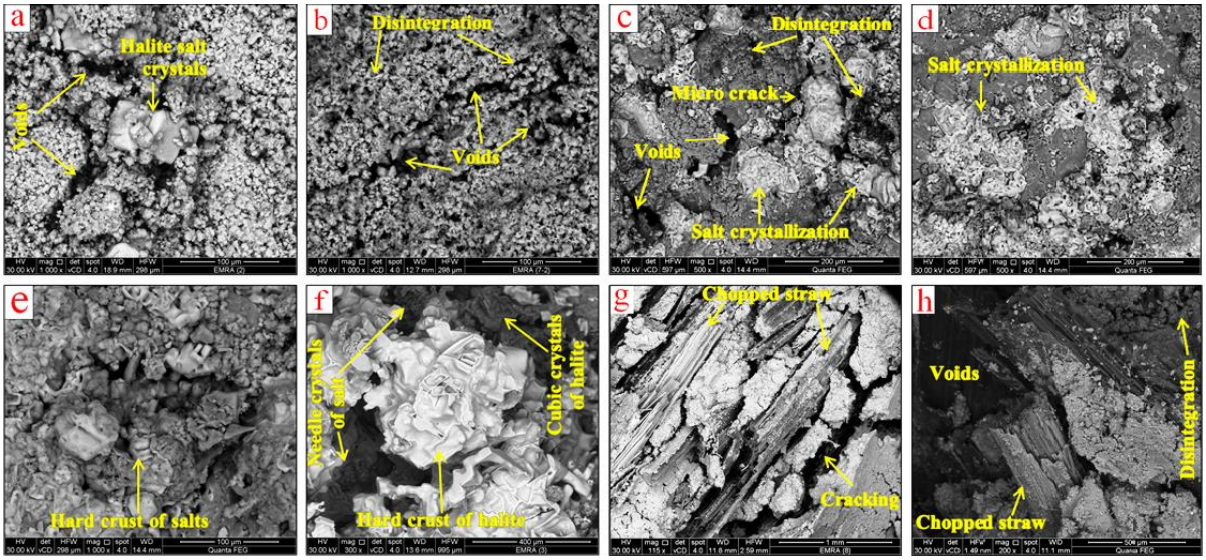

Figure 4 SEM micrographs of studied samples. (a- d) deterioration aspects of limestone, including the formation of cubic halite crystals within the pores, disintegration, voids, salt crystallization in the pores and on the surface, micro-exfoliation and micro cracks; (e& f) a dense coat of salt covering the stone surface; (g& h) deterioration aspects of mud brick including granular disintegration, irregular pores, channels, voids and cracks.

3.4. Mineralogical & chemical compositions

XRD analysis of the limestone sample collected from the great gate of "Ramesses IX" (Figure 5a) indicated that it consists of calcite as the main component and halite as a minor constituent. The EDX spectrum (Figure 5b) confirmed this result, where the high peaks of Ca (29.81 %), C (7.56 %) and O (28.45 %) indicate the existence of calcium carbonate (calcite, $CaCO_3$) as the main component of the sample. The peaks of Cl (14.29 %) and Na (13.24%) were proved the presence of a halite (NaCl). The peak of Si (2.20 %) is related to quartz. The beak of Fe (2.48 %) confirmed the existence of iron oxides.

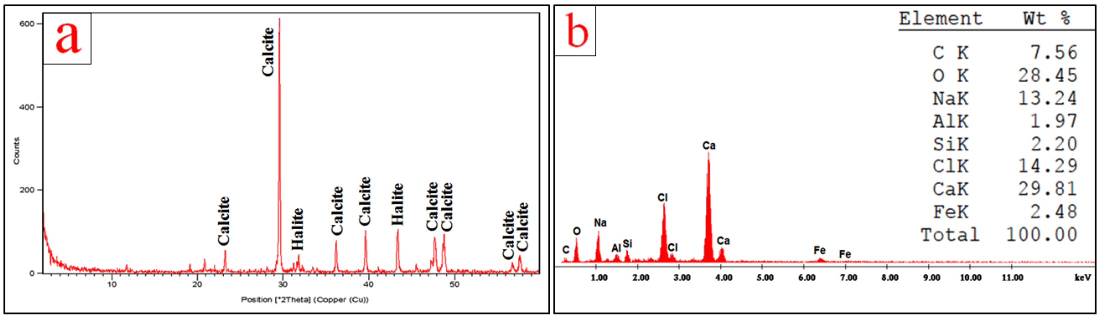

Figure 5 (a) XRD pattern of limestone sample collected from the great gate of "Ramesses IX"; (b) EDX spectrum of the same sample.

