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ANALYSIS AND RESTORATION OF POTTERY ARTEFACTS FROM TELL El-HIR, NORTH SINAI, EGYPT. A CASE STUDY

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Abstract

The research paper aims to identify archaeometric features, damage and restoration of pottery artefacts from Tell EL-Hir in north Sinai Egypt, one of the archaeological sites dating back to Greco-Roman era. Polarized Microscopy PL", Scanning Electron Microscope with Energy dispersive X-ray unit SEM-EDX, X-Ray Diffraction analysis XRD, and Differential thermal analysis DTA were adopted for investigating and analyzing pottery sherds. The research identified archaeometric features proving that the used clay is Nile clay, the additives are sand, grog and calcite. Shaping technique is potter wheel. Surface treatment is slip layer. Firing atmosphere is oxidizing. Firing temperature might be about 726.78 °C for the first pottery vessel and 737.80°C for the second pottery plate. Research also proved that pottery pieces suffer from various damage aspects such as soil deposits, stains, cracking, fracture, loss of some parts, lack of durability, weakness, and salt crystallization. The studied pottery pieces were treated using mechanical and chemical cleaning using a mixture of acetone and toluene at a ratio of 1:2 respectively to remove clay soil deposits. EDTA was applied to remove lime deposits. Hydrogen peroxide 20% was used to remove soot. Nano Silica 1% was applied to strengthen archaeological pottery. Assembling the pottery sherds was done by Paralloid B72 50%. Replacement pottery plate was conducted by a mixture of Micro balloon and grog in a ratio of 2:1, respectively. After treatment, the pottery artefacts were ready for museum display.

***Keywords**: Additives; Damage; Treatment; Poultice; Replacement.*

Introduction

Tell EL-Hir is located near the city of eastern El-Qantra, North Saini Governorate, Egypt [1]. The governorate includes some archaeological sites such as Tell Abu Seifi, Tell Makhzan, Tell Al-Kidwa, Tell Al-Farma, Tell Al-Luli, Tell Habwa [2], which represents one of regions of Lower Egypt provinces [3]. Excavations have revealed many pottery and metals artefacts [4] The excavated pottery pieces suffer from various damage manifestations such as soil sediments, cracking, crystallization of salts, various stains, fractures and loss of some parts [5],which requires examination, analysis, restoration and maintenance based on the results of examinations and analyzes. Restoration and treatment methods depend on damage condition of pottery pieces. Choosing appropriate cleaning method is not an easy and simple process due to multiplicity and diversity of used materials for restoration, treatment, and maintenance of pottery and ceramics. This process has become one of the important topics in the field of treatment [6].

Many different cleaning materials used to depend on the damage. Among modern used materials in cleaning process recently are carbogel [7]. EDTA used to remove various lime

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deposits [8], organic solvents for removal soil sediments [9]. Hydrogen peroxide applied to clean soot [10], dichloromethane give the same result [11]. Nano-micro emulsions cleansers are liquid, stable and transparent O/W micro emulsions [12]. Most of the pottery artifacts have a weak physical structure, which requires strengthening by one of the appropriate consolidants, whether acrylic, silicon, acrylic-silicon, or nano composites. In the past few years, nano composites used to strengthen the pottery objects. Some nano materials were added to acrylic and silicon polymers in order to improve their physicochemical and mechanical properties, where it achieved a significant improvement in the ability of those polymers to strengthen the pottery [13]. Pottery or glass pieces are subject to breakage and the loss of some parts due to burial soil [14], weak internal structure and internal pressures impact [15], or as a result of pressures and external loads [16]. Such pieces need assembly and completion processes. These processes are complex technical techniques [17]. The adhesive and completion materials have varied based on their physical and chemical nature [18]. The materials used in cleaning, strengthening, assembling and completing have varied according to experimental studies in the field of treatment. Therefore, this study aims to present strategies of treatment of pottery artefacts extracted from Tell Hir in North Sinai according to results of examinations, analyzes and damage manifestations proven by research.

Materials and Methods

Materials

Three pottery samples were selected, including 2 pottery samples, as well as one sample of site soil. Those samples used in examinations and analysis that conducted in the research.

Methods

Visual Examination

Visual examination is one of the important methods that show pottery archaeometric features and damage manifestations by using different lenses 4X-6X or USB digital microscope [19].

Examination by polarizing microscope

Polarizing microscope used to study petrographic aspects and firing conditions, additives and damage of mineral components of archaeological pottery [20]. Thin section of pottery sherds was prepared for petrographic examination using polarizing microscope Olympus BX51 TF Japan attached with digital camera under magnification 4X up to 40X. It conducted at faculty of Science, Cairo University.

