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MANUFACTURE TECHNOLOGY AND DETERIORATION OF SOME ARCHAEOLOGICAL POTTERY OBJECTS IN WADI KHARIG IN SOUTH SINAI, EGYPT: CASE STUDY

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Key words: (pottery, slip layer, additions, deterioration, gaps, flaking)

Abstract:

Wadi Kharig is one of the most important archaeological sites in south Sinai containing archaeological pottery and rock art dating back to several ages from prehistoric era until new kingdom; it was discovered by Egyptian expedition of south Sinai antiquities.
Some methods of examination and analysis were performed on archaeological pottery such as polarized microscope, " PLM", Scanning Electron Microscope with Energy Dispersive of X-Ray Unit "SEM- EDX" and X-Ray Diffraction Analysis" XRD" and Thermal analysis" TGA" to identify mineral composition of pottery objects, additive materials, mineral changes, fabric, manufacture, and interpretation of manifestations of deterioration for archaeological pottery in Wadi Kharig in South Sinai.
The research proved that the used clay was Nile clay. Additions were pottery powder "grog", added sand and limestone powder "dolomite". Surface treatment of the first pottery object was slip layer but red wash was used for the second pottery object as one of surface treatment tools. Firing atmosphere of two archaeological pottery objects was an oxidizing atmosphere.
The research also demonstrated that damage manifestations of archaeological pottery objects were weakness, fragility, flaking, cracking, gaps, and crystallization of salts such as halite, carbonate and gypsum, in addition to surface deformation by soiling deposits. The research recommends that materials and methods used in treatment of pottery objects (cleaning, salts extraction, consolidation, display and museum storage) should be chosen based on mineral composition, their different properties and their various deterioration manifestations that have been identified by researcher.

1. Introduction

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Sinai represents eastern entrance to Egypt. It was known in past ages mine, turquoise land and land of green copper. It included many turquoise and copper mines such as Serapit elkhadam mines. Ancient Egyptian settled Sinai through different ages for extracting copper and turquoise, securing eastern borders of Egypt since pre-dynastic era (Castel et al., 1995). Interest in Sinai had increased in old kingdom, where many rock inscriptions had been discovered in Sinai such as wadi Kharig, Maghara, Serapit elkhadam, the Rawd Al-Eir, and wadi Nasib. They showed metallurgical activity in these sites (Tallet et al., 2011), as shown in Fig.1

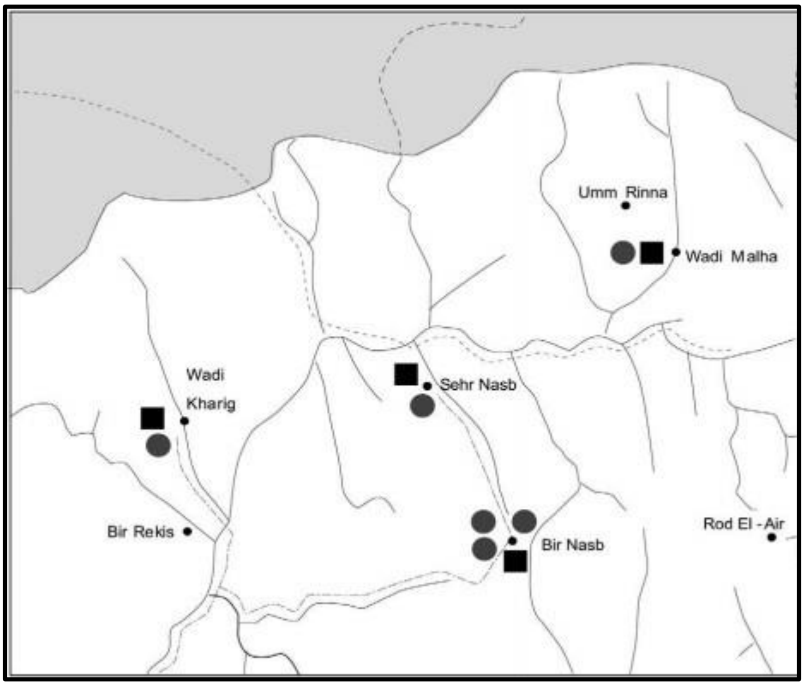


Fig.1. represents mining sites and rock art in South Sinai, Egypt.

Wadi Kharig in south Sinai is considered one of the most important sites of rock art of ancient mining missions for copper and turquoise in all ages. It occupies coordinates (N: 29 ° 03' 00.4 " - E: 33° 20' 56.3"). It included many rock inscriptions such as rock art of king Sahure dated back to the fifth dynasty in old kingdom, Estella of King SesostriesI, which belonged to 12 dynasty in middle kingdom. Archaeological materials as pottery and slag in south Sinai indicated that mining process from pre-dynastic age until new kingdom. It was rocky hills of Nubian sandstone that contained hundreds of inscriptions dating back to middle kingdom (Abdel Motelib et al., 2012), as in Fig.2.

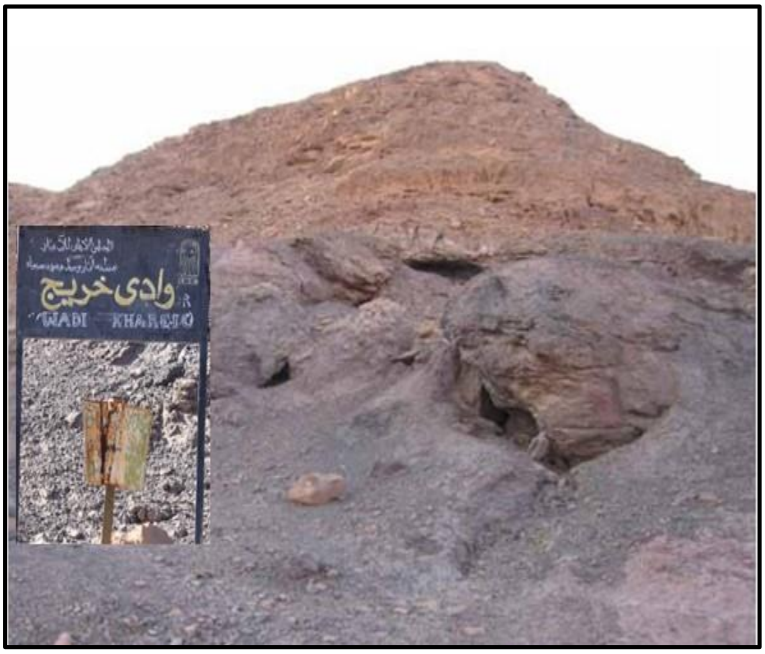


Fig.2. represents wadi Kharig in South Sinai.