The XRD analysis of the limestone sample collected from the small gate of "Ramesses IX" (Figure 6a) clarified that the sample was composed mainly of calcite, quartz and halite. EDX spectrum (Figure 6b) showed a high percentage of Ca (27.41 %), C (10.77 %) and O (35.46 %).

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This illustrated that the calcium carbonate (calcite, $CaCO_3$) is the main component of the sample. The peaks of Na (6.23%) and Cl (6.39 %) proved the presence of halite (NaCl). The peak of Si (6.10%) indicates the existence of quartz. The small concentrations of Fe (1.73 %) indicate the presence of iron oxides, confirmed by the petrographic study.

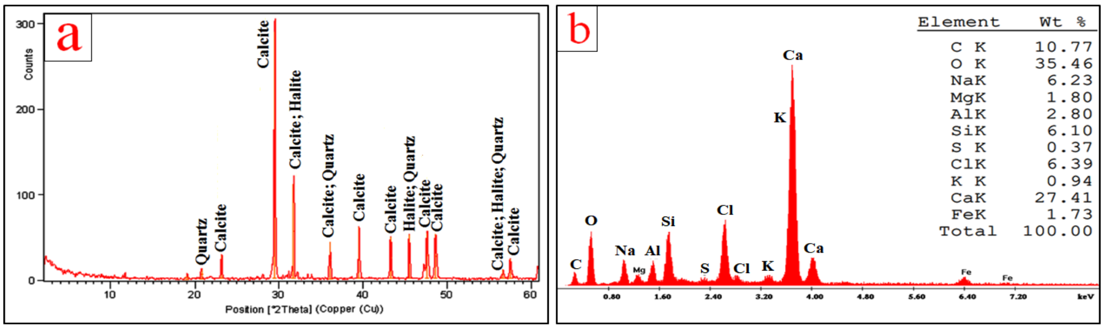

Figure 6 (a) XRD pattern of the sample collected from the salt layer covering the surface of the great gate of "Ramesses IX"; (b) EDX spectrum of the same sample.

The X-ray diffraction pattern of the limestone sample taken from the walls of "Ramesses II" Temple (Figure 7a) clarifies that it is mainly composed of calcite, quartz and halite. Its EDS microanalysis (Figure 7b) showed a high percentage of Ca (21.86 %), C (13.61 %) and O (22.86 %); this illustrates that calcium carbonate (calcite, $CaCO_3$) is the main component of the sample. The high peaks of Na (19.68%) and Cl (18.97 %) proved the presence of a halite mineral (NaCl). The peak of silicon (1.61 %) demonstrated the presence of quartz.

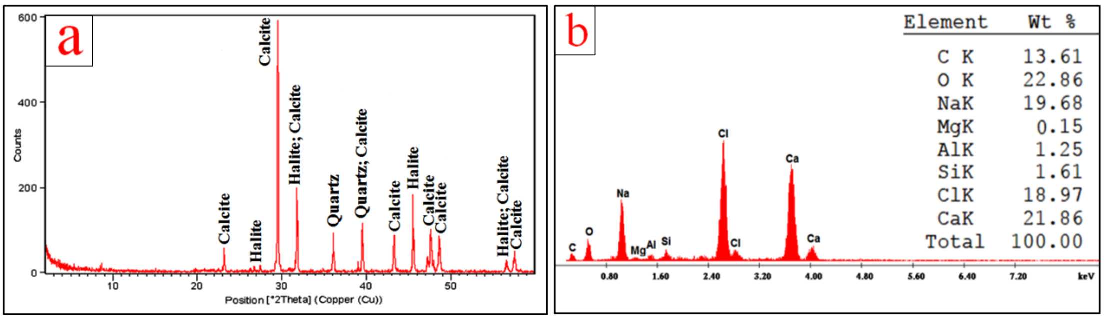

Figure 7 (a) XRD pattern of limestone sample collected from the walls of Ramesses II Temple; (b) EDX spectrum of the same sample.

