Analytical Study and Deterioration of some Firing Pottery kilns in Tel Ayon Mousa in Southern Sinai: Case Study

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Abstract: Firing Pottery kilns in Tel Ayon Mousa in Southern Sinai are the most important archaeological sites in Sinai. The site has various kilns. It is an important industrial archaeological site for firing pottery in Sinai dating back to the late age. It was discovered by the South Sinai Antiquities Area in 1987, it had included many of dome kilns built of brick or limestone and mud brick. Examinations and analysis such as Polarized Microscopy "PLM", Scanning Electron Microscope with Energy dispersive X-ray unit "SEM-EDX" and X-Ray Diffraction analysis "XRD" were carried out on some samples of building material kiln, slag and mortar. The research has proved that the firing pottery kilns were characteristic dome furnaces for the late era; it was reused in different periods from the Greco-Roman to the Islamic period. The research proved that the kilns suffered from various forms of deterioration such as phenomenon of cracking, fracture, flaking, salt crystallization, decomposition, dissolving, immigrating of cementing material or the mortar, as well as fall of some bricks, limestone and mud brick by various damage factors especially wind and rain being the archaeological site was a desert environment including direct and indirect deterioration factors. The research has also proved effect of the human factor on furnaces deterioration, especially the fire next to the kilns by the bedouin causing surface deformation, the kiln was smashed as the site was a military area during the war from 1967 to 1973. Most damage manifestations of archaeological kilns were due to physical, chemical and man-made deterioration factors. The research recommends restoration and rehabilitation of this archaeological site being one of the most important industry centers for Pottery in Sinai. The research also finds the need to raise the archaeological awareness of the residents and specialists as one of the tools for preserving and reviving pottery workshops in Ayon Mousa in Sinai, which was unique technological center of pottery manufacture in Egypt.

Keywords: Kiln, Pottery, Smelting Deterioration, Decomposition, Flaking, Rehabilitation

1. Introduction

Sinai was the eastern entrance of Egypt; its area was 8300 km² with a length of 350 km² [1]. It included many of turquoise and copper mines. The ancient Egyptian had settled Sinai since the Neolithic period extracting copper and turquoise. Mining and pottery manufacture were documented in tombs from old kingdom to Greco-Roman period [2]. South Sinai contained many firing pottery kilns, smelting and extracting copper and tin [3], as well as many inscriptions left by ancient mining missions in Sinai. There were many foundations of smelting metals and firing pottery kilns, in addition to residue of old Mineralization (slag) dating back to different historical periods[4], as in fig.( 1).

Figure.1 firing pottery kilns in Ayon Mousa, South Sinai,
The most important feature of this region was presence of desert deposits, sand dunes and limestone sediments; it was close to the Gulf Suez, 3 km only separated between Ayon Mousa site and Gulf Suez causing existence of Neemolite limestone [5]. Limestone was sedimentary rock consisting of different size granules deposited and combined by silica, iron oxides and clay minerals [6]. Limestone was calcium carbonate as a major component; it was used as a building material or in decorative purposes especially crystalline lime stone [7]. Limestone was characterized by varying and heterogeneous mineral components [8]. Most of deterioration was due to natural or physico-chemical weathering [9]. Wind, Water, Heat played an important role in weathering and furnaces damage [10]. Weathering processes and their impact depended on climatic conditions in the site and the nature of building materials of firing pottery kilns [11]. Sinai was characterized by a dry desert climate, Sinai lacked of rainfall, but there were some floods at irregular times. In the summer, the temperature in Sinai was 40-45 °C, which caused extreme change in the temperature between night and day resulting expansion of quartz or calcite grains and their contraction during the night causing cracking and exfoliation phenomenon due to stresses and strains [12]. Building materials of firing pottery kilns in Ayon Mousa in southern Sinai were exposed to physico-chemical damage especially heat and relative humidity [13]. Daily and seasonal changes moisture was one of the most important deterioration factors and weathering of building materials kilns, the most important moisture sources in Ayon Mousa in southern Sinai were rain, floods and condensation. The relative humidity was 60: 80%, it had a significant impact on the damage of building materials for burning pottery kilns or smelting furnaces. Moisture played an important role in decomposition, dissolving, erosion and dislocation of mortars from burning pottery kilns. Wind was also one of the most serious damage factors for firing pottery kilns in South Sinai causing kilns surfaces deformation occurring some gaps and erosion of components of the building materials kilns [14].