Process of examination and analysis is one of the important processes in study of Archaeometrical archaeological materials and their features (El-Gohary et al., 2019). These processes had developed through last times to identify technological manufacture (Stoltman, 2001), its classification, dating, pottery fabric, additions "Temper" (Schleicher et al., 2008), raw materials (Riederer, 2004), burning temperature, firing atmosphere, furnace (El Gayar et al., 1995), and commercial relations (Kelley, 1976). Examination and analyzes also play an important role in explaining mechanism of pottery damage and identifying its various manifestations before and after excavations, where most pottery objects suffered from severe physical, chemical, and biological damage (Ágnes et al., 2012), a scientific method of treatment and maintenance was to be consistent with nature of deterioration proved by the research (Williams, 1983). Clay minerals are a sedimentary rock consisting of hydrated aluminum silicate (Rowell, 1994). In addition to some impurities such as heavy minerals (Prothero, 2006), there are many factors and manifestations of damage (pottery and mining materials) in Sinai due to climate, soil and rock, stone was nubian sandstone characterized by its different mineral components (Abdel-Motelib, 1987). Wind, water, and heat were considered one of the most important mechanical weathering factors (Muthayya, 1999); it is known as rock fragmentation (Singer and Munns, 1999).This species was more prevalent in desert regions (Craig, 1993). Severity of weathering or damage processes and their effect depended on climatic conditions (Sinha, 1992).

Sinai is characterized by a desert climate, it is considered one of dry areas, it lacks rain but sometimes there are some torrents at irregular intervals, temperature in summer was about 45 Ċ causing thermal expansion and contraction (Shata, 1996). Temperature resulted cracking, fracture and exfoliation (Hamilton, 1978).Daily and seasonal moisture changes were one of the most important factors of physiochemical damage (Oliver, 1997),sources of humidity in wadi Kharig in Sinai were rain, torrents and condensation, relative humidity was about 80% in winter, it had a great impact on damage of archaeological pottery (Skibo and Schiffer,1987).Water played an important role in damage process resulting pressures which led to cracking and breaking of pottery (Rahn, 1990). Wind is also one of natural damage factors carrying many sand grains which caused damage to archaeological surface, wind caused gaps, surface deformation and salt crystallization (Foster, 1974).There were other manifestations of damage due to organic activity by plants and trees,where enzymes and acids are excreted damaging pottery (Mitchell,2009).Chemical weathering introduced chemical changes of mineral composition by decomposition of original minerals and forming new minerals such as calcite (Skibo and Schiffer,1987). Carbonization and solubility were the most important chemical weathering processes that caused corrosion and mineral changes (Carla and David, 2001).The effect of chemical weathering on minerals composition was different according to weathering factors and their conditions in the site (Wild,2001), olivine, pyroxene, amphibole and biotite minerals were converted into iron oxides (Simmons,2002), phenomenon of cracking and decomposition were due to microbiological activity of fungi and bacteria (Sterflinger,2010), human damage was also one of damaged factors by burning or functional use (Hope,1987).This study is an important in field of examining and analyzing archaeological pottery in wadi Kharig in south Sinai, which reveals Archaeometry and deterioration of archaeological pottery in this site being one of the most important mining sites in south Sinai,Egypt.

2. Materials and Methods

2.1. Study Materials

Two samples were selected from the archaeological pottery from wadi Kharig in south Sinai, as well as one sample from archaeological site soil, they were used in examinations and analyzes that were conducted by research.

2.2. Study Methods:

2.2.1.Visual Examination

The visual examination method is considered the first stage of examination process. Lenses and microscopes are used to evaluate technological process, functional use, and nature of the damage (Nagwa, 2016).

2.2.2. Petrographic Examination

It plays an important role in study of petrographic structure for mineral components of archaeological pottery and its fabric (Hamdan et al.,2014), samples were obtained from wadi Kharig in Sinai, thin section of pottery samples were prepared, they were examined by polarized microscope (Nikon Eclipse LV100 pol attached with digital camera under magnification 2x up to80X), the examination was conducted at polarized microscope laboratory at the Department of Geology, Faculty of Science, Cairo University.

2.2.3. Scanning Electron Microscope with Energy Dispersive of X-Ray Unit "SEM- EDX"

Scanning electron microscope equipped with EDX unit plays an important role in identifying morphology of pottery surface, its mineral composition and nature of its damage (Badawi, 2000), pottery samples were examined without prior preparation using FEI Quanta 200 UK "JEOL JSM-840 and ESEM Quanta 200 FEG, XTE 325/D8395", operating conditions "20 kV and 1 x 10^{-9A}", this examination was carried out at environmental scanning electron microscope unit at the National Research Center in Cairo.

2.2.4. X-Ray Diffraction Analysis

X-ray diffraction analysis plays an important role in identifying mineral components of archaeological pottery, type of used clay, industry, effect of burning and burial environment (Alaimo et al., 2004).two Archaeological pottery samples and soil sample were prepared for analysis by X-ray diffraction, the used device was Philips: 'X'Pert Graphics' and 'Identify by Philips Software), the diffraction pattern between “4:70 2θ”, the operating conditions were carried out using Cu – Kα radiation, 40 mA, 45 kV, this analysis was performed at micro analysis center , metallurgy and mining institute in Helwan.

2.2.5. Thermal Analysis

Thermogravimetric Analysis "TGA" is used to determine firing temperature of archaeological pottery due to the loss of weight that occurred during firing of the pottery (Elghareb, 2016). Two archaeological pottery samples in wadi Kharig were analyzed by thermal analysis device Known as Perkin Elmer STA 6000, the temperature program ranged from room temperature to 1000°C, measurement range: ±0.2 to ±1000 μV, heating speed: 0 to +50°C/min, temperature and weight loss were recorded on the chart.

3. Results

3.1. Visual Examination

Visual examination of pottery objects in wadi Kharig in south Sinai had proved that shaping method was pottery wheel for the first object, as shown in Fig.3 a, hollowing method was for the second piece, as shown in Fig.3 b. Surface treatment was slip layer for the first object (Fig.3 a) and red wash for the second piece (Fig.3 b). Pottery suffered from flaking, soil deposits, crystallization of salts, and phenomenon of breaking, as shown in Fig. 3 (a-b).

Fig.3. represents pottery objects in wadi Kharig in South Sinai A: pottery pot, B: pottery plate.

3.2. Examination by Polarizing microscope

A sample of pottery vessel in wadi Kharig in Sinai was examined by polarized microscope (surface), it showed presence of quartz granules, in addition to presence of rutile in an iron oxide-rich matrix, as shown in Fig. 4. For the core, it also showed presence of quartz, rotile, muscovite, biotite, and grog powder in an iron oxide-rich matrix, as shown in Fig.5.