The XRD analyses of the salt sample collected from the gate of "Neb Maat Ra" (Figure 8a) declare the presence of calcite as the main component of the sample, in addition to minor amounts of gypsum and halite. The EDS microanalysis of the same sample (Figure 8b) confirmed this result, by the presence of peaks related to Ca (31.09 %), C (7.47 %) and O (45.94 %), which are the main components of calcium carbonate (calcite, $CaCO_3$). The beak of S (8.14%) indicates the existence of gypsum in the sample, while the beaks of Cl (1.46 %) and Na (1.06 %) emphasize the presence of halite. The beak of Fe (1.41 %) confirmed the existence of iron oxides. Gypsum may be present as a natural mineral in limestone. In addition, it perhaps was formed due to air pollution by a sulfation reaction (SO_2 ions present in the acid deposition react with calcite $CaCO_3$ present in limestone forming gypsum). This process has various adverse effects and risks for the limestone. Indeed, gypsum is soluble in water 200 times more than the solubility of calcite, and it is usually washed away easily. Moreover, particles trapped on its surface can cause blackening (black crust). The gradual development in the thickness of a black crust leads to a decrease in the porosity of the limestone. The black colour also enhances heat absorption and mechanical expansion, ultimately leading to the gradual disintegration of the limestone (Caner., 2018, 1-6; Comitea and Fermo., 2018, 1-10; Rovella et al., 2020, 1-14;

Pappalardo et al., 2022, 2594-2614).

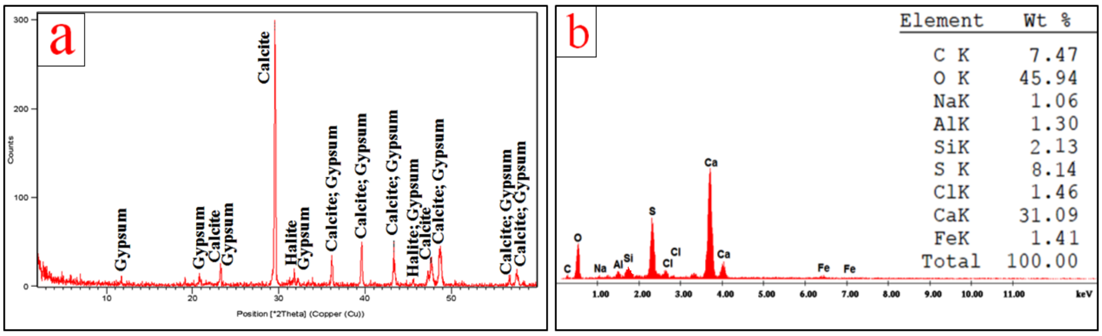

Figure 8 (a) XRD pattern of salt sample collected from The Gate of "Neb Maat Ra"; (b) EDX spectrum of the same sample.

The XRD analyses of the salt sample collected from the statue bases of King "Ramesses II" (Figure 9a) revealed that it consists mainly of halite, nitratine $NaNO_3$, nitrocalcite $Ca(NO_3)_2.4H_2O$ and traces of quartz Sio_2. The EDX spectrum (Figure 9b) confirmed this result by the presence of peaks related to Na (33.50 %) and Cl (7.50 %), which are the main components of the halite mineral (NaCl). The high peaks of Na (33.50%) proved the presence of nitratine. The small concentrations of Si (0.45 %) indicate the presence of quartz. The peaks related to Ca (17.74 %), O (40.81 %) and Na (33.50 %) indicate the existence of nitrocalcite in the sample. Nitrates can be formed in building materials in several ways. One way is when it involves the oxidation of N pollutants emitted by burning fossil fuels. In an environment involving water, nitrogen oxide NOx is converted into nitric acid HNO_3 as a secondary type, acting with calcium carbonate and producing calcium nitrate in the form of a black hard crust. It can also be present in soil due to human activity, organic material decay, or the decomposition of nitrogen products. The infiltration of sewage is a significant source of nitrates, which can then appear as salt efflorescence through rising damp from the soil. Certain types of bacteria can also produce oxidized nitrogen and calcium nitrate by reacting with limestone (Rovella et al., 2020, 1-14; Aly and Hamed., 2020, 895-904; Nagy et al ., 2022, 1-8). It is worth noting that if restorers in conservation work use nitric acid, it has the potential to convert calcium carbonate into calcium nitrate. Sodium chloride salt is considered one of the most widespread salts in Egyptian building materials. It originates in different ways, including sea spray, salt-laden groundwater (sewage water), impurities in construction materials like mortars, and natural stones like Egyptian limestone, which have been formed through marine deposition processes. In addition, hydrochloric acid emissions from various industrial activities contribute to sodium chloride salt. This particular salt species holds a dominant position in Egyptian soil and is recognized as one of the most significant sources of damage. The high concentration of halite in the studied archaeological structures can be attributed to a combination of surrounding deterioration factors, especially sewage water, related to increasing unplanned urbanization in the El-Hiṣn area and the lack of infrastructure (Aly et al., 2015, 1-12; Abdelmegeed and Hassan ., 2019, 183–196). Nitrates and halite are highly damaging due to their high solubility and hygroscopic characteristics, as they have the ability to penetrate deeply and form crystals within the pores of the stone. The repeated process of condensation and evaporation, accompanied by the dissolution and crystallization of salts within the porous system (sub-efflorescence), leads to the formation of micro-cracks and the deterioration of the pore structure (Rovella et al., 2020, 1-14; Benavente et al., 2021, 1-16).