Examination by scanning electron microscope with EDX unit

Scanning electron microscopy, coupled with X-ray electron scattering is a modern instrumental method that involves the co-existence system between the two techniques [21, 22], describes surface morphology and damage aspects [23]. The pottery samples examined by JEOL JSM-840 and SEM Quanta 200 FEG, XTE 325/D8395, the operating conditions were 20kV and 1×10^{-9}A. The resulting image can be formed by secondary electrons and backscatter electrons [24, 25]. This examination carried out by an environmental scanning electron microscope equipped with an EDX unit at Geological Survey Authority in Cairo.

Analysis by X-Ray Diffraction Method

X-ray diffraction analysis is one of the important methods that gives mineral composition of pottery, which helps to identify archaeometric features, and damage manifestations [26]. The device is Philips; the diffraction pattern is between 4–70° 2θ. The operating conditions carried out using Cu-K α radiation 40MA, 45kV, this analysis conducted at National Research Center in Cairo.

Thermal Analysis

DTA analyses was performed using Shimadzu-simultaneous thermal analysis type DTA-50 to determine the firing temperature of our samples and evaluate thermal materials characterization.*i*t explains how individual minerals change during a gradual increase in temperature after heating the sample in approximately 1000°C [27, 28].

Results and discussion

Visual Examination

The visual examination of the pottery pieces proved presence of soil sediments, crystallization of salts, soot, fracture and loss of some parts as shown in figure 1a,b.

Fig. 1. represents the pottery objects, Tell Al-Hir, North Sinai: A - pottery vessel; B - plate

Polarizing Microscope

Two pottery samples extracted from tell El-Hir were examined with polarized microscope. PLM first pottery sample shows quartz grains calcite, grog, plagioclase, iron oxides, and calcareous deposition (10X - CN) as in figure 2. PLM of the same sample also shows quartz grains, calcite, pyroxene, plagioclase, biotite, grog and iron oxides (10X - CN) as in figure 3.

PLM examination for another part shows semi-circular and sharp-angled quartz grains, as well as presence of grog, calcite, plagioclase, pyroxene, lime deposits and internal cracks in matrix rich in iron oxide (10X - CN) as in figure 4.

PLM of the second pottery sample shows semi-circular and acute-angled quartz grains, calcite, biotite and muscovite, polycrystalline quartz grains and iron oxide (10X - CN), as in figure 5. It also shows presence of semi-circular and acute-angled quartz grains, as well as polycrystalline quartz grains, limestone powder, plagioclase, some internal cracks, and iron oxide (10X - CN) as in figure 6.

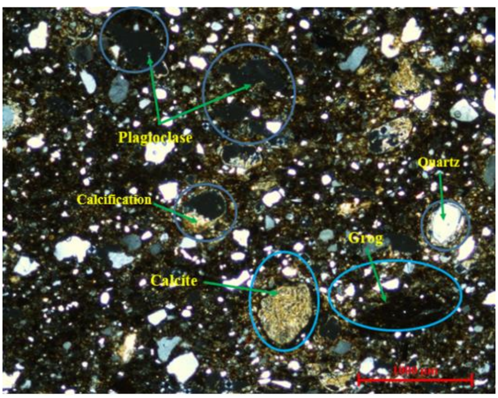

Fig. 2. PLM examination of the first pottery sample shows quartz grains calcite, grog, plagioclase, iron oxides, and calcareous deposition (10X - CN)

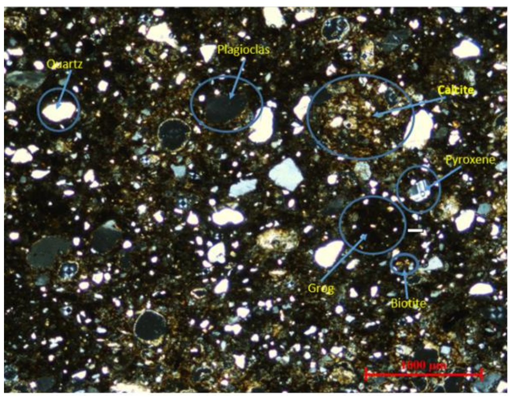

Fig. 3. PLM examination of the same pottery sample shows quartz grains, calcite, pyroxene, plagioclase, biotite, grog and iron oxides (10X - CN)

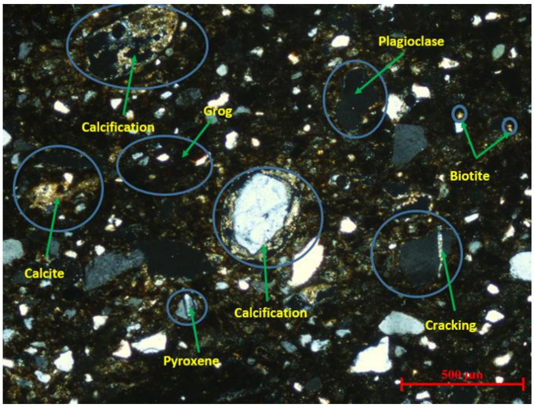

Fig. 4. PLM examination of another part pottery sample shows quartz grains, grog, calcite, plagioclase, pyroxene, calcareous deposits and internal cracks in matrix rich in iron oxide (10X - CN)