The chemical weathering caused chemical change for components of firing pottery kilns, the most important chemical weathering processes were oxidation, carbonation, hydration and solubility [15]. Saline weathering was one of damage factors of firing pottery kilns [16]. Wind was one of the main causes of weathering factors causing erosion and destruction of archaeological furnaces in Siani, wind impact increased according to its speed and carried sand grains size [17].

Man-made damage was also a destructive factor exceeding the natural damage factors; it caused irreversible deterioration such as demolition process to reuse kilns building materials in various building processes. Firing deterioration was occurred due to cooking next to kilns. The high temperature caused change in mineral composition of kiln building materials [18]. Mud brick kilns were severely damaged due to swelling phenomenon [19] occurring many cracks, fractures, aggregates and detachment [20].

II. Materials and Methods

A. Study Materials
Three samples were selected from each kiln to represent building material, slag and mortar, examination, and analysis were carried out for identification of mineral composition, its chemical changes and phenomena of kilns deterioration in South Sinai.

B. Study Methods

B.1. Visual Examination
The visual examination method was the first step of the examination processes, some lenses were used to diagnose and evaluate the burning pottery furnaces deterioration in Ayon Mousa in southern Sinai.

B.2. Petrographic Examination
Polarizing Microscope was an important in petrographic structure study of the mineral components of building material firing pottery kilns. The petrographic examination of samples shows the chemical changes and deterioration, as well as the shape and size of grains and their distribution. The samples were prepared in thin section for the petrographic examination using the polarized microscope (Olympus BX51 TF japan attached with digital camera under magnification 4X up to 40X), it was conducted at the Faculty of Science, Cairo University.

B.3. Scanning Electron Microscope with Energy Dispersive of X-Ray unit "SEM-EDX"
Scanning Electron Microscope with Energy Dispersive of X-Ray unit "SEM-EDX“ provides accurate information on surface morphology, crystalline structure, grains size, its distribution, texture deterioration and mineral composition for components of building material firing pottery kilns. The examination and analysis were done by using SEM FEI Quanta 250 "SEM Environmental Microscope”, the operating conditions were20 kV and 1 × 10° A, it was conducted at environmental scanning electron microscope Unit at the National Research Center in Cairo.

2.2.4- X-Ray Diffraction Analysis
XRD analysis gives mineral components of building material firing pottery kilns in Sinai to diagnose damage phenomena and its mineral changes for building material firing pottery kilns in Sinai. This analysis was carried out at institute of metals in Cairo.
III. Results

A. Visual Examination
Visual Examination showed that firing pottery kilns in Ayon Mousa in southern Sinai suffered from sand accumulation inside the furnaces, they were affected by surrounding environment (desert environment), it had proved existence of erosion, cracking, fractures, weakness, detachment phenomenon due to changes in temperature, relative humidity and wind or physico-chemical weathering [21]. It revealed fall of some bricks or limestone blocks inside the kilns, mud bricks kilns were eroded. The outer layer was exfoliated due to rain and wind. The rate deterioration in the site depended on properties of building materials kilns such as porosity and permeability and climatic conditions [22]. The dimensions of the first kiln were 26 x 11 cm, it had no shoulders, the width was 50 cm, the height was 150 cm, the entrance width was 60 cm and the entrance located in the north side. The dimensions of the second kiln 26 x 11 cm, height 2 m, the entrance height 80 cm, entrance width 50 cm, the entrance was two shoulders attached to sloping stairs to put the pots in the firing room. The dimensions of the third kiln 26 x 11 cm, it had shoulders, the thickness of the wall 50 cm, diameter kiln 150 cm, width of the entrance 60 cm, and the entrance located in the east side, as in fig.(2).