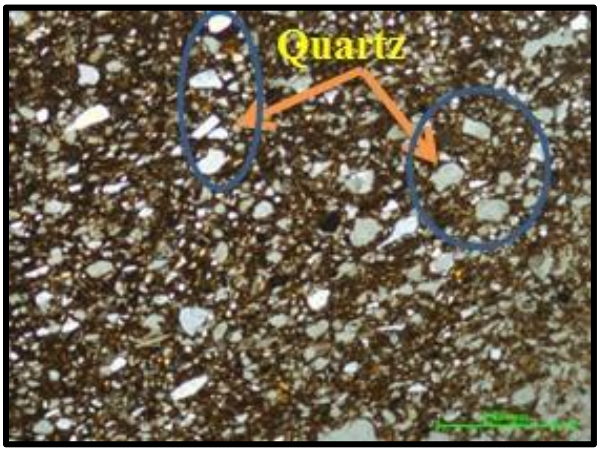


Fig.4. Petrography micrograph of the first pottery sample shows existence of quartz, rutile (10X-CN).

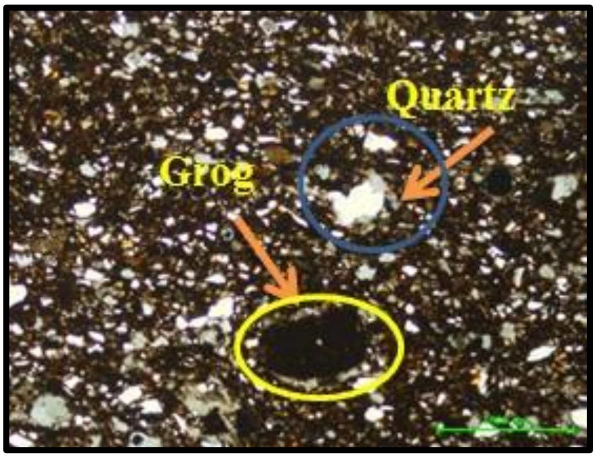


Fig.5. Petrography micrograph of the first pottery sample shows existence of quartz, biotite, and grog (10X-CN).

Another part of the same first pottery sample was examined; it showed presence of quartz granules with sharp and semi-sharp angles and pyroxene in an iron oxide-rich matrix, as shown in Fig.6. It also showed presence of circular and sharp-angle quartz granules, as well as presence of muscovite in an iron oxide-rich matrix, as shown in Fig.7.

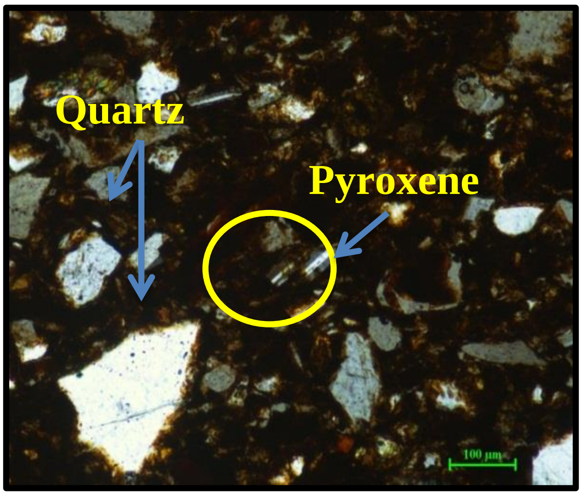


Fig.6. Petrography micrograph of the first pottery sample shows existence of quartz, and pyroxene (40X-CN).

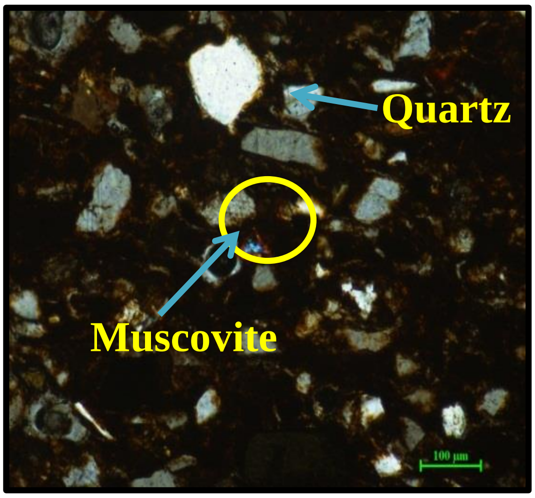


Fig.7. Petrography micrograph of the first pottery sample shows existence of quartz, and muscovite (40X-CN).

The second pottery specimen showed presence of fine grains to coarse grains, as well as lime stone powder and rutile in an iron oxide-rich matrix, as shown in Fig.8. The same pottery specimen also showed presence of circular and semi-circular quartz grains, limestone powder, pottery powder, biotite, and rutile in an iron oxide-rich matrix, as shown in Fig.9.

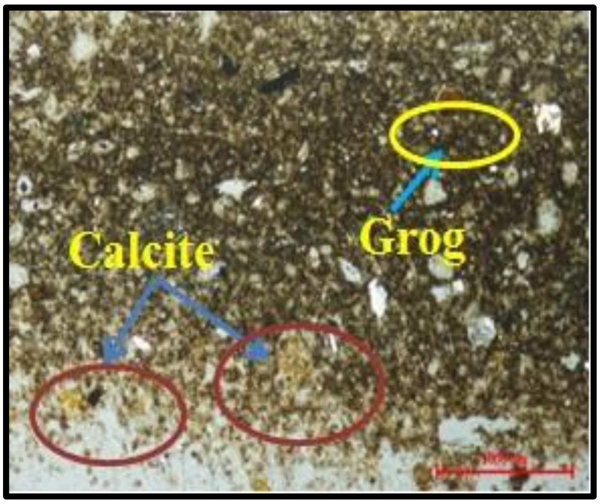


Fig.8.Petrography micrograph of the second pottery sample showed existence of quartz, calcite and rutile (10X-CN).

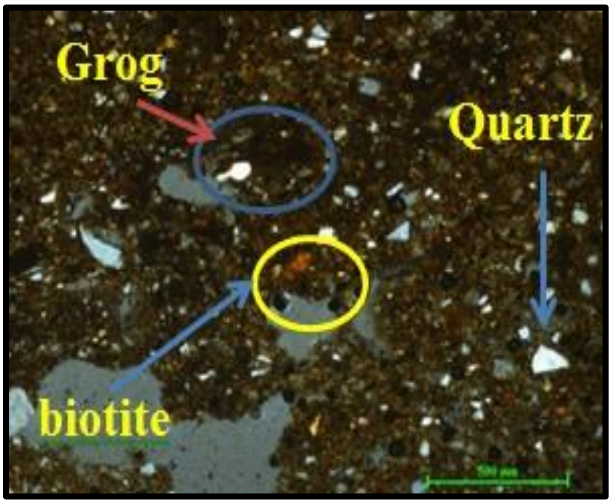


Fig.9.Petrography micrograph of the same pottery sample showed existence of quartz, grog, biotite and rutile (10X-CN).