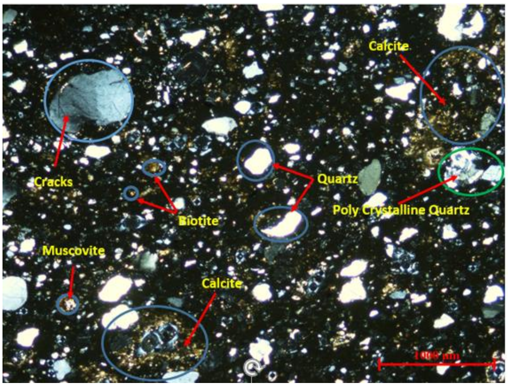

Fig. 5. PLM examination of the second pottery sample shows semi-circular and acute-angled quartz grains, calcite, biotite and muscovite, polycrystalline quartz grains and iron oxide (10X - CN)

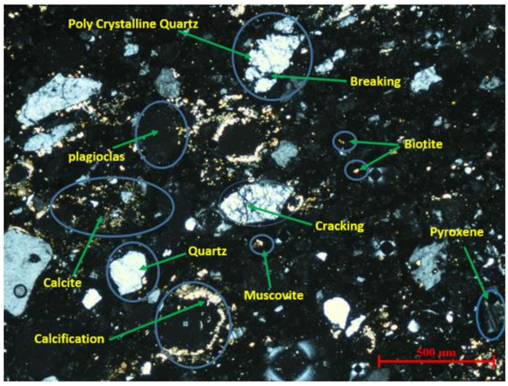

Fig. 6. PLM examination of the same sample shows semi-circular and acute-angled quartz grains, polycrystalline quartz grains, limestone powder, plagioclase, some internal cracks, and iron oxide (10X - CN)

Figure 7 for the same sample shows presence of polycrystalline quartz grains, limestone powder, plagioclase, biotite, muscovite, lime deposit and iron oxide (20X - CN).

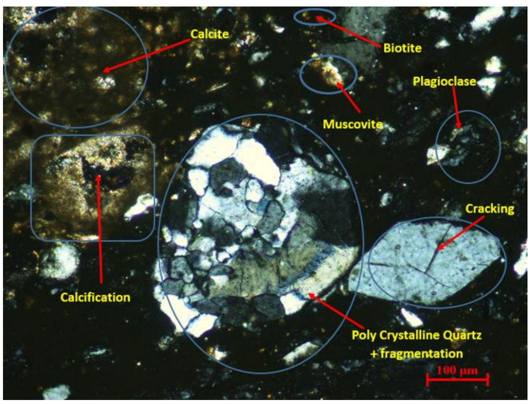

Fig. 7. PLM examination of another part pottery sample shows polycrystalline quartz grains, lime stone powder, plagioclase, biotite, muscovite, lime deposit and iron oxide (20X - CN)

Examination by Scanning Electron Microscope

SEM Examination of the first pottery sample shows that the sample suffers from various damage aspects such as phenomenon of cracking, some stains, quartz grains, and peeling off slip layer (200X), as in figure 8A. SEM Examination also shows phenomenon of cracking, gaps and crystallization of salts (3000X), as in figure 8B.

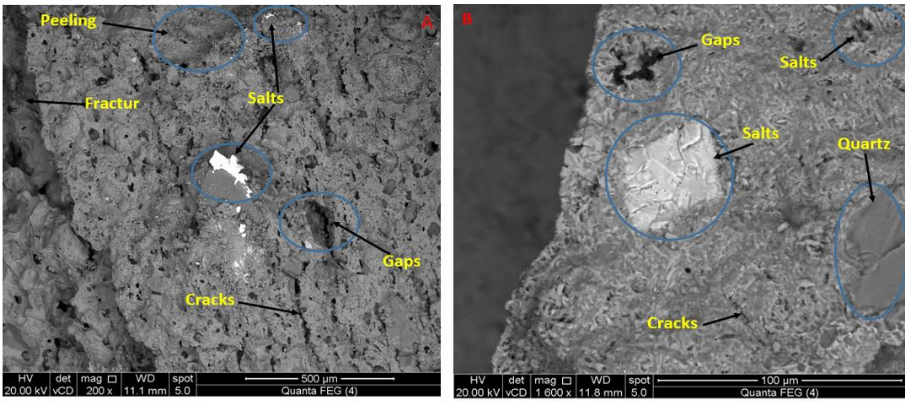

Fig. 8. SEM photomicrograph of the first pottery sample:
A - cracking, quartz grains, peeling, salts, and fracture;
B - cracking, gaps and crystallization of salts, accidently quartz grains

SEM Examination of the second pottery sample shows presence of fractures, cracks, gaps, exfoliation of slip layer and salt crystallization (200X), as in figure 9A. SEM of the same sample also shows phenomenon of crystallization of salts, gaps, cracks and coarse quartz grains (1600X) as in figure 9B.