B. Polarizing Microscope Examination of Different Kilns
B.1. Polarizing Microscope Examination for brick Kiln
Building material kiln and slag samples were examined by polarizing microscope. It showed presence of coarse grains quartz with various sizes and shapes, they were sub round and angular grains, as well as presence of plagioclase, pyroxene, calcite, biotite, iron oxides and grog (10 X-CN) as in fig.(3). The examination by polarized microscope for slag sample of first firing pottery kiln showed presence of quartz, calcite, grog, and iron oxide (10 X-CN) as in fig.(4).

B.2. Polarizing Microscope Examination for lime Stone Kiln
Building material kiln and slag samples were examined by polarizing microscope. It showed presence of calcite grains, fossils, biotite, muscovite, and iron oxides (10 X-CN) as in fig.(5). The examination by polarized...
microscope for slag sample of lime stone kiln showed presence of quartz, calcite, plagioclase, pyroxene, biotite, muscovite, burnt clay (grog) and iron oxide (10 X - CN) as in fig.(6).

3.2.2. Polarizing Microscope Examination for Mud Brick Kiln
Slag sample was examined by polarizing microscope of mud brick Kiln. It showed presence of quartz, calcite, pyroxene, plagioclase, biotite, muscovite, burnt clay (grog) and iron oxides (10 X - CN) as in fig.(7).

C. Examination and Analysis by Scanning Electron Microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX" for firing pottery Kilns.
Five samples represented three firing pottery kilns, the samples were brick, limestone and slag, they were examined by scanning electron microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX"

C.1. Scanning Electron Microscope Examination for Brick Kiln
The first sample of the brick kiln was examined by scanning electron microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX". It showed that the sample had a coarse texture of sub-round and angular quartz grains. Grains were eroded, as well as gaps, soil sediments and crystallized salts as in fig. (8).
Slag sample of the wall of the first kiln built in brick was also examined by scanning electron microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX". It showed presence of a heterogeneous coarse texture of different quartz grains, iron oxides and a combination of the burnt clay (grog) as shown in fig.(9).

Figure 8. SEM photomicrograph of the first kiln sample (brick) shows existence of quartz grains, gaps, cracks and crystallization salt (200 X).

Figure 9. SEM photomicrograph of slag first kiln sample (brick) shows existence of coarse texture of quartz grains, gaps, burnt clay and salt (800 X).

C.2. Scanning Electron Microscope Examination for lime stone Kiln
The second sample of lime stone kiln was examined by scanning electron microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX". It showed that the sample had a coarse texture of calcite granules, the grains suffered from erosion, as well as many gaps, soil sediments salt crystallization as in fig. (10).

Figure 10. SEM photomicrograph of the second kiln sample (lime stone) shows existence of calcite granules, gaps, soil sediments and salts (200 X).

Figure 11. SEM photomicrograph of second kiln slag sample (lime stone) shows existence of quartz grains, gaps, cracks, burnt clay and salt (800 X).

C.3. Scanning Electron Microscope Examination for mud brick kiln
Slag sample of the wall of the third kiln built-in mud brick in Ayon Mousa was also examined by scanning electron microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX". It showed presence of a heterogeneous coarse texture of different eroded quartz grains, gaps, soil sediments, burnt clay and crystallization salts as shown in fig. (12).

Five samples represented three firing pottery kilns, the samples were brick, limestone and slag, they were analyzed by scanning electron microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX"

D.1. Analysis by Scanning Electron Microscope for Brick Kiln

The first sample of the brick kiln was analyzed by scanning electron microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX". It showed presence of carbon, 3.12%, oxygen 16.27%, sodium 12.50%, magnesium 1.09%, aluminum 3.81%, silica 10.25% Sulfur 1.99%, chlorine 26.96%, potassium 1.12%, calcium 16.95% and iron 5.93% as in fig. (13).

Slag sample of the wall of the first kiln built-in brick was also analyzed by scanning electron microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX". It showed presence of that carbon is 1.08%, oxygen is 19.36%, magnesium is 0.96%, aluminum is 1.82%, silica is 31.50%, potassium is 0.67%, calcium is 41.25) And iron% 3.35% as shown in fig.(14).