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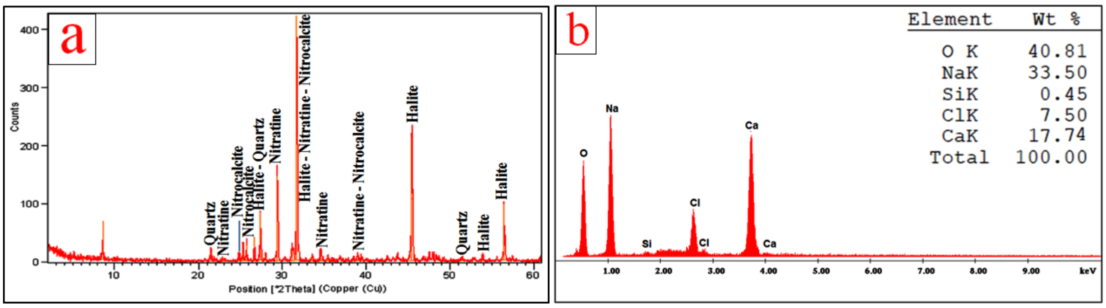

Figure 9 (a) XRD pattern of salt sample taken from the statues bases of the King "Ramesses II"; (b) EDX spectrum of the same sample.

The X-ray diffraction pattern of the hard crust sample taken from the walls of Ramesses II Temple (Figure 10a) revealed that it consists mainly of halite, with minor amounts of quartz and calcite. EDX spectrum of the same sample (Figure 10b) confirmed this result by the presence of peaks related to Na (27.39 %) and Cl (52.73 %), which are the main components of the halite mineral (NaCl). The peaks associated with Ca (5.42 %), C (5.72 %) and O (8.75 %) indicate the existence of calcium carbonate (calcite, $CaCO_3$) in the sample.

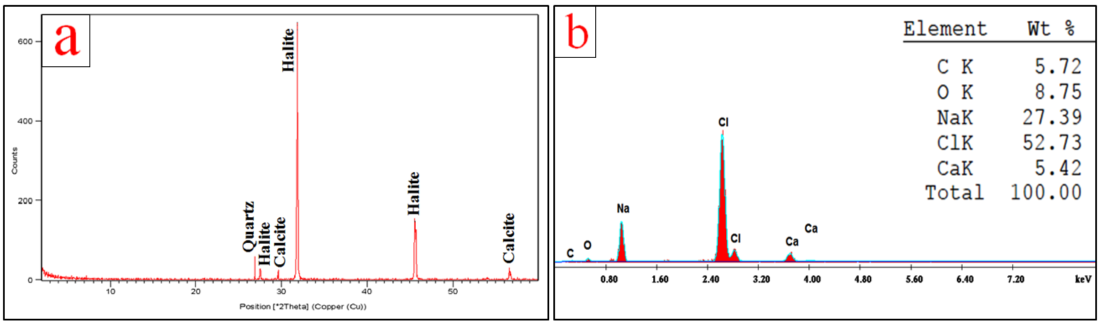

Figure 10 (a) XRD pattern of the sample collected from the dense salt layer covering the stone surface of the walls of Ramesses II Temple; (b) EDX spectrum of the same sample.