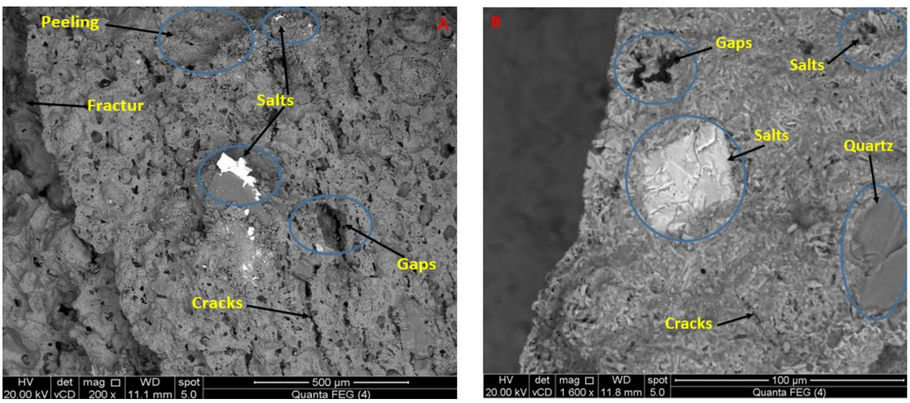

Fig. 9. SEM photomicrograph of the second pottery sample:
A: cracking, gaps, peeling, salts, and fracture;
B - cracks, gaps and crystallization of salts, accidently coarse quartz grains

The results of EDX analysis of three pottery samples as shown in figure 10A-D clarified presence of carbon, oxygen, fluorite, nickel sodium, magnesium, aluminum, silica, Molybdenum, sulfur, chlorine, potassium, calcium, titanium, and iron. These EDX analytical results for three sample results as shown in figure 10A-D and table 1.

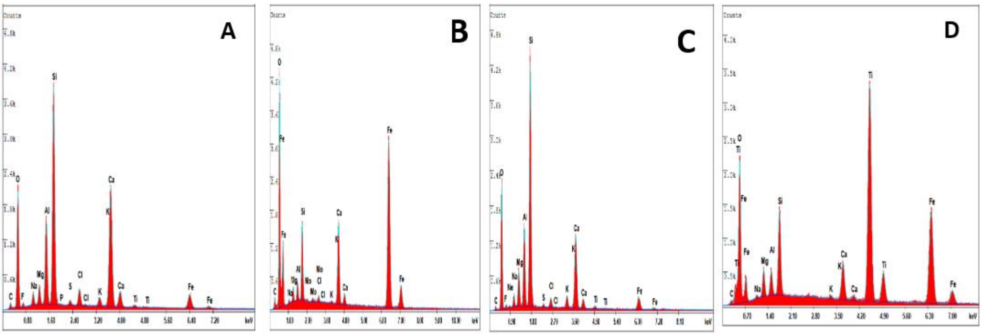

Fig. 10. EDX analysis patterns of pottery samples tell El- Hir, Sinai:
A and B - first pottery; C and D - Second pottery

Table 1. EDX analytical results of investigated pottery shards

	S1		S2	
Elemental Weight (%)	**Surface**	**Core**	**Surface**	**Core**
C	15.17	1.95	1.78	0.66
O	19.95	16.27	27.54	18.25
F	-	-	0.63	-
Ne	-	-	0.32	-
Na	9.82	0.42	1.44	0.31
Mg	2.54	1.10	3.50	1.80
Al	5.16	10.33	8.31	1.50
Si	12.78	13.82	23.93	4.76
Mo	-	0.60	-	-
P	-	-	-	-
S	3.13	-	3.43	-
Cl	16.19	4.45	4.46	-
K	1.01	0.25	2.37	0.35
Ca	9.43	8.25	14.77	6.48
Ti	0.20	-	0.90	30.24
Fe	4.63	51.52	7.57	35.65

X-Ray Diffraction Analysis

Three samples were analyzed, including 2 pottery samples and one sample from the soil of the archaeological site, where X-ray diffraction pattern of the first sample shows the presence of hematite, quartz, albite, calcite and gypsum as in figure 11.