Figure 12. SEM photomicrograph of third kiln slag sample (mud brick) shows existence of eroded quartz grains, gaps, soil sediments, burnt clay and crystallization salts (800 X).

Figure 13. EDX pattern of brick kiln sample, Ayon Mousa, South Sinai

Figure 14. EDX pattern slag sample (brick kiln), Ayon Mousa, South Sinai
The second sample of the lime stone kiln was analyzed by scanning electron microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX". It showed presence of carbon 5.69%, oxygen 31.40%, fluorine 0.94%, sodium 1%, magnesium 1.10%, aluminum 2.87%, silica 6.17 Sulfur, 1.91%, chlorine 1.72%, potassium 0.87%, calcium 41.70%, and iron 4.86% as in fig.(15).
Slag sample of the wall of the second kiln built-in lime stone was also analyzed by scanning electron microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX". It showed presence of carbon dioxide (2.45%), oxygen (27.15%), fluorine (0.94%), sodium (1.24%), magnesium (1.25%) and aluminum 4.43%, silica 36.45%, sulfur 1.74%, chlorine 0.48%, potassium 1.47%, calcium 18.27% and iron 4.86% as shown in fig.(16).

3.4.3. Analysis by Scanning Electron Microscope for Mud Brick Kiln
Slag sample of the wall of the third kiln built-in mud brick was also analyzed by scanning electron microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX". It showed presence of presence of carbon 4%, oxygen 32.94%, sodium 0.92%, magnesium 1.91%, aluminum 2% 11.22%, sulfur 14.12%, chlorine 0.27%, potassium 0.94%, calcium 24.36% and iron 6.32%, as shown in fig.(17).

E. Analysis by X-Ray Diffraction
Nine samples were analyzed by X-ray diffraction analysis; they represented 3 samples for each kiln (building material, slag, mortar).
E.1. Analysis by X-Ray Diffraction for Brick Kiln
Brick kiln sample analyzed by X-Ray Diffraction. The pattern of XRD analysis of brick sample showed presence of albite NaAlSiO₄, quartz SiO₂, halite NaCl, anhydrite CaSO₄, magnetite Fe₃O₄ and hematite Fe₂O₃, as in fig.(18 ).
Slag kiln sample analyzed by X-Ray Diffraction. The pattern of XRD analysis of slag sample showed presence of quartz $\text{SiO}_2$, calcite $\text{CaCO}_3$, halite $\text{NaCl}$, microcline $\text{KAlSi}_3\text{O}_8$ and albite $\text{NaAlSi}_3\text{O}_8$ as shown in fig.(19).

Mortar kiln sample analyzed by X-Ray Diffraction. The pattern of XRD analysis of mortar sample presence of quartz $\text{SiO}_2$, calcite $\text{CaCO}_3$, and anhydrite $\text{CaSO}_4$ as shown in fig. (20).

Lime stone kiln sample analyzed by X-Ray Diffraction. The pattern of XRD analysis of lime stone sample showed presence of calcite $\text{CaCO}_3$ and microcline $\text{KAlSi}_3\text{O}_8$, halite $\text{NaCl}$ as in fig. (21).

Figure 18. XRD pattern brick kiln sample, Ayon Mousa, South Sinai

Figure 19. XRD pattern slag sample (brick kiln), Ayon Mousa, South Sinai

Figure 20. XRD pattern mortar sample (brick kiln), Ayon Mousa, South Sinai

Figure 21. XRD pattern lime stone kiln sample, Ayon Mousa, South Sinai
Slag kiln sample analyzed by X-Ray Diffraction. The pattern of XRD analysis of slag sample showed presence of quartz $\text{SiO}_2$, albite $\text{NaAlSi}_3\text{O}_8$, calcite $\text{CaCo}_3$, halite $\text{NaCl}$, magnetite $\text{Fe}_3\text{O}_4$ as shown in fig.(22).

Mortar kiln sample analyzed by X-Ray Diffraction. The pattern of XRD analysis of mortar sample presence of quartz $\text{SiO}_2$, calcite $\text{CaCo}_3$, halite $\text{NaCl}$ and gypsum $\text{CaSo}_4.2\text{H}_2\text{O}$ as shown in fig. (23).