Another part of the same second sample also showed presence of quartz granules, pottery powder, calcite, rutile, and biotite in an iron oxide-rich matrix, as shown in Fig.10.

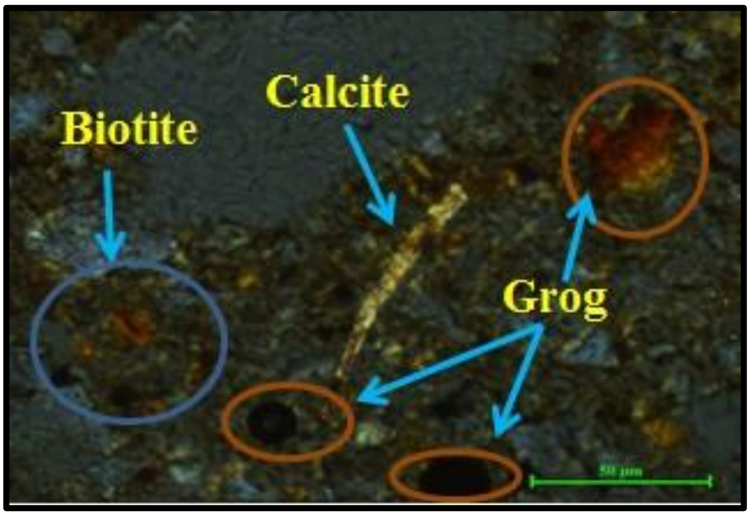


Fig.10. Petrography micrograph of the second pottery sample showed existence of quartz, pottery powder, calcite, biotite and rutile (20X-CN).

3.3. Examination and Analysis by Scanning Electron Microscope coupled with Energy Dispersive of X-Ray Unit "SEM-EDX".

Two archeological pottery specimens in wadi Kharig were examined by scanning electron microscope equipped with EDX unit (SEM-EDX).

3.3.1. Examination by Scanning Electron Microscope.

The examination of scanning electron microscope equipped with EDX unit for the first sample demonstrated presence of fine to coarse pottery fabric, in addition to presence of added quartz granules, gaps, flaking and crystallization of salts, as shown in Fig.11. Examination of another part of the same sample showed presence of quartz granules, crystallization of salts, delicate and micro cracks and some gaps, as shown in Fig.12.

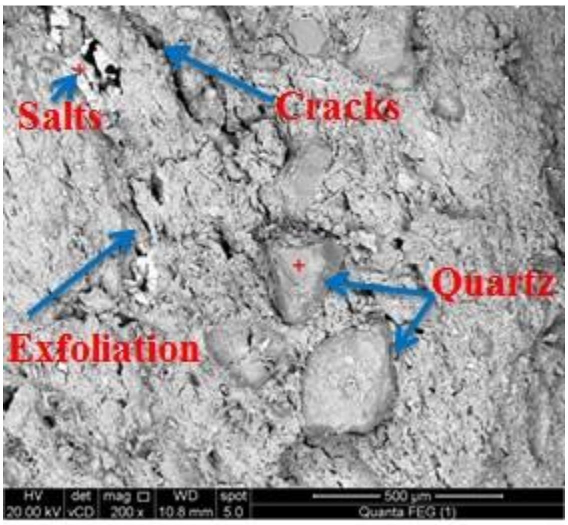


Fig.11. SEM photomicrograph of the first pottery sample.

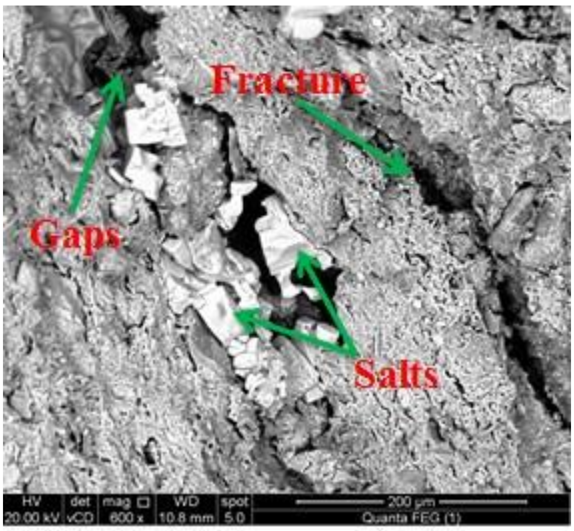


Fig.12. SEM photomicrograph of another part of the same sample.

The examination of scanning electron microscope equipped with EDX unit for the second sample showed presence of fine to medium pottery fabric, in addition to presence of added quartz granules, gaps, flaking and crystallization of salts, as shown in Fig.13. Whereas, the examination of another part of the same sample demonstrated presence of phenomenon of flaking, crystallization of salts, cracks and phenomenon of granulation or fragmentation, as shown in Fig.14.

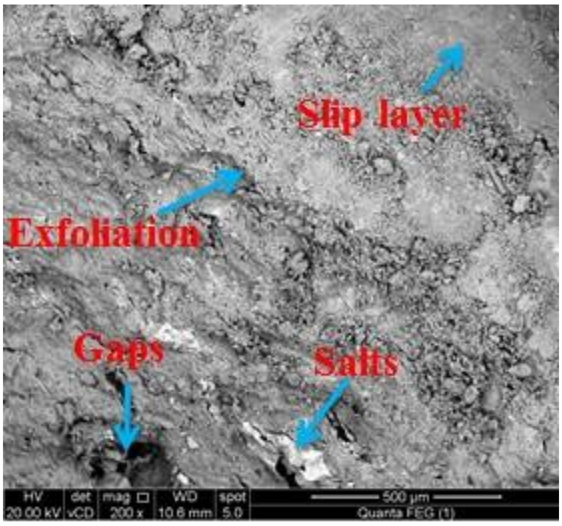


Fig.13. SEM photomicrograph of the second pottery sample.