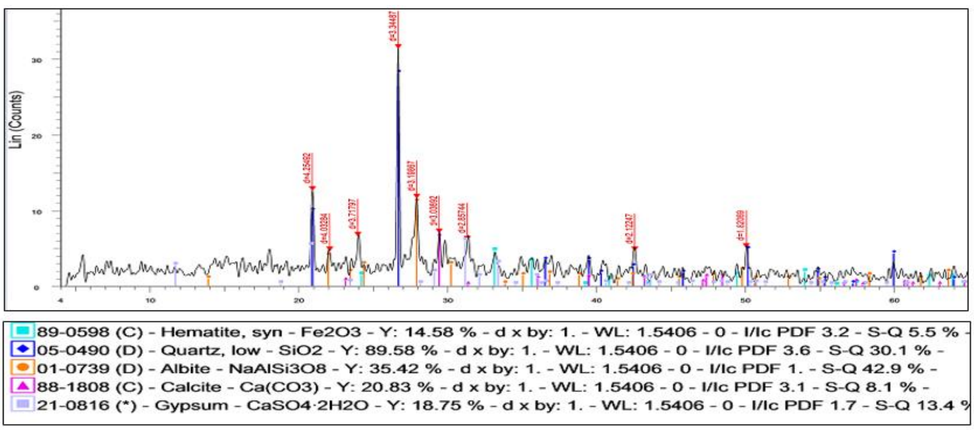

Fig. 11. XRD pattern of the first pottery sample tell El-Hir, Egypt

X-ray diffraction pattern of the second sample confirmed presence of quartz, albite, calcite and gypsum as in figure 12.

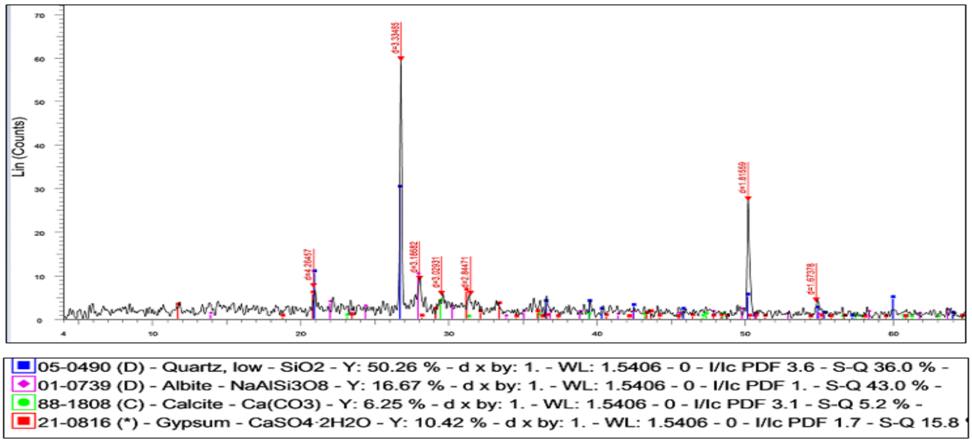

Fig. 12. represents XRD pattern of the second pottery sample, tell El-Hir, Egypt

The analysis of the third soil sample of tell El-Hir in North Sinai proved presence of quartz, albite and calcite, as in figure 13. It is clear from the results of the X-ray diffraction pattern that the soil is sandy clay rich in salt.

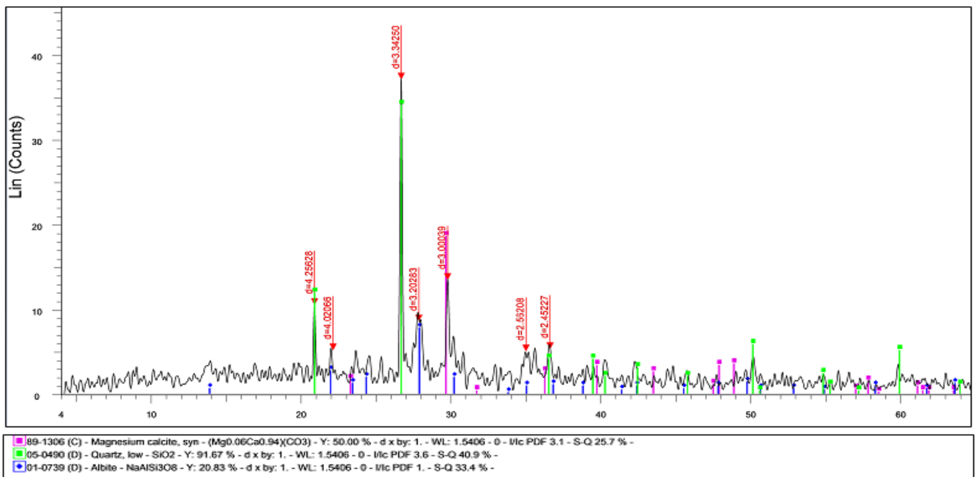

Fig. 13. represents XRD pattern of soil sample, tell L-Hir, Egypt

The results of analysis XRD are shown in the table 2

Table 2. XRD analytical results of investigated pottery shards

Mineralogical Compositions		Pottery Samples		
Minerals	**Chemical composition**	**a**	**b**	**C soil**
Quartz	SiO_2	30.1	36	40.9
Albite	$NaAlSi_3O_{10}$	42.9	43	33.4
Calcite	$CaCO_3$	8.1	5.2	25.7
Hematite	Fe_2O_3	5.5	-	-
Gypsum	$CaSO_4.2H_2O$	13.4	15.8	-

Differential thermal analysis

Differential thermal analysis DTA of the first pottery sample showed minerals changes in firing temperature from 31.21to 79.60°C, the minerals changes decreased from 109.80 to 164.00°C, the minerals changes also increased from 388.83 to 726.78°C, the stability of the mineral changes from 726.78 to 1000°C. The results of mineral changes indicated that the firing temperature might be about 726.78°C, as in figure 14.