The X-ray diffraction pattern of the dense salt layer covering the stone surface of the great gate of Ramses IX (Figure 11a) indicated that the studied sample consists of halite as the main component and calcite as the minor component. EDX spectrum (Figure 11b) confirmed this result, where the high peaks of Cl (25.13 %) and Na (30.06%) proved the presence of halite (NaCl) as the main component of the sample. The presence of calcium carbonate in the sample (calcite, $CaCO_3$) was indicated by the peaks of Ca (4.40%), C (6.99%), and O (29.09%).

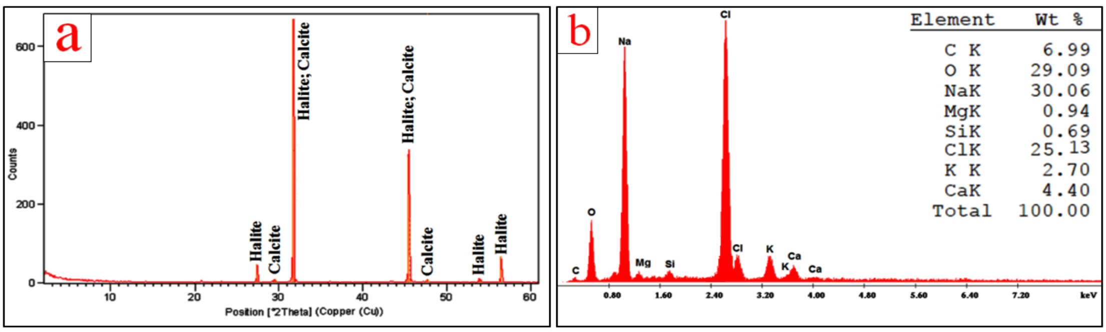

Figure 11 (a) XRD pattern of the sample collected from the dense salt layer covering the stone surface of the great gate of Ramses IX; (b) EDX spectrum of the same sample.

The XRD analysis of the mud brick sample (Figure 12a) indicates that it is composed mainly of Kaolinite Al4 (Si4O10)·(OH)8, montmorillonite $Al_2O_3.4SiO_2{\cdot}H_2O$, albite $NaAlSi_3O_8$, quartz

SiO_2 and halite NaCl. The EDS spectrum in (Figure 12b) confirmed this result by the presence of peaks related to silicon (25.73%), aluminium (6.74%) and oxygen (44.61%), the main components of aluminosilicates (clay minerals). Additionally, the presence of Na (0.42%) and Cl (0.63%) peaks indicated the existence of halite (Sodium Chloride, NaCl).

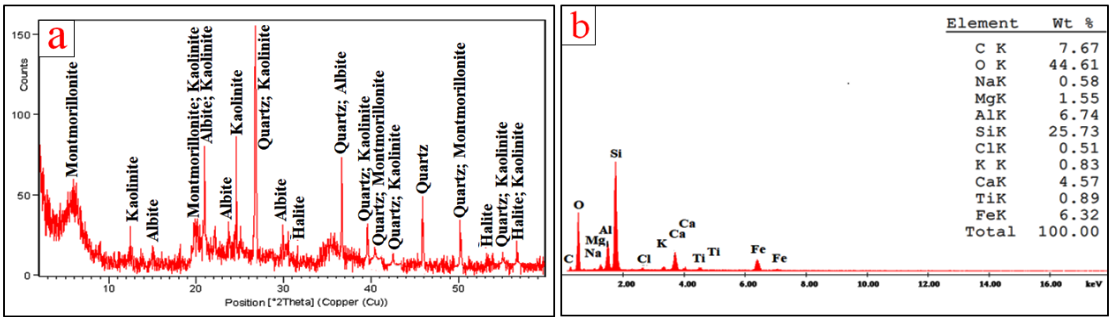

Figure 12 (a) XRD pattern of mud brick sample collected from the archaeological structures at Tell El-Hisn; (b) EDX spectrum of the same sample.

3.5. Physical and Mechanical Properties

Table 1 presents the results of measuring the physical and mechanical properties of the limestone samples. The results presented here demonstrate the aggressive weathering processes of the limestone due to the influence of salt weathering. There is a clear correlation between the salt content and the stone strength. As the amount of salt ions increases, the strength of the stone diminishes (Aly and Hamed., 2020, 895-904). Furthermore, the influence of other degradation factors in the surrounding environment (outdoor environmental) also contributes to the deterioration and weakening of the limestone, ultimately leading to a decline in its physical and mechanical properties.