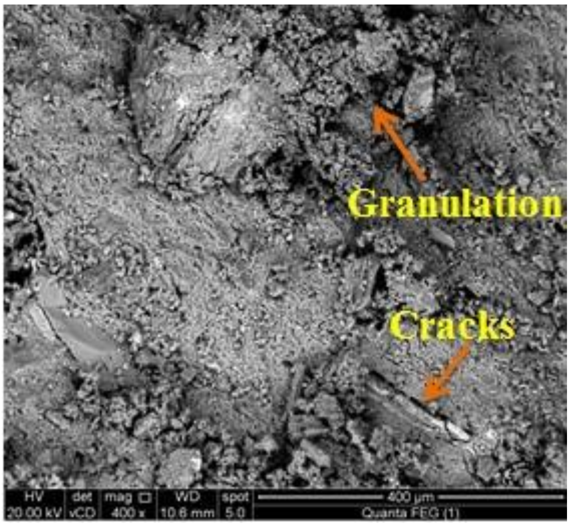


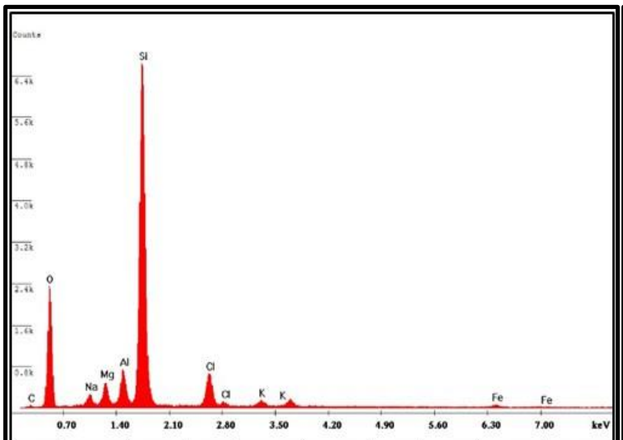
Fig.14. SEM photomicrograph of another part of the same sample.

3.3.2. Analysis of the scanning electron microscopy with EDX.

The results of analysis of the first archaeological pottery sample as shown in figure (15) demonstrated presence of carbon, sodium magnesium, aluminum, silica, chlorine, potassium and iron. The results of analysis of the same first pottery specimen showed presence of oxygen, sodium, aluminum, silica, and chlorine as in figure (16).

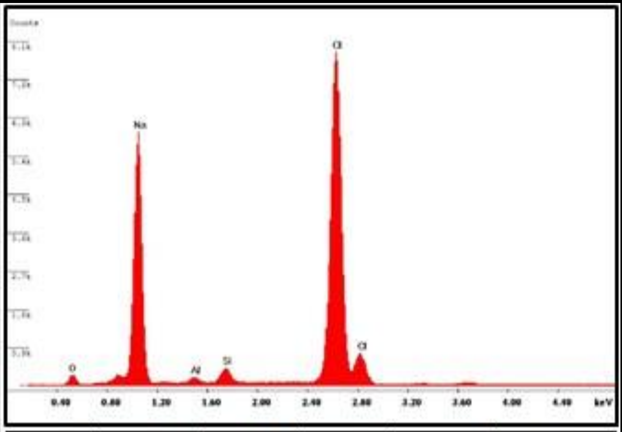
3.3.2. Analysis of the scanning electron microscopy with EDX.

The results of analysis of the first archaeological pottery sample demonstrated presence of carbon, sodium magnesium, aluminum, silica, chlorine, potassium and iron, as shown in Fig.15. The results of analysis of another part for the same first pottery specimen showed presence of oxygen, sodium, aluminum, silica, and chlorine as shown in Fig.16.



C	O	Na	Mg	Al	Si	Cl	K	Fe	Total
2.08	29.05	1.49	2.59	4.50	48.22	7.54	1.64	2.88	100%

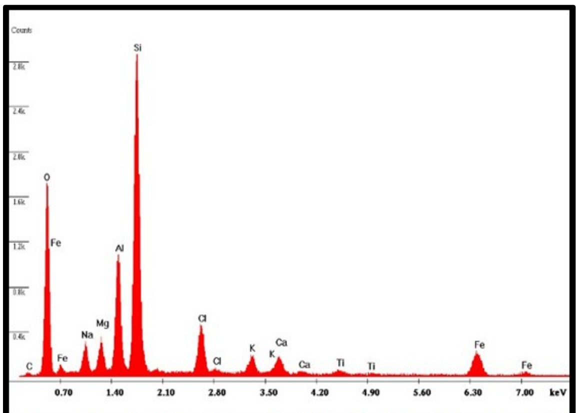
Fig.15. EDX pattern of the first pottery sample of wadi Kharig, Sinai.



O	Na	Al	Si	Cl	Total
3.50	30.22	0.84	2.03	63.41	100%

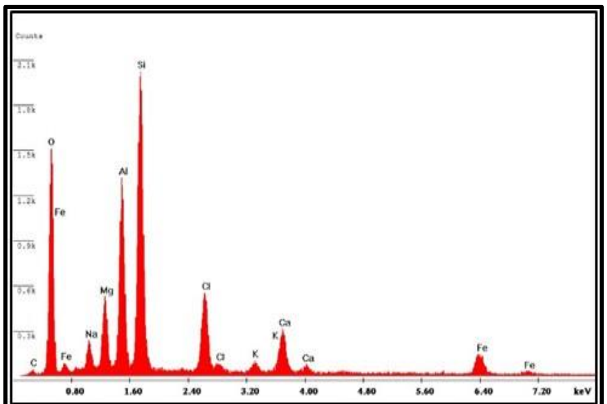
Fig.16. EDX pattern of the same first pottery sample of wadi Kharig, Sinai

The results of analysis of the second archaeological pottery sample demonstrated presence of carbon, sodium, magnesium, aluminum, silica, chlorine, potassium, calcium and iron, as shown in Fig.17. The results of analysis of another part of the same second pottery specimen proved presence of carbon, sodium, magnesium, aluminum, silica, chlorine, potassium, calcium, titanium and iron, as shown in Fig.18.



C	O	Na	Mg	Al	Si	Cl	K	Ca	Ti	Fe	total
1.32	26.80	2.76	2.39	9.89	28.49	6.17	2.76	3.01	1.28	15.14	100%

Fig.17. EDX pattern of the second pottery sample of wadi Kharig, Sinai



C	O	Na	Mg	Al	Si	Cl	K	Ca	Fe	Total
1.08	26.99	4.27	3.77	12.67	23.70	9.44	1.11	6.22	10.75	100%

Fig.18. EDX pattern of the same second pottery sample of wadi Kharig, Sinai

3.4. X-Ray diffraction analysis

The first pottery sample from wadi Kharig in south Sinai was analyzed by X-ray diffraction, XRD pattern contained quartz (SiO_2), hematite (Fe_2O_3), halite (NaCl), albite ($NaAlSi_3O_{10}$), and microcline ($KALSi_3O_8$), as shown in Fig.19. It was clear that the sample was medium-burned because it contained hematite (Fe_2O_3).