3.5.3. Analysis by X-Ray Diffraction for Mud Brick Kiln

Mud brick kiln sample analyzed by X-Ray Diffraction. The pattern of XRD analysis of mud brick sample showed presence of quartz $\text{SiO}_2$, albite $\text{NaAlSi}_3\text{O}_8$, calcite $\text{CaCo}_3$ and gypsum $\text{CaSo}_4.2\text{H}_2\text{O}$ as shown in fig. (24).
Mortar kiln sample analyzed by X-Ray Diffraction. The pattern of XRD analysis of mortar sample presence of quartz SiO$_2$, calcite CaCO$_3$, and gypsum CaSO$_4$$\cdot$2H$_2$O as shown in fig. (26).

IV. Discussion of the Results

Through the results obtained from the research, the visual examination results proved that most of burning pottery kilns in Ayon Mousa in southern Sinai suffered from accumulation of sand deposits around and inside the furnaces due to wind, as well as the kilns were affected by the surrounding environment (desert environment), it has also proved existence of cracking and fracture of firing pottery furnaces. The visual examination also revealed fall and separation of some bricks or limestone blocks inside the kilns, it showed erosion of building materials of the furnace due to rain, condensation and wind, it also proved presence of peeling of the outer layer (protection layer) of most burning furnaces by moisture, temperature changes and difference of expansion and contractions coefficients. Finally it showed human damage by fire or destruction.

The polarized microscope examination showed that first kiln sample was brick of coarse texture, quartz grains of various sizes and shapes, as well as presence of plagioclase, pyroxene, calcite, biotite, muscovite, iron oxides and burnt clay. The polarized microscope examination showed that the used clay in forming the brick was Nile clay because of existence of biotite, muscovite, and iron oxides [26]. It showed that slag sample of brick kiln was rock fragment of quartz, calcite, greg and iron oxides. This proved that the kiln was used to firing pottery not for glass or smelting process because copper or tin was not showed in the slag by polarized microscope examination [27].

The polarized microscope examination showed that second stone kiln sample was lime stone including calcite, biotite, quartz, fossils and iron oxides. It also showed that the slag in stone kiln was quartz, calcite, plagioclase, pyroxene, biotite, muscovite and iron oxides. This proved that the kiln was also used to firing pottery not for glass or smelting process because copper or tin was not showed in the slag by polarized microscope examination.

The polarized microscope examination showed that the slag sample from the third mud brick kiln contained quartz granules, calcite, plagioclase, pyroxene, biotite and muscovite, greg and iron oxides. This mineral content of slag proved similar between the second and third slag sample in its mineral composition.

The scanning electron microscope examination of the first brick kiln sample proved presence of coarse texture of eroded quartz grains as well as presence of some gaps, cracks and crystallization salts. It also proved that slag sample from the first brick kiln was coarse texture of different mineral granules, quartz grains, iron oxides and greg.

The scanning electron microscope of stone sample in the second furnace showed that the sample is coarse texture of calcite, as well as presence of gaps and soil sediments and crystallization salts of chlorides, sulphates and...
carbonates that deteriorated limestone by chemical reactions [28]. The examination by scanning electron microscope of slag sample of the second and third kiln showed that it had a coarse texture of heterogeneous quartz granules of different size and shapes, the grains suffered from erosion, as well as existence of gaps, soil sediments and crystallization salts. This mineral content of slag proved similar between the second and third slag sample in its mineral composition and texture.

- The analysis by scanning electron microscope "SEM-EDX" showed the brick sample including carbon, oxygen, sodium, magnesium, aluminum, silica, sulfur, chloride, potassium, calcium and iron, proving that used clay in forming brick was Nile clay. The salts were chlorides, sulphates and carbonates due to the ground water, rains and condensation.

- The analysis by scanning electron microscope "SEM-EDX" showed stone sample of second furnace proved presence of carbon, oxygen, fluorine, sodium, magnesium, aluminum, silica, sulfur, chloride, potassium, calcium and iron proving that stone was lime stone. The salts were chlorides, sulphates due to the ground water, rains and condensation.