Table 1. The physical and mechanical properties measurements of the archaeological limestone samples.

Tested samples	Bulk Density gcm^3	Water Absorption %	Apparent Porosity %	Compressive Strength (MPa)	Abrasion Resistance Loss in weight (%)
S1	2.56	6.33	16.55	10.2	10.13
S2	2.68	6.24	16.56	8.8	10.22
S3	2.59	6.46	16.59	8.3	11.05
Average value	2.61	6.34	16.57	9.1	10.47

4. Preservation and conservation recommendations

- To ensure the future protection of the site, the management strategy must incorporate the involvement of local residents. Therefore, the Ministry of Antiquities should conduct cultural awareness campaigns for surrounding residents of the area about the historical and archaeological value of the Tell Al-Hiṣn site and how to deal well with the discovered archaeological structures and preserve them from extinction.
- Separation of the site from the local residents by constructing a perimeter fence equipped with surveillance cameras and a permanent guard system.
- Garbage pickup and removal are not only for the site but also for the local residents of the study area.
- An issue related to increasing unplanned urbanization in the Tell El-Hiṣn area is the lack of infrastructure, which directly influences the archaeological site, including the discovered archaeological structures, specifically the harmful effects of sewage water and garbage. Therefore, it is necessary to enhance the study area's infrastructure by establishing a sanitation system that can collect, process, and drain sewage water.

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Additionally, a steel sheet piles wall can be installed along the perimeter of the Tell Al-Hiṣn site to prevent groundwater infiltration inside the site, as it is situated at a lower elevation (Evelyn, 2009, 41).

- Regular clearing of vegetation by mechanical means by completely removing the plant from its roots, in conjunction with chemical methods by spraying herbicides that do not pose a threat to human health, archaeological remains or soil and should be resistant to any fungal or bacterial growth. These procedures are conducted under the oversight of a scientific committee in order to continue oversight and evaluation.
- There should be disinfection of the site and archaeological structures for the effects of biological damage using safe and effective repellent compounds specifically designed to combat termites and silverfish (Abd-Elkareem and Fouad, 2016, 85-96).
- In order to reduce the influence of groundwater and enhance soil aeration, it is advisable to restrict contact between the walls and saturated soil with groundwater. Therefore, it is advisable to establish small limestone-lined trenches surrounding the limestone structures until they reach the depth of the foundations. These trenches can be covered with iron netting or filled with gravel. Furthermore, it is possible to place an adsorbent material that effectively diminishes the amount of water that reaches the foundations (Romana et al., 2023, 150–159).
- In order to protect the inscribed limestone blocks from the potential impacts of groundwater dispersed within the soil, it is crucial to implement preventive measures that prevent direct contact between the blocks and the soil. This objective can be achieved by constructing insulating concrete bases, specifically designed to withstand moisture, at 45 cm above ground level. These bases will serve as a secure foundation for the placement and exhibition of the inscribed limestone blocks.
- Should be implemented of a surface coverage system (Shelter coating) should include the utilization of wooden or metal umbrellas to protect archaeological structures of mud brick and limestone from the detrimental impact of rainwater and direct sunlight (Matero ., 2015, 209-223; Cavicchio, 2022, 21- 24).
- In order to maintain the stability of archaeological mud brick walls, structural gaps, cracks, and foundations must treated with the removal of dust, salts and other debris. The interior surface of these gaps and cracks should be moistened with a light spray of distilled water. The cracks can be filled with prepared new clay mortar, which has the same characteristics as the old mortar, but with the addition of improvers and an antifungal material of Tobsine M (Thiophanate- Methyl- 70% wettable powder). Treatment gaps can be achieved by using new mud bricks, which have the exact specifications as the old ones, with some additives to improve the physicochemical properties. These new bricks were introduced, stacked inside the gaps, and merged with the original using new clay mortar (Manci & Sedikk, 2023, 27-46). The foundation of mud walls can be treated through various methods, including using support pillows or gradually replacing the damaged mud bricks in the foundation with new mud bricks or injection.
- Consolidation of damaged mud bricks using silicon-based products like tetraethyl orthosilicate (TEOS), potassium silicate, Estel 1000, ethyl silicate and methyltrimethoxysilane (MTMOS)(Franzoni et al., 2015, 398-405; La Russa et al., 2019, 4643-4652).
- The implementation of protective covering layers on the mud brick walls, such as capping and plastering) should be considered. This involves the use of modern mud bricks that have good physical and chemical characteristics to construct two additional rows on the archaeological mud brick walls. Modified clay mortar is also used to plaster the walls. These measures serve as a surface protection layer capable of withstanding

the detrimental effects of rainwater and windstorms and reducing mechanical damage caused by animals, birds, and humans.