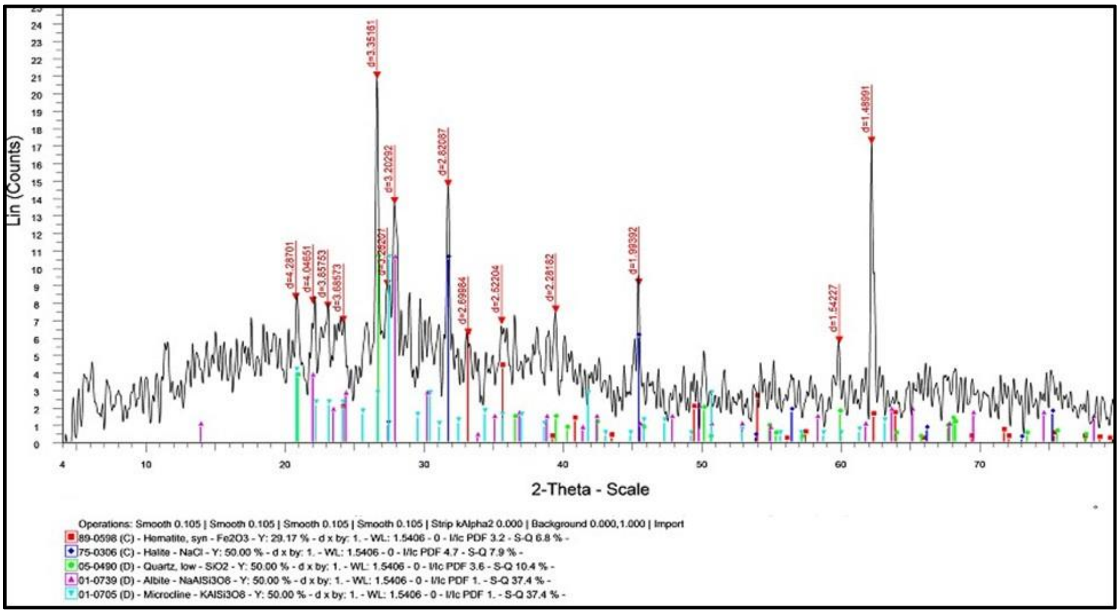


Fig.19. XRD pattern of the first pottery sample of wadi Kharig, Sinai

XRD pattern (second sample) contained quartz (SiO_2), microcline ($KALSi_3O_8$), albite ($NaAlSi_3O_{10}$), and dolomite CaMg (CO_3)2 as shown in Fig.20. It was clear that the sample was medium-burned because it contained dolomite CaMg (CO_3)2.

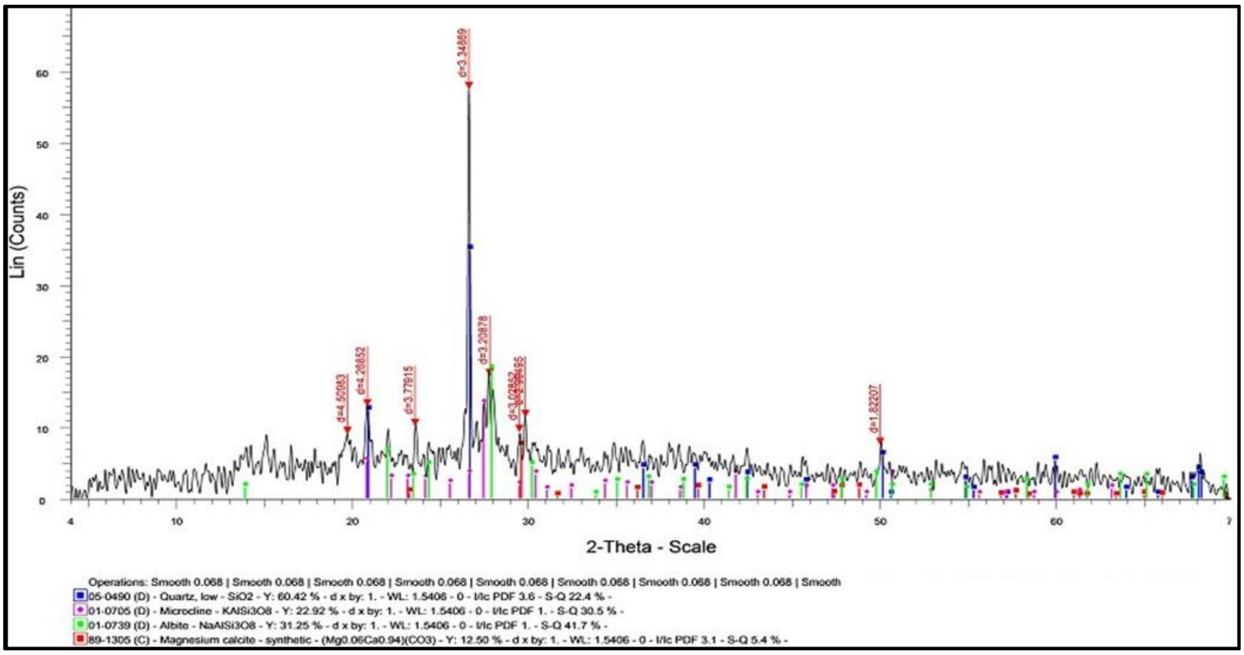


Fig.20. XRD pattern of the second pottery sample of wadi Kharig, Sinai

Soil sample was also analyzed by X-ray diffraction, the XRD pattern contained quartz (SiO_2), microcline ($KALSi_3O_8$), albite ($NaAlSi_3O_{10}$), and halite (NaCl), as shown in Fig. 21. It was clear that soil sample was saline.