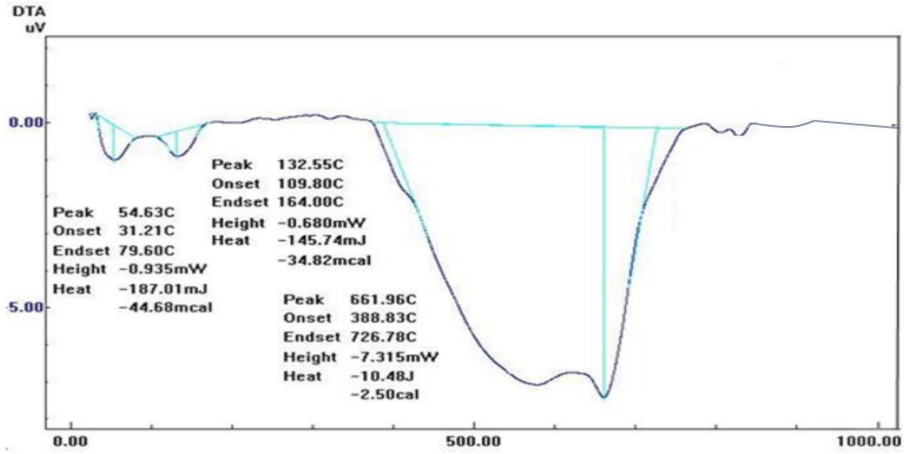

Fig. 14. DTA pattern of the first pottery sample, tell EL-Hir, Egypt

Differential thermal analysis DTA of the second pottery sample showed minerals changes in firing temperature from 294.72to 408.56°C., the minerals changes increased from 479.89 to 737.80°C, the stability of the mineral changes from 737.80 to 1000°C. The results of mineral changes indicated that the firing temperature might be about 737.80°C, as in figure 15.

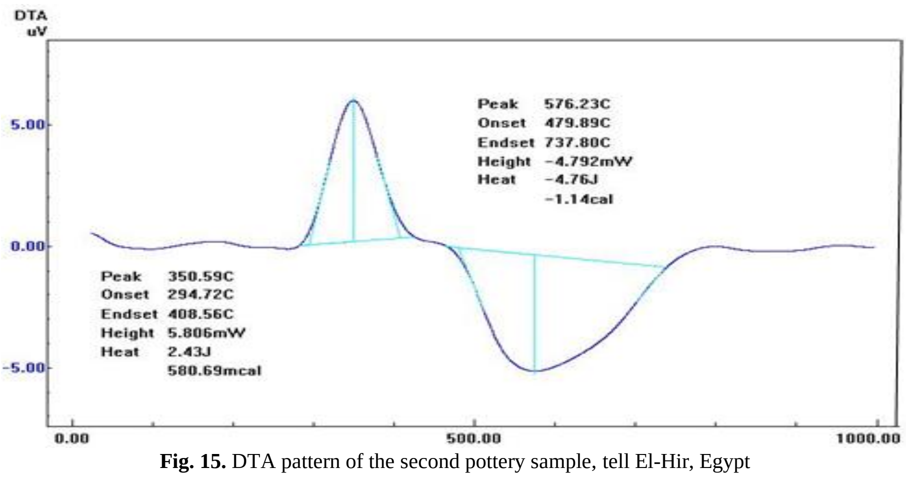

Fig. 15. DTA pattern of the second pottery sample, tell El-Hir, Egypt

Results and Discussion

The visual examination of the pottery pieces from tell El-Hir in North Sinai showed that the pottery samples formed by potter wheel method. It also shows soot, salts, cracking, lost parts and surface sediments as in figure 1A and B. Damage aspects were due to heterogeneity of mineral components [29]. Some researchers attributed the damage to burial in the soil [30]. The exposure environment (post excavation) increases severity of damage [31].

Polarized microscope proved that the used clay is Nile clay due to presence of biotite, muscovite, pyroxene and plagioclase [32] as shown in figures 2-7. PLM examination confirmed presence of some additives such as sand, limestone powder and grog, which are common materials in Egyptian pottery [33] as shown in figures 2-7. PLM examination also proved presence of surface treatment by slip layer. Sometimes, the ancient Egyptian used red wash [34]. The examination also showed presence of some internal cracks due to pressures and strains. Pottery texture was coarse for the first sample and from medium to coarse for the second sample as in figures 2-7. The texture is form and relationship of the granules to each other [35]. The texture varies according to clay, formation technique, surface treatment, and firing [36].