- Consequently, this approach shifts the ongoing deterioration of the ancient mud brick walls to the modern mud bricks and plaster layer (Barnard et al., 2016, 84-100). Another method of capping the upper portions of mud brick walls involves using modern mud bricks and mortar to fill in the missing sections. Additionally, plastic sheets known as "Kartunal" are spread over this layer, followed by a 3-4 mm thick layer of modified mortar. These plastic sheets and modified mortar create a protective barrier for the upper areas of the mudbrick walls. Furthermore, the plastic sheets act as an indicator, signalling any damage caused by rainwater and sunlight. In such cases, the mud mortar layer is regularly renewed to safeguard the integrity of the historic walls (Adam ., 2023, 33-53). Another approach for the preservation of mudbrick structures involves their analysis and documentation using photographic geometrical methods and combining 3D recording and GPS. The structures can then be backfilled or reburial to prevent deterioration from exposure to the open environment (Agnew et al., 2004, 133-135; Matero & Moss., 2004, 213-227; Barton., 2009, 489-504).
- The detachment inscriptions from their original walls (exfoliation), resulting from the effects of salt weathering, should be treated by re-adhering to their locations using an appropriate adhesive. Additionally, it is essential to treat and fill the cracks, fissures, and gaps of the limestone structures using a suitable mortar free from salt and compatible with the original walls' physical, mechanical, and optical properties.
- Consolidating the degraded limestone structures is needed to ensure their longevity and resistance to physicochemical damage factors. This can be achieved by using multifunctional Nanocomposites and consolidation products mixtures, which possess the ability to provide both consolidation and protection effects simultaneously (Barnoos et al ., 2022, 14-63; Manci., 2023, 793-818; Hefni, 2023, 293– 309).
- It is advisable to relocate the stone gates of "Ramesses IX" and other inscribed stone blocks from their present positions and transport them to a museum after undergoing the necessary restoration. The transfer of the chapel of "Neb Maat Ra" to the Saffron Museum at Ain Shams University occurred recently, as observed.

5. CONCLUSION

Excavation work at the archaeological site of Tell Al-Hiṣn revealed many archaeological structures of limestone and mud bricks. Those structures have been highly affected by various damage factors associated with the burial environment, including subsurface water, salts, microorganisms, and soiling. However, following their discovery, they were exposed to more aggressive deterioration factors related to the surrounding environment. These factors include sewage water, variations in temperature grades, air pollution, salt weathering, biological and microbiological attacks, as well as intentional and unintentional human interference.

The visual observation and condition survey of the discovered structures revealed many dominant deterioration patterns. These include exfoliation, cracking, gaps, bulges in the lower parts, pits, open joints, discoloration, staining, soiling, missing parts, erosion, crumbling to splitting, disintegration, salt efflorescence, accumulation of dirt and soot, granular disintegration (powdering), scaling, graffiti, and scratches. These patterns clearly indicate the complexity of the deterioration mechanisms because they are related to various physical, chemical, biological, microbiological and human factors. The results obtained with petrographic, morphological and mineralogical studies concluded that the studied limestone consists mainly of calcium carbonate (calcite) as fine-grained (micrite) and traces of very fine-grained quartz as well as the presence of halite. The study also revealed that the salt crusts

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covering limestone structures contained halite, gypsum, nitratine and nitrocalcite salts, which had an adverse effect on limestone.

Furthermore, it indicated that the studied mud brick is composed mainly of clay minerals (Kaolinite, montmorillonite and albite), quartz, organic fibres, fragments of limestone, chert, sandy ironstone and firebrick as well as halite, which is one of the deterioration aspects. The existence of halite crusts covering the surface of archaeological structures reflects the impact of aggressive weathering processes resulting from sewage associated with the random urban expansion surrounding the site, which lacks infrastructure. The presence of gypsum crusts signifies the reaction between calcite $CaCO_3$ present in limestone and sulphuric acid H_2SO_4, which is formed by the oxidation of SO_2 released by burning fossil fuels in an environment involving water and catalysts. These processes pose various adverse effects and risks for the limestone, including the formation of numerous microcracks, fissures, and the black crust, as well as a decrease in the limestone's porosity. The occurrence of Nitratine $NaNO_3$ nitrocalcite indicates the interaction between calcite present in limestone and nitric acid HNO_3, which is formed through the oxidation of N pollutants emitted by burning fossil fuels in an environment involving water. The infiltration of sewage is a major source of nitrates, and specific bacteria can produce oxidized nitrogen and calcium nitrate by reacting with limestone.