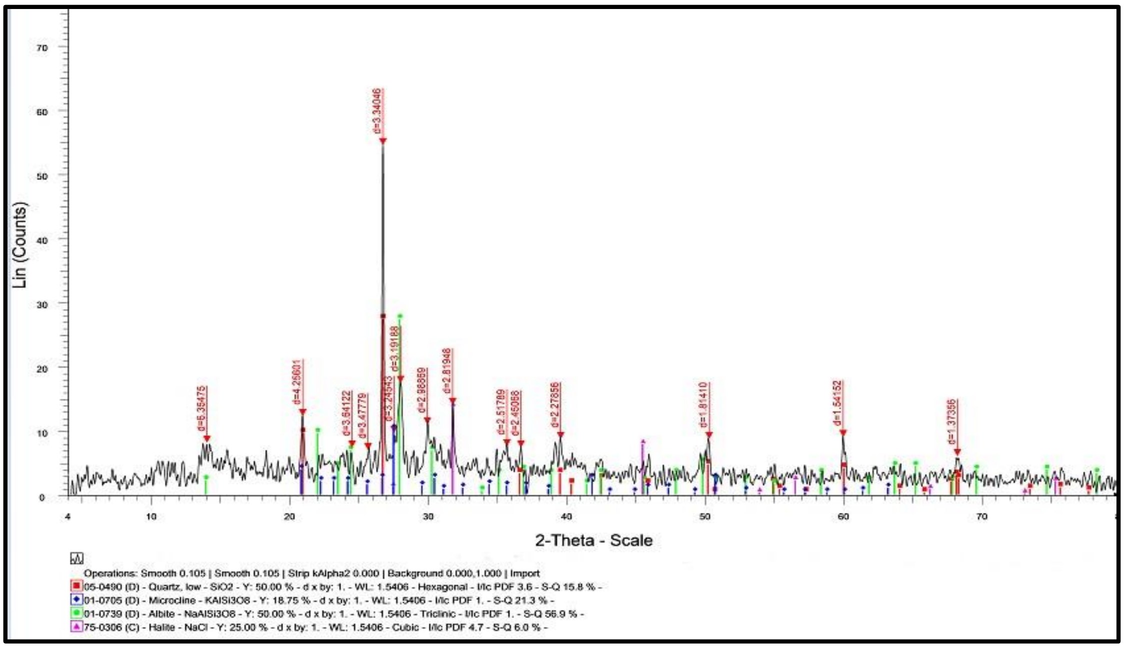


Fig.21. XRD pattern of soil sample of wadi Kharig, Sinai

3.5. The thermal analysis

The thermal analysis TGA (first pottery sample, wadi Kharig, south Sinai) showed weight loss was due to firing temperature. Result of weight loss was 12.99 milligrams at firing temperature from 244.20 °C to 433.80 °C, weight loss was decreased to 1.9 mg from 445.18 °C to 760.90 °C, weight loss was stable at firing temperature from 760.90°C to 800 °C. The results of weight loss indicated that firing temperature was about 760.90°C, as shown in Fig. 22.

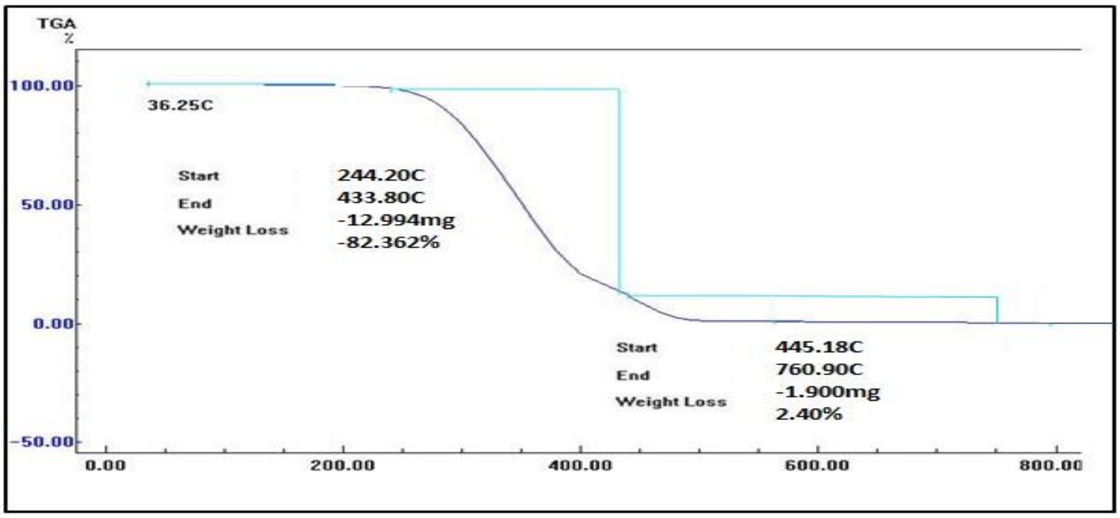


Fig.22. TGA pattern of the first pottery sample, wadi Kharig, Sinai.

Thermal analysis TGA showed weight loss (second pottery sample). Result of weight loss was 3.98 milligrams at firing temperature from 38.05°C to 134.99°C, weight loss was decreased to 1.92 mg from 446.72°C to 673.49°C, weight loss was decreased to 0.030 mg from 744.89° C to 800.10 °C. The results of weight loss indicated that the firing temperature was about 800.10° C, as shown in Fig.23.

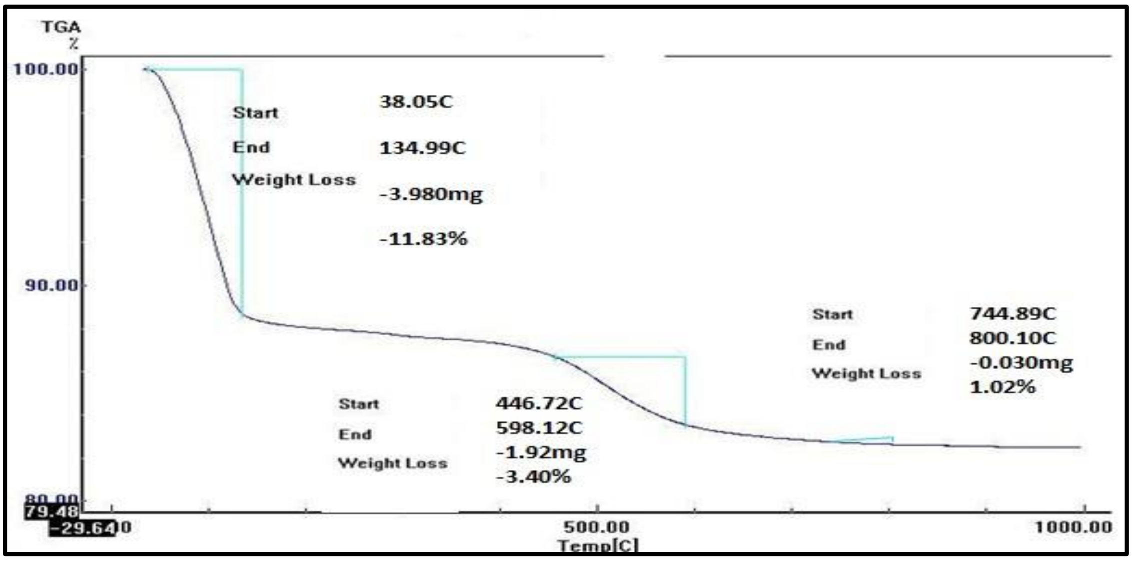


Fig.23. TGA pattern of the second pottery sample, wadi Kharig, Sinai.