- The analysis by scanning electron microscope "SEM-EDX" showed mud brick sample of third furnace proved presence of carbon, oxygen, sodium, magnesium, aluminum, silica, sulfur, chloride, potassium, calcium and iron proving that used clay in forming the mud brick was montomorillonite as it contained high percentage of magnesium. It proved that clay was Nile clay not desert clay.

- The analysis by scanning electron microscope "SEM-EDX" of slag sample from three kiln showed presence of carbon, oxygen, sodium, magnesium, aluminum, silica, sulfur, chloride, potassium, calcium and iron. This proved that the kilns were also used to firing pottery not for glass or metal smelting process because copper (Cu), tin (Zn) and lead (Pb) were not shown in the slag by scanning electron microscope "SEM-EDX.

- X-ray diffraction analysis has shown that the used clay in brick or mud brick manufacture in the first and third kiln were Nile clay being containing albiteNaAlSi\textsubscript{3}O\textsubscript{8}, quartz SiO\textsubscript{2}, magnetite FeO\textsubscript{4} and hematite FeO\textsubscript{3}.

- X-Ray diffraction analysis of stone sample of the second kiln showed presence of calcite CaCO\textsubscript{3} and microcline KAlSi\textsubscript{3}O\textsubscript{8}, halite NaCl. It proved that the kind of stone in the archaeological site was lime stone. The XRD analysis of slag samples of three kilns showed presence of quartz SiO\textsubscript{2}, calcite CaCO\textsubscript{3}, halite NaCl, microcline KAlSi\textsubscript{3}O\textsubscript{8}, albite NaAlSi\textsubscript{3}O\textsubscript{8}, magnetite FeO\textsubscript{4} and hematite FeO\textsubscript{3}. This proved that the kilns were also used to firing pottery not for glass or metal smelting process because copper (Cu), tin (Zn) and lead (Pb) were not identified in the slag by XRD

- The XRD analysis of mortar samples of three kilns showed presence of quartz SiO\textsubscript{2}, calcite CaCO\textsubscript{3}, anhydrite CaSO\textsubscript{4}. It proved that the kind of mortar in the archaeological site was sand and lime.

- The XRD analysis of building materials, slag and mortars samples of three kilns proved presence of chlorides, sulphates and carbonates in all samples.

V. Conclusion

The research found a number of great important results in pottery kilns. Firing Pottery kilns in tel Ayon Mousa in Southern Sinai are an important industrial archaeological center because the site contained kilns with a unique architectural design through which to know development of pottery technology in this site. The research has proved that the firing pottery kilns were characteristic dome furnaces for the late era; it was reused in different periods from the Greco-Roman to the Islamic period. The research proved that type of building materials used in the firing pottery kilns where the used clay in brick or mud brick manufacture in the first and third kiln was Nile clay but it proved that the kind of stone in the second kiln in the site was lime stone. The research also proved that slag of three kilns contained quartz, calcite, halite, microcline, albite, magnetite hematite indicating that the kilns were used to firing pottery not for glass or metal smelting process because copper, tin and lead were not identified in the slag by examinations and analysis. The research proved that mortar samples of three kilns showed presence of quartz, calcite, anhydrite and gypsum proving that the kind of mortar used in building archaeological kilns was sand and lime mortar.

The research proved that the kilns suffered from various forms of deterioration such as phenomenon of cracking, fracture, flaking, salt crystallization, decomposition, dissolving, immigrating of cementing material or the mortar, as well as fall of some bricks, limestone and mud brick by various damage factors especially wind and rain. The research has also proved effect of the human factor on furnaces deterioration, especially the fire beside the kiln or demolition of furnaces as the site was a military area during the war from 1967 to 1973 AD. The research recommends restoration and rehabilitation of this archaeological site being one of the most important industry centers for Pottery in Sinai. The research also finds the need to raise the archaeological awareness of the residents and specialists as one of the tools for preserving and reviving pottery manufacture workshops in Ayon Mousa in South Sinai in Egypt.

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