Building materials are suffering significant damage from nitrates and halite due to their high solubility and hygroscopic nature. As they both have the ability to penetrate deeply into the pores and form salt crystals. The repeated process of condensation and evaporation, accompanied by the dissolution and crystallization of these salts within the porous system (sub-efflorescence), leads to the formation of micro-cracks and the deterioration of the pore structure. Consequently, the physical and mechanical properties of the studied limestone were negatively affected. The rising subsurface water on the site soil, salt weathering, dense growth of plants and human encroachments present the greatest challenges and obstacles to periodic treatments and conservation processes of the discovered structures. Therefore, the recommendations for preservation are based on two strategies. The first strategy includes embedding the surrounding community in conservation and protection plans to prevent negative activities, such as prohibiting throwing garbage inside or near the site. In addition to prevention and protection procedures from the influence of the prevailing deterioration factors. This includes constructing a perimeter fence equipped with surveillance cameras and a permanent guard system, creating shelters, and removing the garbage and vegetation. The solutions to the groundwater problem include enhancing the infrastructure and installing the steel sheet piles along the site's perimeter, creating small limestone-lined trenches surrounding the limestone structures, and constructing insulating concrete bases for the limestone blocks. The second strategy includes various restoration and conservation procedures for discovered limestone and mud brick structures. These include documentation, cleaning, filling gaps, treating cracks, consolidation and reconstruction, as well as capping and plastering only for mud brick structures.

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توصيف مواد البناء ومخاطر التلف للمبانى الأثرية المكتشفة بموقع تل الحصن، هليوبوليس، مصر

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الملخص

يركز هذا البحث على توصيف مواد البناء وعوامل تدهور المباني الأثرية المكتشفة بموقع تل الحصن، هليوبوليس، مصر؛ لتحقيق الغرض المذكور؛ تم دراسة مواد البناء باستخدام ميكروسكوب الضوء المستقطب، الميكروسكوب الإلكتروني الماسح المزود بوحدة تشتيت طاقة الأشعة السينية (SEM-EDX)، حيود الأشعة السينية (XRD)، بالإضافة للفحص البصري، مع تحديد بعض الخصائص الفيزيائية والميكانيكية. أظهرت النتائج أن الحجر الجيري المدروس يتكون من الكالسيت كمكون رئيسي والكوارتز كمكون ثانوي والهاليت. أظهرت عينات القشور التي تم جمعها من الأسطح المتدهورة وجود الهاليت والجبس والنيترات كبلورات ملحية تملأ المسام وتغطي سطح الحجر. يتكون الطوب اللبن المدروس من الكاولينيت والمونتموريلونيت والألبيت والكوارتز والهاليت. وقد أظهرت الهياكل الأثرية المكتشفة فى موقع الدراسة أضرارا كبيرة مرتبطة بتأثير بيئة الدفن، فضلاً عن تأثير الظروف البيئة الخارجية المحيطة بها بعد اكتشافها. لذا أوصى المؤلف باستراتيجيتين: الأولى تركز على إجراءات الوقاية والحماية من تأثير عوامل التدهور السائد. وتشمل الاستراتيجية الثانية مجموعة من إجراءات الترميم والصيانة للمبانى الأثرية المكتشفة.

بيانات المقال

تاريخ المقال
تم الاستلام في ١٥ يناير ٢٠٢٤
تم استلام النسخة المنقحة في ٤ أبريل ٢٠٢٤
تم قبول البحث في ١٧ أبريل ٢٠٢٤
متاح على الإنترنت في ١٧ أبريل ٢٠٢٤

الكلمات الدالة

تل الحصن؛
الحجر الجيرى؛
الطوب اللبن؛
خصائص؛
عوامل تلف؛
مظاهر تلف؛
الفحص بالميكروسكوب الألكترونى الماسح؛
التحليل بوحدة تشتيت الطاقة؛
التحليل بحيود الأشعة السنية.