4. Discussion of Results

Through extracted results from the research, it is clear from visual examination that the first pottery object was formed by the Potter wheel and the second piece was shaped by hollowing method, the surface treatment was slip layer for the first and red wash for the second object. Treatment was identified through industrial technology, as reported byShepared,(1981) and Arnold, (1993). Sandy soil deposits, crystallization of salts, breaking, cracking and surface deformation, as shown in Fig.3 (a & b).All damage manifestations were due to burial environment and climate changes after excavation (exposure environment) (Stambolov and Vanaspern de Boer, 1976). According to El-Gohary, 2015, it was also known as extrinsic deterioration factors. Crystallized salts, surface deformation, and cracking are indicators for impact of extrinsic deterioration factors (Petrakis and Steiger, 2015). Pottery was porous so it absorbed aqueous and saline solutions from the soil (Buys and Oakley, 2014).Soil is a source of salts for buried pottery in the soil (Cronyn, 1996).When archaeological pottery exposed to temperature, salts crystallized causing cracks or flaking of slip layer (Lehmann, 1970).

The rate of damage depends on chemistry of burial environment, type of salts and technology of industry (Elghareb, 2007). Pottery surface deformation is due to different soil deposits, dissolved salts. Breaking phenomenon is due to mechanical damage of the soil (Rice, 1996).Pottery pots had thermal energy (firing) that lead to cracking of pottery body to get rid of its potential energy (Mohamed, 1991). Through our presented paper, it can be claimed that soluble salts commonly associated with pottery damage are halite NaCl and carbonate salts. Source of halite salts is soil, as it is a distinctive feature of Egyptian soil (Abdel Fattah, 1990). When salt crystallizes (re- crystallizes), it increase in its volume more than original size which leads to collapse of internal structure of pottery body (Ahmed, 2003). Source carbonates salts is due to acidic rains or decomposition of dolomite (added temper), Where tempers were added during kneading and preparing clay for shaping (Mohamed, 1997).When salt re- crystallizes, salts caused cracking , flaking of slip layer and surface deformation (Ashley Smith, 1999). Pottery is Fragile (Torraca, 1982). cracks was due to evaporation of water by drying, firing (Selsing, 1961 and Goffer 2007).Salts crystallization and thermal shocks cause some damage manifestation such as cracking and weak cohesion of grains (Jin and Mai, 2005). Through our presented results, cracks, salts, flaking and gaps are shown as in Fig.11, Fig.12, Fig.13 and Fig.14. Surface deformation is due to burial in soil for long periods of time (Hodge, 1986). In our case study, Surface deformation is due to soil deposits, man-made deterioration is writing with the ink as shown in Fig.3 b.

Polarized microscope proved that used clay in shaping was Nile clay; it is due to presence of muscovite, biotite, as shown in Fig.5 & 7, pyroxene, as shown in Fig. 6, rutile and iron, as shown in Fig. 5 & 9. According to Saad (2018), Mica, pyroxene, rutile and iron are Characteristic of Nile clay. Through presented examination results, it also showed that additives such as sand, grog powder, as shown in Fig.5&9 .Limestone powder, as shown in Fig.8 &10. Examination also demonstrated that burning atmosphere was an oxidizing atmosphere due to presence of hematite (Grimshaw, 1971). According to Tite (2007), presence of magnetite and black core indicates reduced atmosphere. Upon our presented paper, PLM examination also demonstrated presence of coarse fabric for the first archaeological specimen as shown in Fig.6 & 7, medium fabric for the second specimen as shown in Fig. 8 & 9. According to Salah (2019), sodium oxide, potassium oxide, calcium oxide, iron oxide, titanium oxide and iron are Characteristic of Nile clay in Egypt. In our presented paper, SEM-EDX examination demonstrated that used clay was Nile clay as it contained sodium, potassium, calcium, iron, titanium and iron, as shown in Fig.15, Fig.16, Fig.17, and Fig.18.low carbon ratio indicates reduced atmosphere and presence of calcite showed that firing atmosphere is reducing (Rigby and Freestone,1997).In our presented research, SEM-EDX results also showed fire atmosphere, carbon ratio was 2.08% for first sample, as shown in Fig.15, it was 1.08% for second sample, as shown in Fig.18. Slip layer flakes if it is thick layer (Reed, 1988). In our SEM-EDX results, it demonstrated that slip layer was peeling, as shown in Fig.11&13. SEM-EDX also showed presence of crystallization of salts such as halite, carbonates and sulfates in specimens, as shown in Fig.16 &18.

XRD pattern contained quartz (SiO_2), hematite (Fe_2O_3), albite ($NaAlSi_3O_{10}$), and microcline ($KALSi_3O_8$), as shown in Fig.19, mineral components are characteristic of Nile clay in Egypt (elghareb,2017) .XRD also demonstrated that additives materials were dolomite powder for the second pottery specimens, as shown in Fig.20. In XRD results, it also showed that it contained halite salt (NaCl). Halite is soluble salt that caused pottery damage in our study case; halite salt is characteristics of Egyptian soil. Salts are due to acidic rain effect or surface ground water (Poulwencil, 1978).XRD also showed that burning atmosphere was an oxidizing due to presence of hematite, as shown in Fig.19.

Thermogravimetric Analysis "TGA" is used to determine firing temperature of archaeological pottery due to loss of weight (Matějkováa and Stoksikb,2011) .TGA of the first pottery sample showed firing temperature effect, weight loss was 12.99 milligrams for losing mechanical combined water of clay at firing temperature from 244.20 °C to 433.80 °C, weight loss was decreased to 1.9 mg from 445.18 °C to 760.90 °C, as a result of decomposition of carbonate into CaO and Co_2, burnt organic residues into Co_2 and chlorides, as shown in Fig.21.TGA of the second pottery sample showed firing temperature effect, weight loss was 3.98 milligrams for losing mechanical combined water of clay at firing temperature from 38.05 °C to 134.99 °C, weight loss was decreased to 1.92 mg from 446.72°C to 673.49 °C, weight loss was decreased to 0.030 mg from 744.89° C to 800.10 °C as a result of decomposition of carbonate into CaO. This indicates microstructure changes of pottery body. Through thermal analysis of our case study, TGA proved that burning temperature of the first pottery was about 760.90°C; burning temperature of the second pottery was about 800.10°C.

Conclusion

Research proved through the results, clay used in shaping of archaeological pottery (wadi Kharig, South Sinai, Egypt) was Nile clay. The additives were sand, pottery powder" Grog" and dolomite powder. Surface treatment was slip layer for the first pottery object and red wash for the second pottery object. It proved that burning atmosphere of the first and second pottery objects were oxidized. The research also demonstrated some damage manifestations such as crystallization of halite, carbonates, weakness, fragility, surface deformation, granulation, fragmentation, gaps, scaling, and cracking, as well as phenomenon of flaking of slip layer. The research recommends that materials and methods used in treatment of pottery objects (cleaning, salts extraction, consolidation, display and museum storage) should be chosen based on mineral composition and different properties, as well as various deterioration manifestations that have been identified by researcher.

Declaration of Competing Interest

I disclose that I have no any financial and personal relationships with other people or organizations that could inappropriately influence my work reported in this paper, No conflict of interest exits in the submission of this manuscript.

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