SEM examination showed presence damage aspects, such as cracks, gaps, fractures, crystallization of salts and stains, as in figures 8A and B & 9A and B. Cracks in our studied pottery are due to internal pressures and crystallization of salts [37]. SEM- EDX confirmed medium burning of the first pottery for presence of carbon dioxide with a high percentage 15.17% for the surface area and low percentage 1.95% for the core area. Good burning for the second pottery sample, where the percentage of carbon reached 1.78% for the surface and 0.66% for the core area. This indicates that the burning atmosphere was oxidizing atmosphere for pottery samples, as in table 1.

SEM-EDX confirmed presence of calcite, chloride, and sulfate salts, where the percentage of chlorine in the first pottery sample reached 16.19% for the surface and 4.45% for the core. For the second sample, the percentage of chlorine reached 4.46% for the surface as in the table 1. It also proved presence of calcium, where the percentage of calcium in the first pottery sample reached 9.43% for the surface and 8.25% for the core. For the second sample, the percentage of calcium reached 14.77% for the surface and 6.48% for the core as shown in the table 1. The percentage of sulfur in the first pottery sample reached 3.13% for the surface, while the percentage of sulfur in the second pottery sample reached 3.43% for the surface, which confirms presence of chloride and sulfate salts as in table 1. The crystallization of salts is one of the most important manifestations of damage because pottery artifacts is a hygroscopic material that absorbs saline water from the soil [38].

XRD proved presence of calcite as one of the temper additives [39]. The analysis also proved presence of gypsum and carbonates due to burial soil [40]. XRD analysis also revealed presence of albite, quartz and calcite in the archaeological site sample, which confirms that the soil is calcareous clay soil.

Differential thermal analysis DTA of the first pottery sample showed the firing temperature was about 726.78°C for the first pottery, where the hydroxyl group of mechanical combined water for clay was lost at firing temperature from 31.21 to 79.60°C. The minerals changes increased by hydroxyl group loss of chemical combined water of clay, burning and oxidation of organic matter from 109.80 to 164.0°C and then the minerals changes were significantly increased due to decomposition of carbonate into CaO and CO_2, organic residues and chlorides from 388.83 to 726.78.

for the second pottery, the firing temperature was about 737.80°C, where the hydroxyl group of mechanical and chemical combined water of clay, burning and oxidation of organic matter from 294.72 to 408.56°C and then the minerals changes were significantly increased due to decomposition of carbonate into CaO and CO_2, organic residues and chlorides from 479.89 and 737.80°C [41].

Treatment and maintenance

The study of the various examinations and analyzes to our studied pottery artifacts from tell El-Hir in north Sinai proved presence of soil sediments, as well as chloride, sulfate salts, and soot. Pottery objects suffer from breaking and loss of some parts. Pottery pieces were characterized by weakness and lack of durability. According to damage conditions, results of examinations and analyzes and scientific studies in this field. Restoration, treatment and maintenance processes were applied on our studied pottery artefacts.

Recording and Documentation

The pottery pieces in our site El-Hir in North Sinai were photographed as shown in figure 16 a, b.

Fig. 16. Case Studies for pottery artefacts: A - the first object; B - the second object

Cleaning

The pottery pieces were cleaned of soil deposits mechanically using various brushes. The sediments that were highly adherent to the surface were cleaned using metal scalpels. The cleaning method was carried out from top to bottom not to scratch the pottery [42]. It has given positive results, as in figure 17 a-f.

Fig. 17. Cleaning of pottery artifacts: A - before cleaning; B - during cleaning; C - after mechanical cleaning of the first object; D - the second object before cleaning; E - during cleaning; F - after mechanical cleaning

Chemical cleaning was carried out in order to clean the various soil deposits using organic solvents. A mixture of acetone and toluene in a ratio of 1:2 respectively was prepared; chemical cleaning was done locally for each piece separately [43]. Soot was removed using hydrogen peroxide 20% [44]. The calcareous sediments were cleaned using EDTA; it was applied as a poultice [45], as in figure 18 a,f .

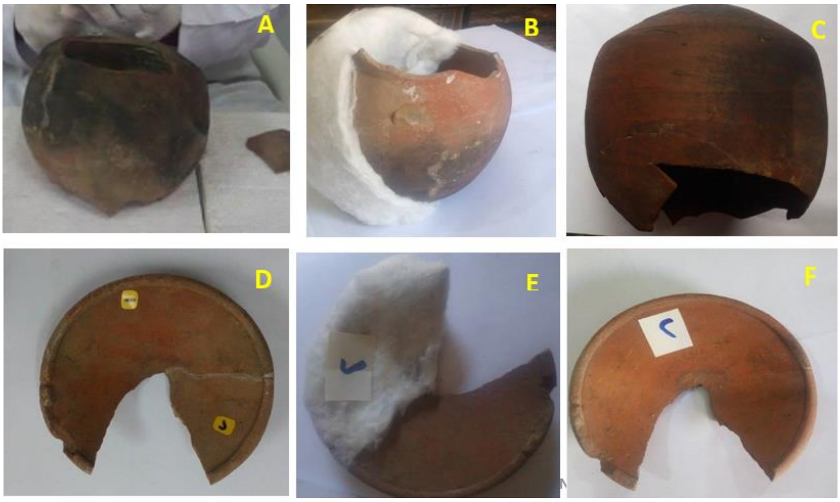

Fig. 18. represents cleaning of pottery artifacts: A - before cleaning; B- during cleaning; C - after chemical cleaning of the first object; D - the second object before cleaning; E - during cleaning; F - after chemical cleaning

Removal of Salts

The salts were removed mechanically using brushes and scalpels. Cleaning was done under lenses 6 X not to scratch the pottery [46]. Then, this was followed by making poultice of distilled water to remove soluble salts such as halite salts [47]. EDTA poultice was applied to remove calcareous deposits of carbonates and sulfates salts [48].

Consolidation

The pottery artefacts extracted from Tell El-Hir in North Sinai were strengthened using nano silica 1% [49]. Spraying method was applied in strengthening process [50].

Assembling

The initial assembly process was started to find out the places of fracture of pottery sherds [51]. Then, the pottery sherds were assembled using Paralloid B 72 dissolved in toluene 50% [52], as in figure 19 a, b, c.

Fig. 19. represents assembly of the first pottery object: A - before assembly; B - during assembly; C - after assembly

Filling Gaps

After the assembly process, it was noticed that the pottery artefacts were missing some parts. Replacement materials applied using a mixture of micro balloon and Pottery powder in a ratio of 2:1. First, alumina paper applied under the missing part to match the lost part. Second, completion material was prepared, which is a mixture of micro balloon and Pottery powder in a ratio of 2:1. Third, the completion material was applied. After an hour, it was shaped and polished using various molding tools [53]. Thus, the pottery pieces restored the artistic and archaeological form that it was before burial in the soil, as shown in figure 20 a, b,c .

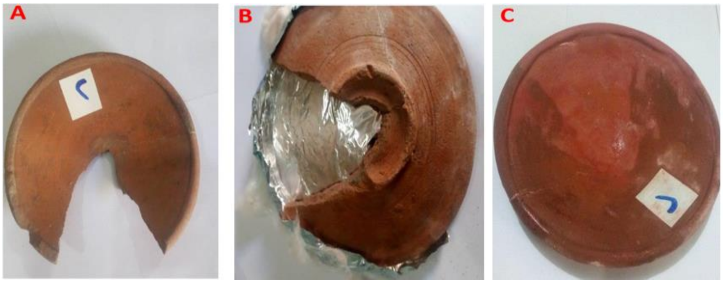

Fig. 20. Completion of pottery plate: A - Before completion;
B - After applying the reinforcing material (aluminum paper);
C - After completing the second pottery object

After replacement, the pottery was strengthened, protected and isolated using Paralloid B 82 (3%) by brushing. Pottery artifacts were ready to museum display as in figure 21 a, b.

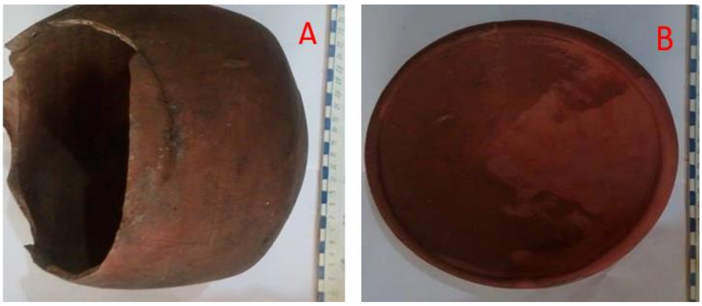

Fig. 21. Pottery objects after treatment: A - pottery vessel; B - plate

Conclusion

The research has reached a set of results of great importance in identifying the archaeometric features at tell El-Hir in north Sinai. Clay was Nile clay. The additives were sand, grog, and limestone powder. Shape technique used in manufacture of pottery pieces is potter-wheel forming technique. Surface treatment is of slip layer. Burning atmosphere was an oxidizing atmosphere. Firing temperature might be about 726.78°C for the first pottery vessel and 737.80°C for the second pottery plate. Research paper also proved that most of the pottery monuments from tell El-Hir in north Sinai suffer from damage aspects such as soil deposits, stains, fracture and loss of some parts, salts crystallization (chlorides, sulfates and carbonates), weakness and lack of durability of pottery pieces. The research found using of a mixture of acetone and toluene at a ratio of 2:1, respectively to remove clay soil deposits. Hydrogen peroxide 20% used to remove soot. Lime deposits were cleaned by EDTA. Nano-silica at a concentration of 1% used in strengthening archaeological pottery by spraying method. Mixture of micro balloon and pottery powder at a ratio of 2:1 respectively used in completing lost parts in our pottery objects. It is preferable to display the pottery artefacts in the museum at a temperature of 20°C and a relative humidity of 55-60%.

Acknowledgments

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